Designation: D7282 – 06

StandardPractice for
Set-up, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements[1]

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 reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers consensus criteria for the calibration and quality control of nuclear instruments. This practice is provided for establishing appropriate quality control parameters at instrument startup, calibration of nuclear counting instruments and the continuing monitoring of quality control parameters. Calibrations are usually performed to establish the operating parameters of the instrument. This practice addresses the typically used nuclear counting instruments: alpha spectrometer, gamma spectrometer, gas proportional counter and liquid scintillation counter.

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

1.3 *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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]
D1129 Terminology Relating to Water
D3648 Practices for the Measurement of Radioactivity
D4375 Practice for Basic Statistics in Committee D19 on Water

2.2 *Other Standards:*
ISO/IEC 17025 General Requirements for the Competence of Testing and Calibration Laboratories[3]
ISO 1995 Guide to the Expression of Uncertainty in Measurement[3]
ANSI N42.22 Traceability of Radioactive Sources to the National Institute of Standards and Technology (NIST) and Associated Instrument Quality Control[4]
ANSI N42.23 Measurement and Associated Instrumentation Quality Assurance for Radioassay Laboratories[4]
ANSI/HPS N13.30 Performance Criteria for Radiobioassay[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *acceptable verification ratio (AVR)*—ratio of the difference between 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. See Eq 10 in 16.2.13.

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

3.1.3 *calibration*—determining the instrument response to a known amount of radioactive material.

3.1.4 *calibration source (CS)*—a known quantity of radioactive material, traceable to a national standards body, prepared for the purpose of calibrating nuclear instruments.

3.1.5 *continuing instrument quality control*—measurements taken to ensure that an instrument responds in the same manner subsequent to its calibration.

3.1.6 *instrument check source (ICS)*—a radioactive source, not necessarily traceable to a national standards body, that is used to confirm the continuing satisfactory operation of an instrument.

3.1.7 *instrument contamination check (ICC)*—a measurement to determine if a detector is contaminated with radioactivity.

3.1.8 *instrument quality control chart*—a chart developed to evaluate the response of an instrument to predetermined, statistically based limits.

3.1.9 *instrument quality tolerance limit*—a limit established to evaluate the acceptable response of an instrument.

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[1] 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] 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.

[3] Available from International Organization for Standardization (ISO), 1 rue de Varembé, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

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

3.1.10 *known value (KV)*—known value of the analyte activity added to the verification sample. See Eq 7 in 16.2.11.

3.1.11 *mean and standard deviation*—see Practice D4375.

3.1.12 *measured value (MV)*—measured value of the verification sample. See Eq 4 in 16.2.9.

3.1.13 *measurement quality objective (MQO)*—quantitative or qualitative statement of a performance objective or requirement for a particular method performance characteristic (**1**).[5]

3.1.14 *national standards body*—an organization such as National Institute of Standards and Technology (NIST) or another national standards body that provides standards traceable to BIPM (Bureau International des Poids et Mesures (International Bureau of Weights and Measures)). Traceability is accomplished with guidance from ANSI N42.22.

3.1.15 *quality manual (QM)*—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.

3.1.15.1 *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 instrument check source (ICS), or instrument contamination check (ICC), or background subtraction count (BSC), or a combination thereof, failure.

3.1.16 *relative standard deviation (RSD)*—relative standard deviation of the mean expressed as a percentage (also known as coefficient of variation). See Practice D4375 and 16.2.7.

3.1.17 *sample test source (STS)*—a sample, sample aliquant, or final product of a chemical or physical process prepared for the purpose of activity determination.

3.1.18 *working calibration source (WCS)*—a calibration source (see 3.1.4), including those diluted or prepared by chemical procedure for the purpose of calibrating an instrument.

3.2 For definition of other terms used in this practice refer to Terminology D1129.

4. Summary of Practice

4.1 This practice summarizes information and guidance for set-up, 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 testing	Sections 10 through 13
Calibration	Sections 14 through 19
Continuing instrument quality control testing	Sections 20 through 25

5. Significance and Use

5.1 This practice is consistent with a performance-based approach wherein the frequency of re-calibration and instrument testing is linked to a laboratory's continuing performance with its quality control results. 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, QC 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 most likely reside in computer storage. This data remains in the computer storage where it is readily retrievable and as necessary is used to produce plots, graphs, spreadsheets and other types of displays and reports. Frequency and performance of data storage backup should be specified in the laboratory QM.

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 cognizant of hazards involved with all materials and processes employed and comply with any and all applicable health and safety procedures, plans and regulations. Material Safety Data Sheets are a source of information.

INSTRUMENT SET-UP

7. Scope

7.1 Instructions are provided for initial set-up 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 set-up 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 Set-up

9.1 *Gas Proportional Counting Initial Instrument Set-up:*

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[5] The boldface numbers in parentheses refer to the list of references at the end of this standard.

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

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 Establish appropriate instrument operational 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). Instrument set up and configuration should be optimized for the intended applications. For example, it may be desirable to perform voltage plateaus and optimize discriminator settings using a distributed source or a specific radionuclide (for example, a 2-in. diameter ^{230}Th source as opposed to a point source containing ^{210}Po) when intended applications use a different source geometry or radionuclide. If instrument set-up and configuration deviates from the defaults recommended by the manufacturer, the configuration and procedure to be used shall be specified in detail in the laboratory QM. Operating parameters should be established to produce consistency in detection characteristics across multiple detectors used for a common application. When the instrument operational parameters are satisfactorily established, record the "as-left" instrument settings for future reference.

9.2 *Gamma Spectrometry Initial Instrument Set-up:*

9.2.1 Upon initial set-up, after major repair or service, or when QC results indicate the need to reestablish operating parameters for an instrument, measure a suitable calibration source (that is, ICS or WCS) as specified in the laboratory QM and/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 set-up and initial calibration records should be maintained per applicable record requirements. ISO/IEC 17025 includes information regarding the type of records to save.

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 Establish 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 establish the desired energy per channel relationship. When the instrument operational parameters are satisfactorily established, record the instrument settings for future reference.

9.3 *Alpha Spectrometry Initial Instrument Set-up:*

9.3.1 Upon initial set-up, after major repair or service, or when QC results indicate the need to reestablish operating parameters for an instrument, measure a suitable calibration source (that is, ICS or WCS) as specified in the laboratory QM and/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 set-up and initial calibration records should be maintained per applicable record requirements. ISO/IEC 17025 includes information regarding the type of records to save.

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 operational parameters are satisfactorily established, record the instrument settings for future reference.

9.4 *Liquid Scintillation Counting Initial Instrument Set-up:*

9.4.1 Upon initial set-up, after major repair or service, or when QC results indicate the need to reestablish operating parameters for an instrument, measure a suitable calibration source (that is, ICS or WCS) as specified in the laboratory QM and/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 set up and initial calibration records should be maintained per applicable record requirements. ISO/IEC 17025 includes information regarding the type of records to save.

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 Establish the instrument operational 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 operational parameters are satisfactorily established, record the instrument settings for future reference.

INITIAL INSTRUMENT QUALITY CONTROL TESTING

10. Scope

10.1 Quality control testing may be initiated prior to instrument calibration to ascertain the instrument's operability and stability, or immediately after the calibration is complete to establish the continuing quality control parameters. The purpose of the instrument quality control testing is to verify that the instrument operating conditions are *(1)* acceptable for analysis of sample test sources and *(2)* equivalent to those to be established during calibration. Continuing instrument quality control results are compared to control charts, tolerance limits or 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 quality control performance parameters shall be monitored. These performance parameters may 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 quality control performance parameters are the quality control chart and tolerance limit. Instrument quality control protocols shall be clearly defined in the laboratory QM.

11.2 Quality control charts are used to monitor those continuing instrument quality control 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 the reported sample test source result a tolerance limit may be used. The laboratory QM shall indicate the appropriate tool, quality control chart or tolerance limit, for monitoring each continuing instrument quality control performance parameter.

12. Establishing the Control Chart

12.1 Using the appropriate ICS or ICC perform at least 7 measurements of the particular instrument quality control parameter, ensuring that the measurement conditions are reproducible and match the sample analysis conditions as closely as possible. For the initial establishment of the control chart, these measurements may be performed sequentially over a short period of time, but should span at least a 24-h period. To provide a better reflection of the variability of the instrument over time, some laboratories may choose to augment the initial control data set with additional points and update limits once 20 to 30 data points are available. In each case, the ICS or ICC being used should be removed from the instrument and re-inserted so that the control chart reflects variability in sample positioning.

12.1.1 For each instrument quality control parameter that uses a radioactive source, accumulate sufficient net counts to obtain a relative count uncertainty of <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, ^{99}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 Analogous to 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 is the BSC. The laboratory's QM shall specify the necessary 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.

12.2 Calculate the mean and standard deviation (see Practice D4375) of the measured parameter. Create a control chart with the measurement result on the vertical axis and the observation number (or measurement date) on the horizontal axis. Draw a horizontal line on the chart to represent the mean of the measurement value. Additional horizontal lines set as "warning limits" and "control limits," typically at two and three standard deviations, should be drawn. For parameters based on short-lived radionuclides it may be necessary to include a decay-correction factor in the warning and control limits, that is, limit lines will have a slope. The quality control data should be evaluated to establish that it is normally distributed, although very low counts are more likely to have a Poisson distribution. In this case the data could be tested against the Poisson model. 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.

12.3 Many instruments are provided with operation and analysis software that may include performance check and instrument QC charting capabilities. Stand-alone 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 measurements, it must support the statistical evaluation of the necessary parameters and be able to compare individual performance measurements with the established warning and control limits and advise the operator of performance measurement warnings and failures. The software used must be documented as specified in the laboratory QM.

13. Instrument Tolerance Limits

13.1 The purpose of the tolerance limit is to provide a comparison of measured instrument performance to acceptable instrument performance, and may be expressed as a percent (%) deviation of a mean performance measure. The basis for the tolerance limit may also be taken from the MQOs associated with a project or statement of work. A statistical evaluation must be performed to ensure that tolerance limits are achievable.

13.2 The tolerance limit differs from the control chart in that it is not based on statistical measures, but instead 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 criterion). The tolerance limit cannot be more restrictive than the control chart because a method or test cannot be expected to perform better than is statistically possible. 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 insure appropriate actions are taken before a tolerance limit is crossed.

13.3 For each performance parameter to be charted, establish the tolerance limit. The tolerance limit should be selected such that operation of the instrument just within the limits will not adversely affect the performance of the test or method. The tolerance limits should not be more restrictive than three times the measured standard deviation of the distribution of the control chart data set. For parameters based on short-lived radionuclides it may be necessary to account for radioactive decay when evaluating quality control data.

13.4 Create a tolerance chart with the measurement result on the vertical axis and the observation number (or measurement date) on the horizontal axis. Draw a horizontal line on the chart to represent the mean of the measurement values, and draw a horizontal line at the tolerance limit above and below the mean. These lines are called "tolerance limits."

13.5 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 measurements, it is necessary that the software be able to compare individual performance measurements with the established tolerance limits and indicate an out of tolerance condition. Stand-alone charting software can also be used for this purpose. The software used must be documented as specified in the laboratory QM.

CALIBRATION

14. Scope

14.1 The calibration process establishes the response of an instrument to a calibration source. The calibration source shall have a certified value (with uncertainty) that is traceable to a national standards body.

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 the performance criteria like those specified in standards such as ANSI N42.23 and ANSI/HPS N13.30, and References (**1**) and (**2**) are more probable when the calibration acceptance criteria in this practice are achieved 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 initial instrument set-up in preparation for counting calibration sources (CS or WCS). 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 sample test source residue mass varies little and the efficiency change is less than ±5 % over the expected mass range for the test or there is a near-massless sample test source.

16.2.2 To control the potential bias from a non-representative sample test source, the preparation method of the calibration sources should be as equivalent as practical to that used to prepare 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.

16.2.3 A minimum of three calibration samples shall be used. One CS is adequate when it is prepared by a separate entity, such as an independent laboratory or a commercial vendor.

16.2.4 A blank sample shall also be processed in association with the working calibration sources. The blank sample result should be compared to the laboratory's performance criteria as defined in its QM.

16.2.5 Select a WCS whose activity is sufficient to produce a count rate not exceeding 5000 counts per second (s^{-1}). It is essential that the count rate of the WCS be such to avoid instrument dead time that will result in lost counts. Count the WCS for a counting period to accumulate sufficient net counts to obtain a relative standard counting uncertainty of <1 % (10 000 net counts minimum).

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 Correct the WCS activity value for the radioactive decay (from the reference time to the time of the measurement). Calculate the counting efficiency, ϵ_{WCS}, using the equation defined in the laboratory QM or with example Eq 1.

$$\epsilon_{WCS} = \frac{R_a - R_b}{A_{WCS} \times Y_{WCS} \times DF} \tag{1}$$

where:

ε_{WCS} = single point efficiency of WCS (counts per second per becquerel (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 applicable,
DF = decay factor for the calibrating radionuclide $e^{-\lambda(t_1-t_0)}$,
λ = (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).

16.2.6.1 Eq 1 accounts for the total efficiency of the radionuclide even when the probability of alpha or beta emission per decay, stated as a decimal fraction is less than 1.0.

16.2.6.2 Calculate the combined standard uncertainty $u_c(\epsilon_{WCS})$, using the equation defined in the laboratory QM or with example Eq 2.

$$u_c(\epsilon_{WCS}) = \sqrt{\frac{\frac{R_a}{t_a} + \frac{R_b}{t_b}}{A_{WCS}^2 \times Y_{WCS}^2 \times DF^2} + \epsilon_{WCS}^2 \times \left(\frac{u^2(A_{WCS})}{A_{WCS}^2} + \frac{u^2(Y_{WCS})}{Y_{WCS}^2}\right)} \tag{2}$$

where:

$u_c(\varepsilon_{WCS})$ = the combined standard uncertainty of the single point efficiency ε_{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}, and
$u(Y_{WCS})$ = the standard uncertainty of Y.

NOTE 2—The other terms are defined for Eq 1.

16.2.6.3 Correction for decay during counting may be made by multiplying *DF* by the value, DF_a, obtained using Eq 3.

$$DF_a = \frac{1 - e^{-\lambda t_a}}{\lambda t_a} \tag{3}$$

where:

t_a = duration of count.

16.2.7 The uncertainty of the efficiency measurement is combined with other associated standard uncertainties to determine the efficiency calibration uncertainty. See Practice D4375 for the determination of the mean and standard deviation of the efficiency when two or more calibration samples are used. The estimated relative standard deviation (RSD, coefficient of variation) should be <5 %. The efficiency calibration uncertainty shall be included in the combined standard uncertainty of the sample result.

16.2.8 The single point efficiency calibration shall be verified prior to use by analyzing one sample that contains the same radionuclide prepared from a second primary (parent) source obtained from a supplier that is traceable to a national standards body. A blank sample should also be analyzed with the verification sample. Compare the blank sample result to the laboratory's performance criteria as defined in its QM. If a second primary (parent) source is unobtainable or not practical, a separate dilution of the original primary (parent) source shall be used. The activity placed in this sample should approximate the amount used in the calibration sample. (Note, the laboratory QM may establish alternate criteria for the activity and counting uncertainty to be used for the verification samples.) Measure this sample according to 16.2.5.

16.2.9 Calculate the verification sample activity, *MV*, using the equation defined in the laboratory QM or with example Eq 4.

$$MV = \frac{R_a - R_b}{\epsilon \times Y \times DF} \tag{4}$$

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),
ε = detection efficiency (see Eq 1 and 16.2.6),
Y = chemical yield of the verification sample, if applicable,
DF = decay factor for the calibrating radionuclide $e^{-\lambda(t_1-t_0)}$,
λ = (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).

16.2.9.1 To correct for decay during counting refer to Eq 3.

16.2.10 Calculate the combined standard uncertainty $u_c(MV)$, using the equation defined in the laboratory QM or with example Eq 6.

$$u_c(MV) = \sqrt{\frac{\frac{R_a}{t_a}+\frac{R_b}{t_b}}{\epsilon^2 \times Y^2 \times DF^2}+MV^2 \times \left(\frac{u^2(\epsilon)}{\epsilon^2}+\frac{u^2(Y)}{Y^2}\right)} \tag{5}$$

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 ϵ, and
$u(Y)$ = the standard uncertainty of Y.

NOTE 3—The other terms are defined for Eq 4.

16.2.10.1 The uncertainty components included in Eq 6 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 ISO 1995, or Reference **(3)**.

16.2.11 Calculate the known value, KV, using the equation defined in the laboratory QM or with example Eq 7.

$$KV = AC \times V \tag{6}$$

where:

KV = known value of the activity added to the verification sample,
AC = activity concentration in becquerels per litre (Bq/L) of the calibration source used to prepare the verification sample, and
V = volume of the calibration source used.

16.2.12 Calculate the combined standard uncertainty $u_c(KV)$, using the equation defined in the laboratory QM or with example Eq 9.

$$u_c(KV) = \sqrt{(u^2(AC) \times V^2)+(AC^2 \times u^2(V))} \tag{7}$$

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 calibration source used to prepare the verification sample, in becquerels per litre (Bq/L), and
$u(V)$ = standard uncertainty of the volume of the calibration source used.

NOTE 4—The other units are as defined in Eq 7.

16.2.12.1 Refer to the statement on uncertainty in 16.2.10.1 after Eq 6.

16.2.13 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 10, the acceptable verification ratio (AVR), where:

$$AVR = \frac{|KV - MV|}{\sqrt{u_c^2(KV)+u_c^2(MV)}} \leq 2.0 \tag{8}$$

where:

KV = known value of the activity added to the verification sample,
MV = measured value of the verification sample as calculated with Eq 4,
$u_c(KV)$ = combined standard uncertainty of the known value, and
$u_c(MV)$ = combined standard uncertainty of the measured value.

NOTE 5—This equation is similar to the one used in Reference **(1)**, Chapter 18, acceptance criteria for evaluating laboratory control samples. A "z" value of 2 is typical; however other "z" values may be used.

16.2.13.1 Refer to the statement on uncertainty in 16.2.10.1 after Eq 6. If the sample result fails to meet the *AVR* test, the system should be recalibrated and the test performed again.

16.2.14 When the criteria defined in 16.2.7 and 16.2.13 are met, the calibration is acceptable. The efficiency (ε_{WCS}) determined in 16.2.6 is used for calculating sample test source results.

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

16.3.1 To control the potential bias from a non-representative test source, the preparation method of the working calibration sources should be as equivalent as practical to that used to prepare the sample test sources to be analyzed. 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 In the situation that both alpha and beta emitting radionuclide counts are being done simultaneously separate sets of calibration sources shall be prepared.

16.3.3 Use a series of at least seven working calibration sources in replicate that will produce planchets or filters with sample test source residue mass such that the method expected range of residue is bracketed. One WCS should be near each end of the mass range, but not zero.

16.3.4 If more than two working calibration sources fail the laboratory's chemical yield requirements as established in the QM during the preparation process such that the loss causes one calibration point to be removed (assuming that the two are replicates), the calibration effort shall be restarted. Otherwise, when the minimum chemical yield is not achieved, the calibration data shall be removed from the population determining the curve. Consult the laboratory QM for chemical yield requirements.

16.3.5 Select a WCS whose activity is sufficient to produce a count rate not to exceed 5000 s^{-1}. Count the WCS for a counting period to accumulate sufficient net counts to obtain a relative standard counting uncertainty of <1 % (10 000 net counts minimum).

NOTE 6—It is essential that the count rate of the WCS be such as to avoid instrument dead time that will 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.

16.3.6 Correct the WCS activity value for the radioactive decay (from the reference time to the time of the measurement). Calculate the individual WCS efficiencies (ε_{WCS}) using the equation defined in the laboratory QM or with example Eq 11.

$$\epsilon_{WCS} = \frac{R_a - R_b}{A_{WCS} \times Y_{WCS} \times DF} \qquad (9)$$

where:

ε_{WCS} = single point 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, $e^{-\lambda(t_1 - t_0)}$,
λ = (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).

16.3.6.1 To correct for decay during counting refer to Eq 3.

16.3.6.2 Eq 11 accounts for the total efficiency of the radionuclide even when the probability of alpha or beta emission per decay, stated as a decimal fraction is less than 1.0.

16.3.7 Plot the efficiencies calculated from the working calibration sources against their residue mass.

NOTE 7—The calibration curve, (also referred to as an attenuation or absorption curve) should have a general downward trend, when the dependent variable Y (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 curve must be continuous and smooth. The slope of the line 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. The efficiency in the alternate channel (that is, crosstalk) should be continuous and smooth and have a general upward trend with decreasing 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.

16.3.8 Determine the equation of the calibration curve using the efficiencies calculated from the calibration samples. Several forms of equations may be used for these purposes among which are the polynomial equation and the power functions such as am^b, $a/(b + m)^c$, ae^{-bm} and $\ln(m)^a$, where m is sample mass, and a, b and c are fit parameters. In the case of the polynomial equation, 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.

16.3.9 Compare the observed efficiency to the efficiency calculated from the calibration curve at that test source residue mass. A variety of software packages, including basic spreadsheet applications, can optimize the calibration coefficients in order to minimize the difference between observed values and the fitted values obtained from the calibration.

16.3.10 Evaluate the agreement of the fitted curve to the actual values to ensure that it is appropriate to the residue mass range of the sample test source. In the absence of laboratory-defined acceptance criteria, the observed counting efficiency for each data point should deviate by less than 10 % relative to the calibrated value calculated from the fitted curve for that given sample test source residue mass. If all data points meet the acceptance criteria the curve is acceptable and the analyst should proceed to 16.3.11. Single data points that exceed the ±10 % acceptance criteria may be removed 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. In addition, visual outliers may be removed at the analyst's discretion. Decisions to exclude outliers should be documented. In no case should more than 20 % of the data points be removed. After removing the selected data points the analyst must return to the beginning of 16.3.8, perform a new fit of the remaining data points, and test the new fit against the acceptance criteria described above. If removal of another point would result in more than 20 % of the points being removed, the calibration process should be stopped and the protocol evaluated before the process is continued.

16.3.11 If the difference of a datum value from the mean of the paired values (paired efficiencies) is greater than 10 % for alpha or greater than 7.5 % for beta, delete the data pair. Return to 16.3.8 and perform another statistical fit to the data. Select a fit that has a smooth (not serpentine) curve through or between all the data points. Select a fit that has the best 95 % confidence limit around the fitted curve and/or has all data points within 7.5 % or 10 % relative to the calibrated value calculated from the fitted curve for alpha or beta respectively.

16.3.11.1 The loss of one replicate of a pair should not be cause for rejecting the calibration. See 16.3.4 and 16.3.10.

16.3.12 The standard deviation of the fitted curve calculated by the regression software in 16.3.8 is used with the other known uncertainties, such as the radioactive source and residue mass measurement uncertainties, to produce the standard uncertainty of the efficiency calibration curve. The efficiency calibration uncertainty shall be included in the combined standard uncertainty of the sample test source result.

16.3.13 To complete the acceptance of the calibration curve, the calibration curve (equation) shall be verified by analyzing three verification samples that contain the same radionuclide from a second primary (parent) source obtained from a supplier that is traceable to a national standards body. Include a blank sample with the verification samples. If a second primary (parent) traceable source is unobtainable or not practical, the calibration equation (curve) shall be verified by analyzing three verification samples that contain the same radionuclide from another dilution of the original primary (parent) traceable source. The residue masses of the verification samples should be distributed across the mass range of the efficiency curve. The activity placed in this sample should approximate the amount used in the calibration sample. Note the laboratory QM may establish alternate criteria for the activity and counting uncertainty to be used for the verification samples. Measure the verification samples according to 16.3.5. The measured value should fall within the quality control limits as established in the laboratory QM. The *AVR* test, Eq 10 in 16.2.13, may also be used.

16.3.14 When the criteria defined in 16.3.4, 16.3.7, 16.3.10, 16.3.11 and 16.3.13 are met, the calibration is acceptable. The calibration curve determined in 16.3.8 is used for calculation of sample test source results.

NOTE 8—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 Spectrometry Instrument Calibrations

17.1 Refer to the guidance in Sections 7 to 13 for counting the ICS and ICC at initial instrument set-up in preparation for counting calibration sources (CS or WCS). 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 10 calibration data points. The gamma-ray energy range in a CS is typically 50 keV to 2000 keV. Select a CS whose activity is sufficient to produce a count rate not to exceed 2000 s^{-1}. To control bias from a non-representative test source, the CS geometry should be as equivalent as possible to that used for sample test sources.

17.2.1 The density and Z number of a CS can have an effect on the detection efficiency, especially at energies < ~ 100 keV.

NOTE 9—It is essential that the count of the CS be such to avoid instrument dead time (typically <5 %) that will 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 10—A WCS may also be used instead of a CS.

17.3 Accumulate an energy spectrum using sealed, calibrated activity sources (that is, CS or WCS) traceable to a national standards body, 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 of <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 CS and the peak location data from the calibration spectrum establish the energy per channel relationship (energy calibration) as:

$$En = Offset + Ch \times Slope \tag{10}$$

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, and may include higher-degree polynomial terms to account for minor non-linearity in the energy calibration.

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

$$FWHM = Offset + En \times Slope \tag{11}$$

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, and may include higher-degree polynomial terms to account for minor non-linearity in the resolution calibration.

17.6 Calculate the full-energy peak efficiency, ε_f, using the equation defined in the laboratory QM or with example Eq 14:

$$\epsilon_f = \frac{R_n}{A_{CS} \times I_\gamma \times DF} \tag{12}$$

where:

ε_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 CS at the reference date and time of the calibration source,
I_γ = probability per nuclear decay for the gamma ray,
DF = decay factor for the calibrating radionuclide, $e^{-\lambda(t_1 - t_0)}$,
λ = 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 CS count (date and time).

17.6.1 To correct for decay during counting refer to Eq 3.

17.6.2 The calibration process (and subsequent sample test source analysis) 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." 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 random probability that any two or more gamma-rays emitted from the sample test source may be collected simultaneously. Random summing effects can be calibrated for and corrected for by making empirical measurements of sources with differing source strengths in the same counting geometry. Random summing is minimized to an inconsequential level by locating the source or sample such that the measurement system count rate is kept low (for example, less than 2000 s^{-1}). True coincidence or cascade summing is a consequence of the decay scheme of specific radionuclides. There are numerous radionuclides whose decay scheme provides for the simultaneous emission of two or more gamma-rays, which have some probability of being collected together. A common example of this is ^{134}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 as 1401 keV. This effect is not count rate dependent, but 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.

17.6.3 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}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).

17.6.4 True coincidence or cascade summing can be calibrated for and corrected for 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.

17.7 Many 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 instructions for specific requirements.

17.8 Mathematical modeling techniques may be used for the efficiency determination process 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.

17.9 Plot the values for the full-energy peak efficiency (as determined in 17.6) versus gamma-ray energy. Compare the efficiency curve to the typical efficiency curve for the detector type. The curve should be smooth, continuous and have a shape similar to the detector type being used. The plot will allow the determination of efficiencies at energies for which a CS is not available and to show that the algorithms used in computerized systems are providing valid efficiency calibrations. Select the fit that has the best 95 % confidence limit around the fitted curve and/or has all data points within ±8 % of the value of the fitted curve. This is accomplished by calculating the bias between the actual efficiency and the efficiency calculated with the fitted curve.

17.9.1 If the bias between an actual efficiency and a fitted efficiency value is greater than 8 %, the source of the bias may be coincidence summing. This is probable with a small source (or sample) configuration in very close proximity to the detector, such as a 50 mm diameter filter on the face of a 70 mm detector. In this case, the actual efficiency value should be corrected (see 17.6), the source should be located farther from the detector, or a calibration standard should be obtained with radionuclides lacking a cascade decay scheme.

17.10 Count a second CS as verification, using a second primary (parent) CS obtained from a supplier that is traceable to a national standards body. This calibration source (that is, CS or WCS) shall have at least two gamma-ray peaks of interest in the 50 keV to 2000 keV range and in the same counting geometry. An alternate second CS may be chosen whose radionuclides have gamma emissions that span an energy range similar to that of the original CS. If a second primary (parent) traceable source is unobtainable or not practical; the calibration equation (curve) shall be verified by analyzing a CS that contains the same radionuclides from another dilution of the original primary (parent) traceable source. Compare for each gamma-ray peak of interest, the efficiency from the initial efficiency calibration curve to the appropriate efficiency from the second calibration curve. If the calculated difference for any peak does not exceed ±8 % based on the initial curve, the new calibration curve determined in 17.6-17.9 is considered acceptable. The energy curve determined in 17.4 and the efficiency calibration curve determined in 17.9 are entered into the detector efficiency file and are used for the analysis of sample test sources. If the efficiency calibration curve was found unacceptable, go to 17.11.

17.11 If the difference between the original and verification measurements deviates by more than 8 % relative to the original measurement, verify the new efficiency with two additional measurements, one being with the original CS and the other a CS having 80 % or more of the same gamma-ray peaks of interest as the initial CS, or use the alternate CS as discussed in 17.10.

17.12 Calculate the efficiency for each gamma ray peak of interest in the verification counts.

17.13 Calculate the difference, for each gamma-ray peak, between the efficiency of the original and the verification measurements. For acceptable results, the difference for any gamma-ray peak of interest shall not deviate by more than 8 % relative to the original measurement.

17.14 Calculate the mean efficiency and the experimental standard deviation of the mean efficiency for each gamma-ray peak of interest. The relative standard deviation of the mean should not exceed ±5 %. When the criteria in this section and in 17.13 are met, the calibration is acceptable. The energy calibration curve determined in 17.4 and the efficiency curve calibration determined in 17.9 are used for analyzing sample test sources.

NOTE 11—An example of a gamma spectrometry calibration is provided in Appendix X3.