Designation: D7282 – 14

Standard Practice 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. The values given in parentheses are mathematical conversions that are provided for information only and are not considered 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:*

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

3. Terminology

3.1 *Definitions:*

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *acceptable verification ratio (AVR), n*—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.

3.2.1.1 *Discussion*—See Eq 10 in 16.2.13.

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

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

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

3.2.6 *instrument check source (ICS), n*—a radioactive source, not necessarily traceable to a national standards body, that is used to confirm the continuing satisfactory operation 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 American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

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

[5] 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.

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

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

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

3.2.10 *known value (KV), n*—known value of the analyte activity added to the verification sample.

3.2.10.1 *Discussion*—See Eq 7 in 16.2.11.

3.2.11 *mean, n*—see Practice D4375.

3.2.12 *measured value (MV), n*—measured value of the verification sample.

3.2.12.1 *Discussion*—See Eq 4 in 16.2.9.

3.2.13 *measurement quality objective (MQO), n*—quantitative or qualitative statement of a performance objective or requirement for a particular method performance characteristic (**1**).[6]

3.2.14 *national standards body, n*—an organization such as National Institute of Standards and Technology (NIST) or another similar national body that provides standards traceable to BIPM (Bureau International des Poids et Mesures (International Bureau of Weights and Measures)).

3.2.14.1 *Discussion*—Traceability is accomplished with guidance from ANSI N42.22.

3.2.15 *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.

3.2.15.1 *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.

3.2.16 *relative standard deviation (RSD), n*—relative standard deviation of the mean expressed as a percentage (also known as coefficient of variation).

3.2.16.1 *Discussion*—See Practice D4375 and 16.2.7.

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

3.2.18 *standard deviation, n*—see Practice D4375.

3.2.19 *working calibration source (WCS), n*—a calibration source (see 3.2.4), including those diluted or prepared by chemical procedure for the purpose of calibrating an instrument.

3.3 *Acronyms:*

3.3.1 *ADC*—analog digital conversion

3.3.2 *AVR*—acceptable verification ratio

3.3.3 *BIPM*—Bureau International des Poids et Mesures. Also known as International Bureau of Weights and Measures.

3.3.4 *BSC*—background subtraction count

3.3.5 *CS*—calibration source

3.3.6 *DF*—decay factor

3.3.7 *FWHM*—full width 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 *QC*—quality control

3.3.16 *QM*—quality manual

3.3.17 *RSD*—relative standard deviation

3.3.18 *STS*—sample test source

3.3.19 *WCS*—working calibration source

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.

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

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. 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:*

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 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 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 50.8-mm (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 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 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 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 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 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 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.

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

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 $u_c(\varepsilon_{WCS})$, using the equation defined in the laboratory QM or with example Eq 2.

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

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.

$$MV = \frac{R_a - R_b}{\varepsilon \times Y \times DF} \qquad (4)$$

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.

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

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 JCGM 100:2008 or Reference **(3)**.

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

$$KV = AC \times V \qquad (6)$$

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.

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

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:

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

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.

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

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:

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

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.

18. Alpha Spectrometry Instrument Calibrations

18.1 Refer to the guidance in Sections 7 to 13 for counting the ICS and ICC at initial instrument set-up in preparation for counting a CS or WCS. For instruments already in use count the ICS and ICC as prescribed in Section 24.

18.2 The energy, resolution and efficiency calibration may be established from the same calibration source.

18.3 For energy calibration, the CS should have two or more alpha emitting radionuclides whose energies span the region used for alpha spectrometry. If the energy calibration source is used independently of the efficiency calibration source, it does not need to be traceable to a national standards body. Electroplated calibration sources are available commercially. However, prepared coprecipitated calibration sources are not readily available on the open market; therefore the working calibration sources are typically prepared in the laboratory. Optimal results are achieved with a CS whose activity is approximately 100 Bq; this would produce a count rate of about 30 s^{-1}. To control bias, the sources used for efficiency calibration shall be prepared in the same manner as the sample test sources. The laboratory QM shall specify the alpha spectrometry counting geometry(ies).

18.3.1 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 approximately 100 Bq is a typical value and may vary according to instrument type, manufacturer and laboratory QM. Be aware of the probability of contamination from recoil, especially when ^{228}Th is present. Users should consult the manufacturer's specifications.

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

18.4 Establish the energy and resolution calibration in accordance with manufacturer's recommendations or laboratory protocol.

18.5 Count the energy and resolution CS to obtain sufficient net counts during the measurement period that will produce a relative counting uncertainty of <1 % (10 000 net counts minimum per alpha peak region of interest).

18.6 Perform the energy curve calibration as indicated in the manufacturer recommendations or plot the information manually using a spreadsheet or graph paper. Verify that the observed alpha energies of the key radionuclides in the energy calibration fall within <30 keV of the known value. Verify that the gain and resolution (FWHM) meet the manufacturer's recommendation or laboratory acceptance criteria.

18.7 Using the alpha emission data from the CS and the peak location data from the calibration spectrum, establish the energy per channel relationship (energy calibration) as:

$$En = Offset + Ch \times Slope \tag{13}$$

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).

18.7.1 Note that most modern spectrometry software packages perform this calculation, and may include cubic and quadratic terms to account for minor non-linearity in the energy calibration.

18.8 For efficiency calibration for sample test sources analyzed without an alpha emitting tracer, the CS shall have one or more radionuclides that are traceable to a national standards body. For efficiency calibration for sample test sources analyzed with alpha emitting tracers it is not necessary to use a CS traceable to a national standards body and the relative standard counting uncertainty may be <2 % (2500 net counts minimum). The detector shall be calibrated with a CS or WCS prepared in the same manner as sample test sources (electroplated or coprecipitated).

18.9 Count the efficiency CS one to three times. If the calibration sample is counted more than one time each, remove the CS and rotate approximately 90° before each count. The efficiency of a solid-state alpha spectrometry detector is not a function of energy over the typical energy range of emitted alpha particles, 3 to 8 MeV, due to the high stopping power of alpha particles in silicon and its relatively low band gap **(6)**. In some applications, instrument software averages the efficiency weighting, as appropriate, multiple alpha peaks. If more than one alpha emitter is used in the CS the total activity may be used to calculate the efficiency. The total relative counting uncertainty should be <1 % (10 000 net counts minimum per alpha peak region of interest).

18.10 Calculate the efficiency, ε_{CS}, from each count (s^{-1} Bq^{-1}) using the equation defined in the laboratory QM or with example Eq 16. When applicable, a correction is made for the probability of alpha emission per decay. Average the efficiencies, if counted more than once, and determine the standard deviation (see Practice D4375). If a WCS is used, replace CS with WCS.

$$\varepsilon_{\mathrm{CS}} = \frac{R_{\mathrm{a}} - R_{\mathrm{b}}}{A_{\mathrm{CS}} \times Y_{\mathrm{CS}} \times I_{\alpha} \times DF} \tag{14}$$

where:

$\varepsilon_{\mathrm{CS}}$ = efficiency of the individual CS ($\mathrm{s}^{-1}\ \mathrm{Bq}^{-1}$),
R_{a} = count rate (s^{-1}) of CS,
R_{b} = count rate (s^{-1}) of instrument background,
A_{CS} = activity (Bq) of the CS at the reference date and time of the calibration source,
Y_{CS} = if applicable, chemical yield of the WCS,
I_{α} = probability of alpha emission per decay, stated as a decimal fraction,
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).

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

18.11 When a sample test source is analyzed without a tracer the efficiency calibration shall be verified prior to use by analyzing a separate radionuclide that is traceable to a national standards body. 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.) 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.

18.12 Calculate the verification sample activity, MV; combined standard uncertainty $u_{\mathrm{c}}(MV)$; verification sample KV; combined standard uncertainty $u_{\mathrm{c}}(KV)$; and AVR using the equations defined in the laboratory QM or with Eq 4, Eq 6, Eq 7, Eq 9, and Eq 10, respectively, as defined in this standard. If the sample result fails to meet the AVR test, the system should be recalibrated and the test performed again.

18.13 The standard deviation calculated in 18.10, or the counting uncertainty when a single count is used for the efficiency determination, is used with other uncertainties such as the radioactive source and measurement uncertainties to determine the efficiency calibration uncertainty. When an alpha emitting tracer is added to the sample test source, the efficiency calculated using Eq 16 does not affect the sample test source result, but is used to calculate the chemical yield. However, when an alpha emitting tracer is not added to the sample test source, the efficiency calculated using Eq 16 is used; in this case the efficiency calibration uncertainty shall be included in the combined standard uncertainty of the sample test source result.

18.14 When the measurements in 18.6, 18.7, 18.10, and 18.12 are completed, the calibration is acceptable. The calibration efficiency, $\varepsilon_{\mathrm{CS}}$, determined in 18.10, is used for calculating sample test source results.

19. Liquid Scintillation Counting Instrument Calibrations

19.1 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 25.

19.2 This procedure is primarily for the calibration of a liquid scintillation counter (LSC) for a specific radionuclide using internal standards (CS or WCS) associated with the external standards ratio or the external standard technique. Counting efficiency of a LSC may be reduced by a number of factors, such as impurities, colored materials or from a chemical carrier used for yield monitor when one is used. This calibration applies to each radionuclide of interest.

19.3 Determine the expected range of quenching used in the procedure and select the quenching agent such as the chemical carrier or reagent added during sample processing, or a surrogate quenching agent such as nitromethane or chloroform that will produce the quench. At least 7 calibration sources with varying quench should be used. Working calibration sources may be used in place of the calibration sources. The WCS is typically prepared in the laboratory. Select a CS or WCS whose activity is sufficient to produce a count rate not to exceed 5000 s^{-1} to prevent loss of counts from coincidence. To control potential bias from a non-representative sample test source the preparation of each CS and WCS should be identical to the procedure used to prepare the sample test sources. In some situations the WCS will be carried through the entire chemical separation prior to preparation for liquid scintillation counting. The laboratory QM shall specify cocktail composition, water to cocktail ratios, counting volumes and the scintillation vials used. Some radionuclides are not available from a national standards body as traceable calibration sources.

19.3.1 Commercially prepared quench calibration sources are available for tritium and certain other radionuclides. Such calibration sources are not necessarily prepared with the same water/sample/cocktail ratio that is used in the laboratory and the counting efficiency from this CS might be slightly different than that from standard solutions prepared in the laboratory matrix. Since these commercial sources do not necessarily closely match varying sample test source configurations used in each laboratory, one must carefully evaluate whether the sample test source configuration is reproduced closely enough to meet data quality objectives.

NOTE 13—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.

19.4 Calibration sources and working calibration sources for liquid scintillation counting are prepared by adding known amounts of a radionuclide traceable to a national standards body and varying amounts of quenching agent (such as nitromethane or chloroform) to a mixture of liquid scintillation cocktail and other matrix elements which closely matches that observed in sample test sources. A poor match between the matrix of the calibration source and sample test source may significantly impact the quality of the liquid scintillation calibration.

19.5 Count each CS to accumulate sufficient net counts during the counting period to obtain a relative count uncertainty of <1 % (10 000 net counts minimum).

19.6 After the count, record the count rate (s^{-1}) and quench indicating parameters for the selected region of interest. Tabulate the data and calculate the efficiencies in (s^{-1} Bq^{-1}) as shown below. Correct the CS emission rates for the decay from the reference time to the time at which the count rate is measured, if not performed by instrument software. Calculate the quenched efficiency, ε_{CS}, for each quenched CS using the equation defined in the laboratory QM or with example Eq 19.

$$\varepsilon_{CS} = \frac{R_a - R_b}{A_{CS} \times Y_{CS} \times DF} \tag{15}$$

where:

ε_{CS} = single radionuclide efficiency of the individual CS (s^{-1} Bq^{-1}),
R_a = count rate (s^{-1}) of CS,
R_b = count rate (s^{-1}) of instrument background,
A_{CS} = activity (Bq) of the CS at the reference date and time of the calibration sample,
Y_{CS} = if applicable, chemical yield of the CS, normally used for a WCS,
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).

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

19.7 Determine the best fit of the line to the efficiency values. This can be performed manually; however, a regression analysis of the data may be performed with the use of commercially available statistical packages to obtain a calibration equation.

19.8 Determine the equation of the calibration curve using the efficiencies calculated from the calibration sources. Plot the fitted efficiencies with the actual efficiencies against the quench indicating parameters. Evaluate the agreement of the fitted curve to the actual values to ensure that it is a typical liquid scintillation efficiency curve for the radionuclide of interest. A variety of options are available in the software for calculating the form of the equation. The laboratory QM should include guidance for this.

19.9 The calculated residual values and standard deviation as well as the correlation coefficient (r^2) may be used as a means to evaluate how well the fitted curve fits the data. Select a fit that has the best 95 % confidence limit around the fitted curve and/or has all data points less than 7.5 % relative to the calibrated value calculated from the fitted curve. The agreement of the curve to the plotted data should also be visually evaluated to ensure that it represents a typical liquid scintillation counting efficiency curve for the radionuclide being calibrated. The laboratory QM should specify the options for the form of the equation.

19.10 The standard deviation of the fitted curve calculated in 19.9 is used with the other known uncertainties, such as the radioactive source and residue mass measurement uncertainties, to produce the uncertainty of the efficiency calibration curve. The efficiency calibration uncertainty shall be included in the combined standard uncertainty of the sample test source result. The statistical package software typically provides the equation uncertainty.

19.11 To complete the acceptance process, the calibration curve (quench curve) shall be verified by analyzing three verification samples that contain the same radionuclide from a second primary (parent) obtained from a supplier that is traceable to a national standards body.

19.12 If a second traceable source is unobtainable or not practical, the calibration curve (quench curve) shall be verified by analyzing three verification samples prepared from a separate dilution of the original primary (parent) source.

19.13 The activity placed in the verification samples should be adequate to provide approximately ±5 % counting uncertainty with the laboratory's normal sample test source counting time. Note the laboratory QM may establish alternate criteria for the activity and counting uncertainty to be used for the verification samples.

19.14 Calculate the verification sample activity, MV; combined standard uncertainty $u_c(MV)$; verification sample KV; combined standard uncertainty $u_c(KV)$; and AVR using the equations in the laboratory QM or with Eq 4, Eq 6, Eq 7, Eq 9, and Eq 10, respectively, as defined in this standard. The measured value of the verification samples should fall within the quality control limits established in the QM. The Acceptable Verification Ratio, Eq 10, may also be used.

NOTE 14—An example liquid scintillation calibration curve is provided in Appendix X4.

19.15 When the criteria defined in 19.9 and 19.14 are met, the calibration is acceptable. The calibration curve determined in 19.8 is used for calculating sample test source results.

CONTINUING INSTRUMENT QUALITY CONTROL TESTING

20. Scope

20.1 Information and guidance is provided in this section for performing continuous quality control functions and for evaluating continuing instrument quality control data. Information in 21.1 to 21.5 is of a general nature and applies to Sections 22, 23, 24, and 25. Gas proportional counting, gamma spectrometry, alpha spectrometry and liquid scintillation counting are addressed in Sections 22, 23, 24, and 25, respectively, on a specific basis. See also Practices D3648 for additional information.

21. Significance and Use

21.1 After the control chart(s) and tolerance limit(s) are established, as defined in Sections 12 and 13, respectively, continuing instrument quality control measurements should be performed and plotted at the frequency prescribed in the laboratory QM. Recommended frequencies for performance measurements are included in the individual instrument sections below. Compare each continuing instrument quality control measurement to the warning limits and control limits of the control chart or tolerance limit to establish instrument control. It may be necessary to adjust the control chart limit, tolerance limit or ICS value for decay when making the comparison.

21.1.1 The ICS shall be the same one used to establish the performance control chart. The ICS may also be the CS or WCS.

21.2 When a continuing instrument quality control measurement result falls outside the control limit, recount to confirm the measurement result. If a second measurement is also outside the control or tolerance limit, the instrument or detector shall be considered out of calibration. Investigate and correct the identified problem when acceptable performance can not be achieved. If the problem continues and the measurement result continues to fall outside the control limit and following the laboratory's QM the instrument shall be removed from service until the problem is resolved and documented. Consult the laboratory's QM (or other restricting documents and end-user communications) for guidance regarding the disposition of previous sample test source measurements under these circumstances.

21.3 For continuing instrument quality control measurement results that repeatedly fall outside the warning limit, but inside the control limit, consult the laboratory's QM for guidance. If measurements continue to fall outside the warning limit the instrument shall be removed from service until the problem is resolved and documented. Consult the laboratory's QM (or other restricting documents and end-user communications) for guidance regarding the disposition of sample test source measurements under these circumstances.

21.4 The control chart or tolerance limit or equivalent should be reviewed periodically (for example, every quarter) to look for trends. Measurement data should normally vary to a limited degree. The laboratory QM shall define the minimum frequency for review of control charts and/or tolerance limits.

21.4.1 Control chart measurement data should normally fall outside the warning limit but inside the control limit about 5 % (1 in approximately 20 measurements) of the time based on a Gaussian distribution. Measurement data should normally fall outside the control limit less than 1 % (1 in approximately 100 measurements) of the time based on a Gaussian distribution. Any trend other than randomly above and below the mean or consistently increasing or decreasing toward a control limit) should be investigated in a timely manner and in accordance with the laboratory's QM.

21.4.2 During the normal course of a laboratory's operational year many data points (>100) may be collected, depending on the sample test source count length, measurement period and type of instrument.

21.5 A periodic evaluation of background data to determine whether detectors have become contaminated for use shall be performed. The laboratory QM shall provide guidance for determining the presence of contamination and the actions to be followed when this has been determined. If contamination is confirmed, the laboratory will initiate investigations into the effect on sample test source measurements.

22. Quality Control for Gas Proportional Counters

22.1 Count the ICS to accumulate sufficient net counts in the main alpha or beta particle response channel used for the instrument check to obtain a relative count uncertainty of <1 % (10 000 net counts minimum). The ICS should be counted at least weekly or before use if the intervals between uses exceed one week. Major (main alpha or beta particle response channel) and minor (alpha particle response into the beta channel or beta particle response into the alpha channel) channel performance should also be checked when the cross-talk feature is used. The laboratory QM shall specify the frequency for the ICS count.

22.2 Compare the ICS measurement results to the control limits.

22.2.1 Tolerance limits may be used in lieu of control limits as directed by the laboratory QM and as discussed in Section 13. Several subsequent sections also provide instructions for comparing a quality control parameter to control limits. The use of tolerance limits as directed by the laboratory QM in these circumstances is appropriate.

22.3 An ICC is used to check for possible contamination of the detector (instrument). Count the ICC for at least the same duration that was used to establish the control chart. A BSC is counted to determine the value to use for subtraction from the sample test source count. The BSC shall be counted at least as long as the sample test source is counted. Major and minor channel performance should also be checked when the cross-talk feature is used. The laboratory QM shall specify the frequency of ICC and BSC measurements. If a laboratory decides to combine counts from several short counts, for example, several instrument contamination check counts to make a BSC, the laboratory QM shall establish adequate control over the counts and the appropriate statistics shall be used when the uncertainty of the sample test source result is determined.

22.4 Compare ICC or BSC measurement results to the appropriate control limits.

23. Quality Control for Gamma Spectrometry Systems

23.1 The ICS should have at least three measurable gamma-ray photopeaks (for example, one nuclide with at least three gamma energies or three radionuclides with three measurable photopeaks), with appropriate gamma emission energies to span the energy range of emissions anticipated in the sample test sources. One gamma-ray photopeak or one radionuclide with one photopeak may be used when only one radionuclide is determined.

23.2 Count the ICS to accumulate sufficient net counts in each main channel used for the instrument check to obtain a relative count uncertainty of <1 % (10 000 net counts minimum). The laboratory QM shall specify the frequency for making the ICS count. The ICS should be counted at least weekly or before use if the intervals between uses exceed one week.

23.3 For each peak used for the instrument check, compare the total peak area counts (count rate) of the ICS to the control limits.

23.4 For each peak location used for the instrument check, compare the peak location of the ICS to the control limits.

23.5 When suitable, for each photopeak used for the instrument check, compare the peak width (resolution) of the ICS to the control limits.

23.6 An ICC is used to check for possible contamination of the detector (instrument). For each gamma spectrometry system at least three regions of interest should be evaluated. When only one radionuclide is determined, monitor the background of the applicable photopeak. For certain system inherent or detector contamination radionuclides, the BSC is counted at least as long as the sample test source to determine the radionuclide's photopeak background count rate value to subtract from the radionuclide's gross photopeak count rate measured during analysis of the sample test source. The laboratory QM shall specify the frequency of BSC and ICC measurements. If a laboratory decides to combine counts from several short counts, for example, several instrument contamination check counts to make a BSC, the laboratory QM shall establish adequate control over the counts and the appropriate statistics shall be used when the uncertainty of the sample test source result is determined.

23.7 Compare BSC or the ICC measurement results to the appropriate control limits.

24. Quality Control for Alpha Spectrometry Systems

24.1 Instrument check sources are used for detector efficiency and energy calibration and can be used for their verification. This may be accomplished by using one ICS for both functions or one ICS for detector efficiency verification and another for energy calibration. The ICS that is used for energy calibration shall have at least two alpha emitters. One alpha emitter is adequate for detector efficiency verification.

24.2 Count the ICS to accumulate sufficient net counts to obtain a relative count uncertainty of <1 % (10 000 net counts minimum) in each selected region of interest. The ICS should be counted at least monthly or before use if the intervals between uses exceeds one month. The laboratory QM shall specify the frequency for the ICS count. For laboratory specific reasons, a 1 % counting uncertainty may be impractical and in these cases the laboratory QM may deviate from this practice.

24.3 For each alpha peak region of interest used for the instrument check, compare the net peak area counts (count rate) of the ICS to the control limits.

24.4 For each alpha peak location (centroid) compare the peak centroid location of the ICS to the control chart limits. When the alpha spectrometry software adjusts the gain and base level, a control chart for peak location is not necessary.

24.5 When suitable, for each alpha peak used for the instrument check, compare the peak width (resolution) of the ICS to the control limit.

24.6 An ICC is counted to check for possible contamination of the detector. Determine the background for each region of interest used for the radionuclides measured. A BSC is counted to determine the value to use for subtraction from the sample test source. The BSC shall be counted as long as the sample test source is counted. The QM shall specify the frequency of BSC and ICC measurements. If a laboratory decides to combine counts from several short counts, for example, several instrument contamination check counts to make a BSC, the laboratory QM shall establish adequate control over the counts and the appropriate statistics shall be used when the uncertainty of the sample test source result is determined.

24.7 Compare ICC and BSC measurement results to the appropriate control limits.

25. Quality Control for the Liquid Scintillation Counter

25.1 Liquid scintillation counting instrument check sources and liquid scintillation counting vials containing liquid scintillation cocktail used for instrument contamination check counts are typically obtained from the manufacturer. They are usually prepared and sealed in glass. The laboratory should be aware that these commercially prepared materials may have expiration dates.

25.2 Count the ICS to accumulate sufficient net counts to obtain a relative count uncertainty of <1 % (10 000 net counts minimum). The ICS should be counted at least weekly or before use if the intervals between uses exceed one week. The laboratory QM shall specify the ICS count frequency.

25.3 Compare the ICS measurement results to the control limits.

25.4 Compare the quench value of the ICS to the instrument manufacturer's limits or laboratory established control limits. The laboratory QM shall specify the control limits for quench.

25.5 Count the ICC at the same frequency as the ICS. Compare the measurement result of the ICC to the control limits. The laboratory QM shall specify the ICS and ICC count frequency.

25.5.1 The count data from the ICC shall not be used for background subtractions. A BSC that is analyzed and counted with the sample batch is typically used for background subtraction. The effect of quench on the BSC shall be established.

26. Keywords

26.1 activity; alpha; beta; calibration; control chart; gamma; gas proportional; liquid scintillation; measurements; quality control; spectrometry

ANNEX

(Mandatory Information)

A1. WORK FLOW CHARTS

A1.1 See Figs. A1.1-A1.4.

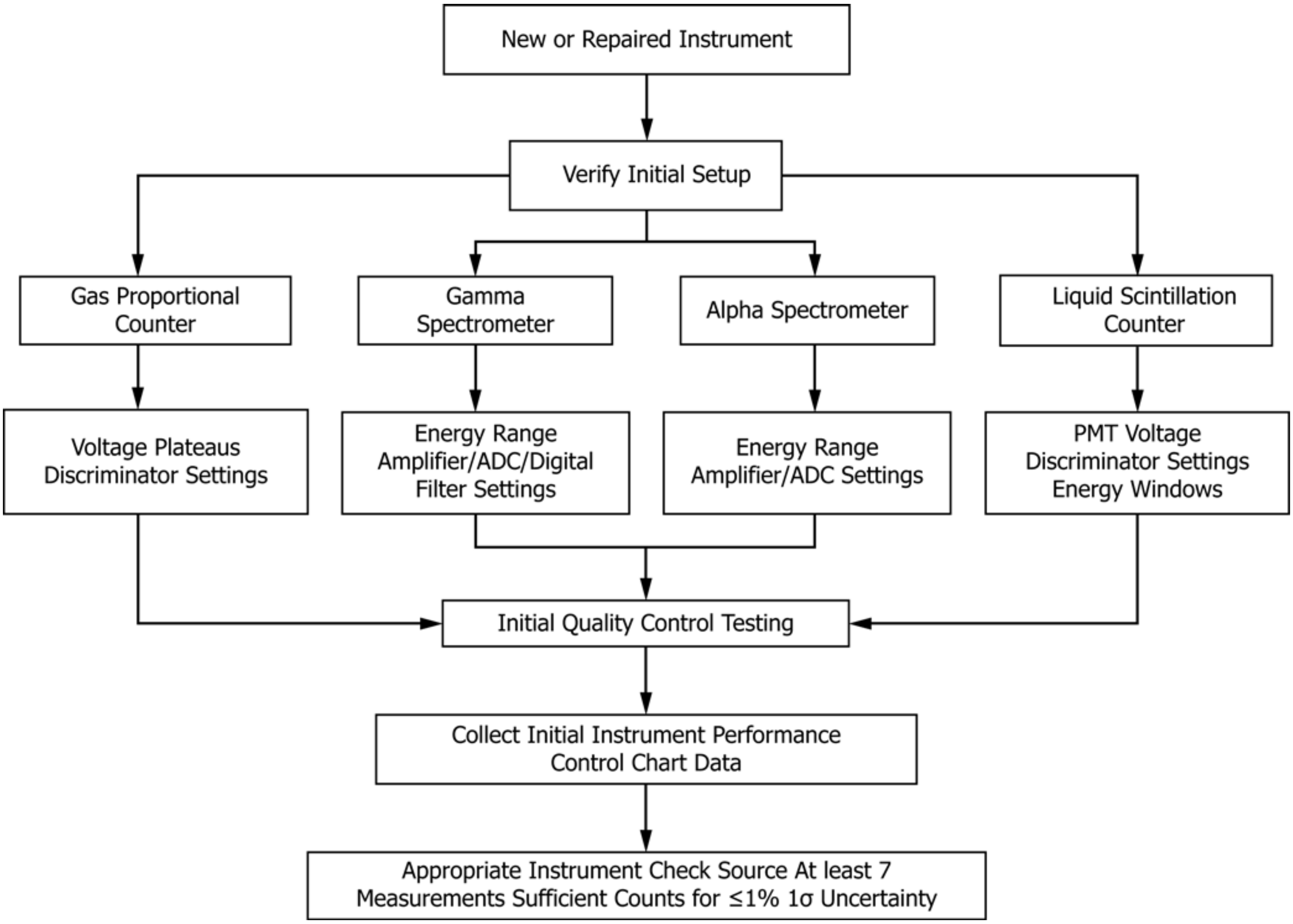

FIG. A1.1 New or Repaired Instrument Work Flow Chart

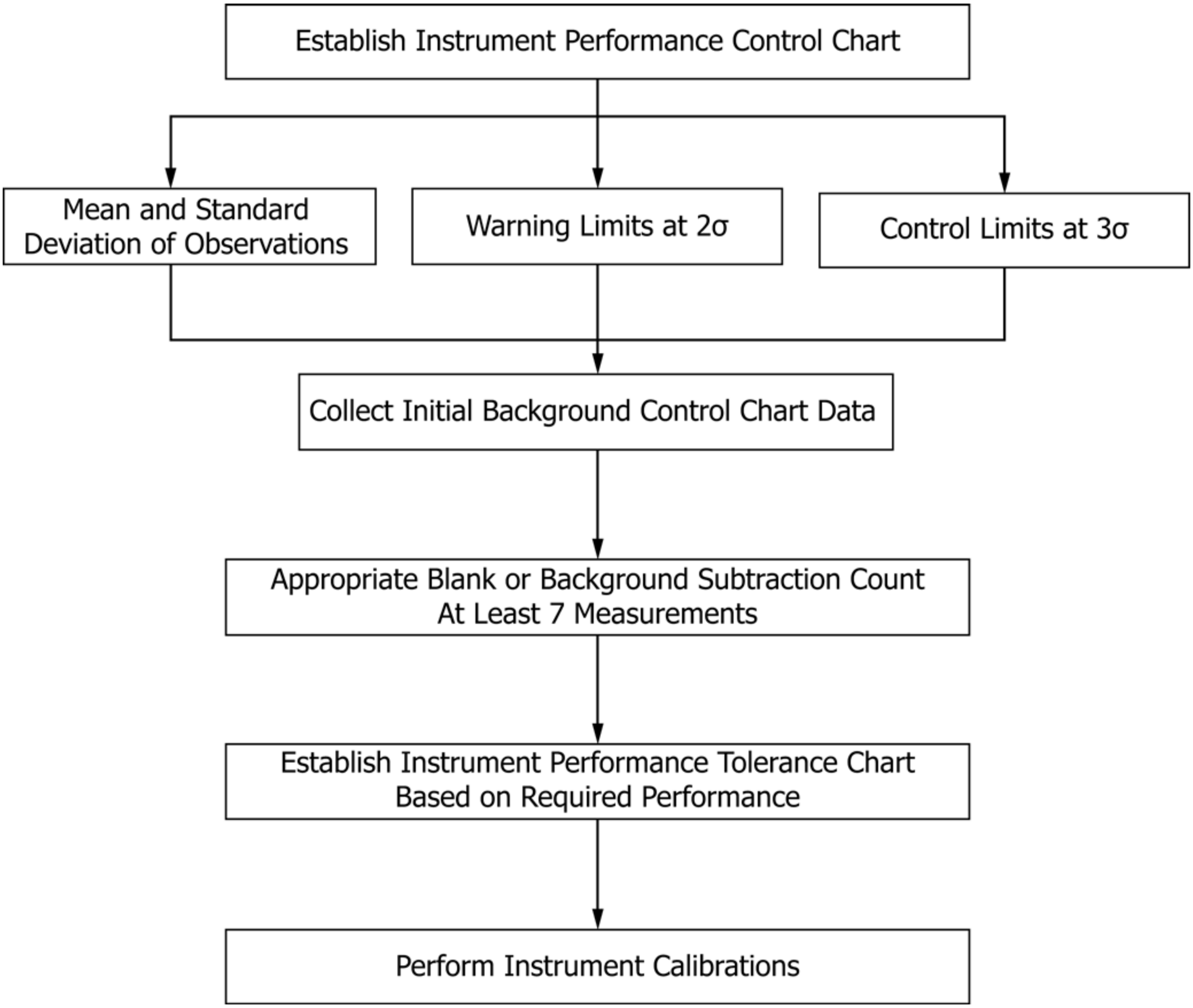

FIG. A1.2 Establish Instrument Performance Control Chart Work Flow Chart

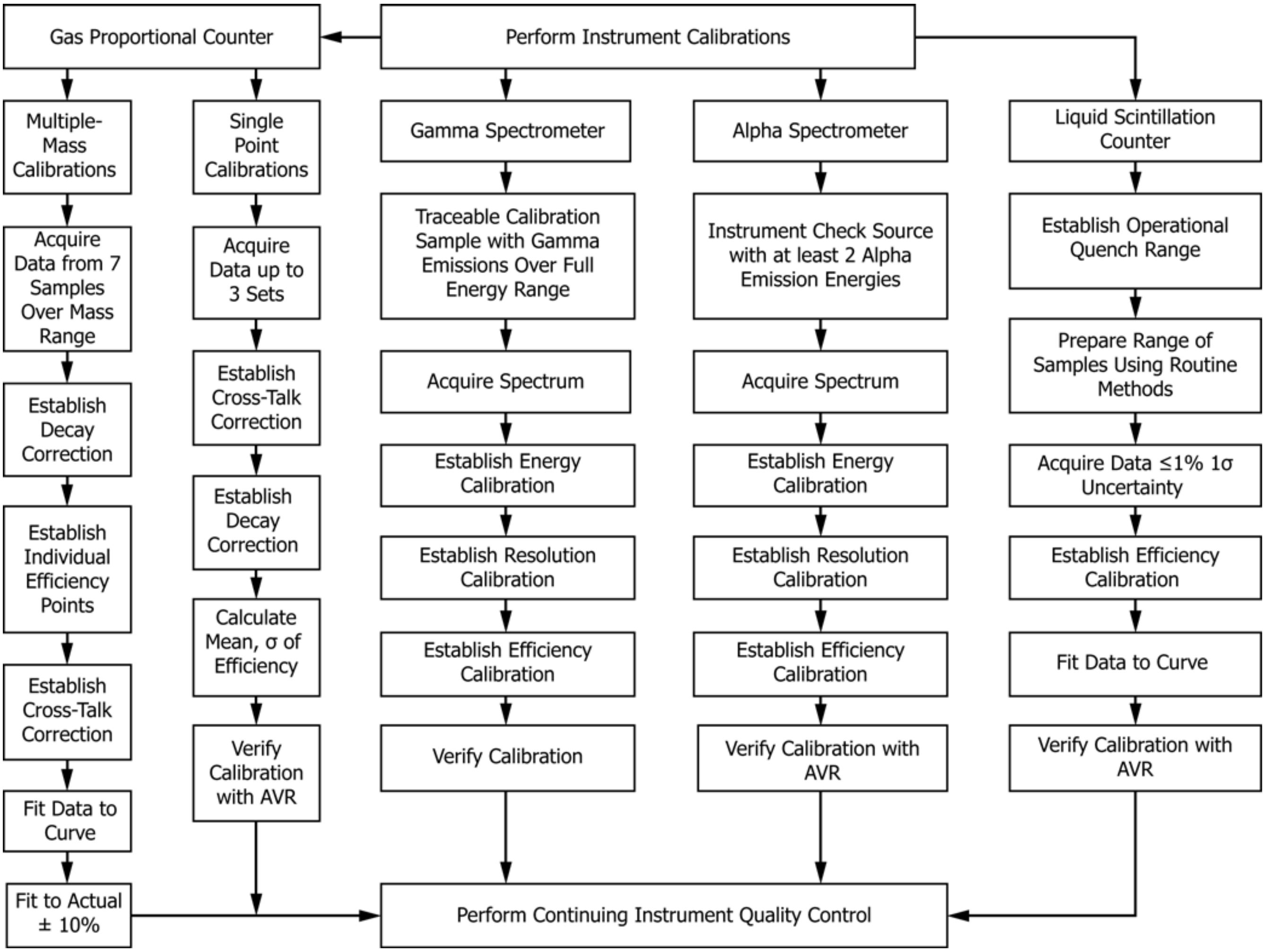

FIG. A1.3 Perform Instrument Calibrations Work Flow Chart

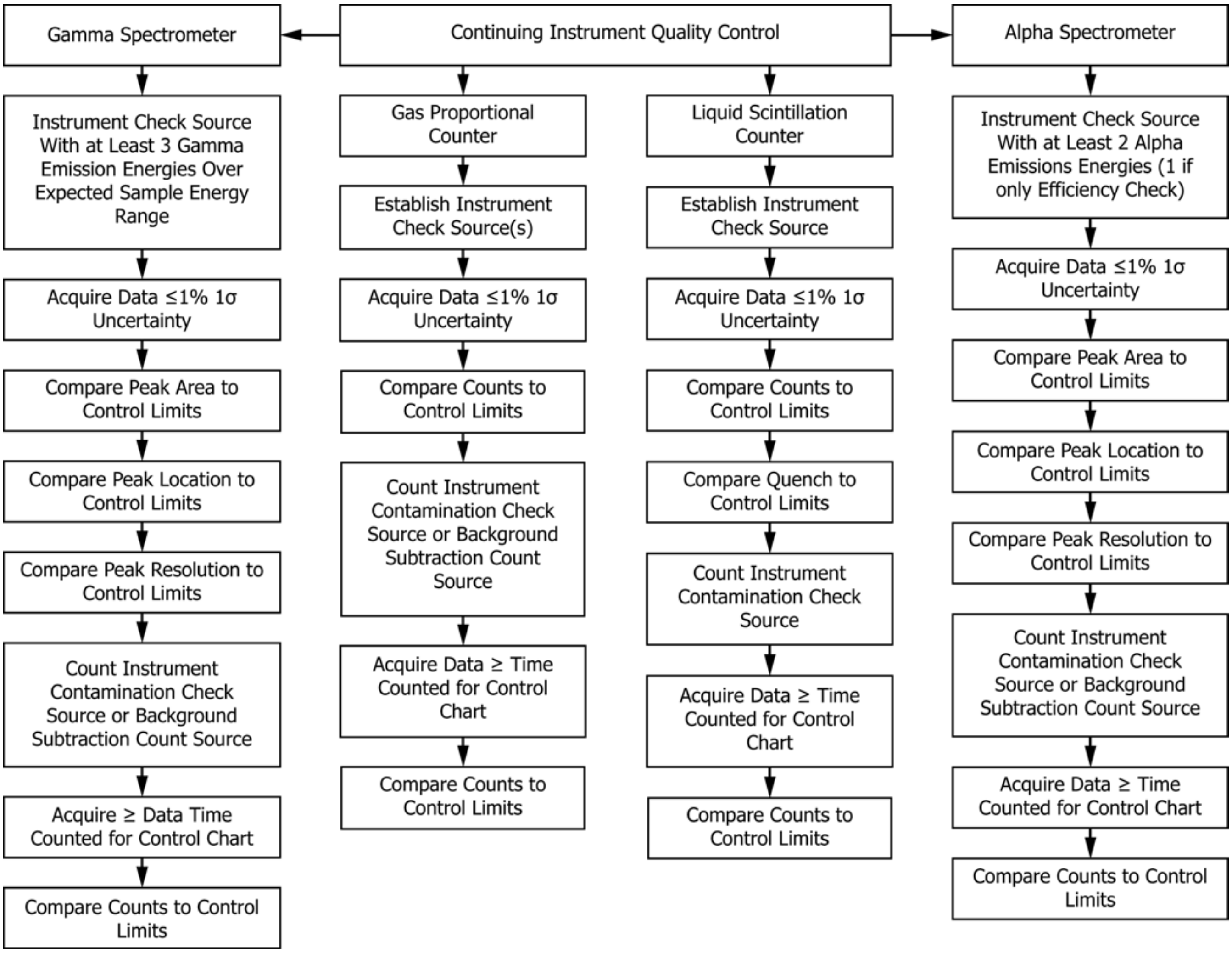

FIG. A1.4 Continuing Instrument Quality Control Work Flow Chart

APPENDIXES

(Nonmandatory Information)

X1. GROSS ALPHA CALIBRATION USING Am-241

X1.1 An ^{241}Am CS was added to the amount of water residue needed to achieve the desired weight. The solution was evaporated to dryness, transferred to a 38.1-mm (1½ -in.) planchet and counted on a windowless gas proportional counter. (See Fig. X1.1.)

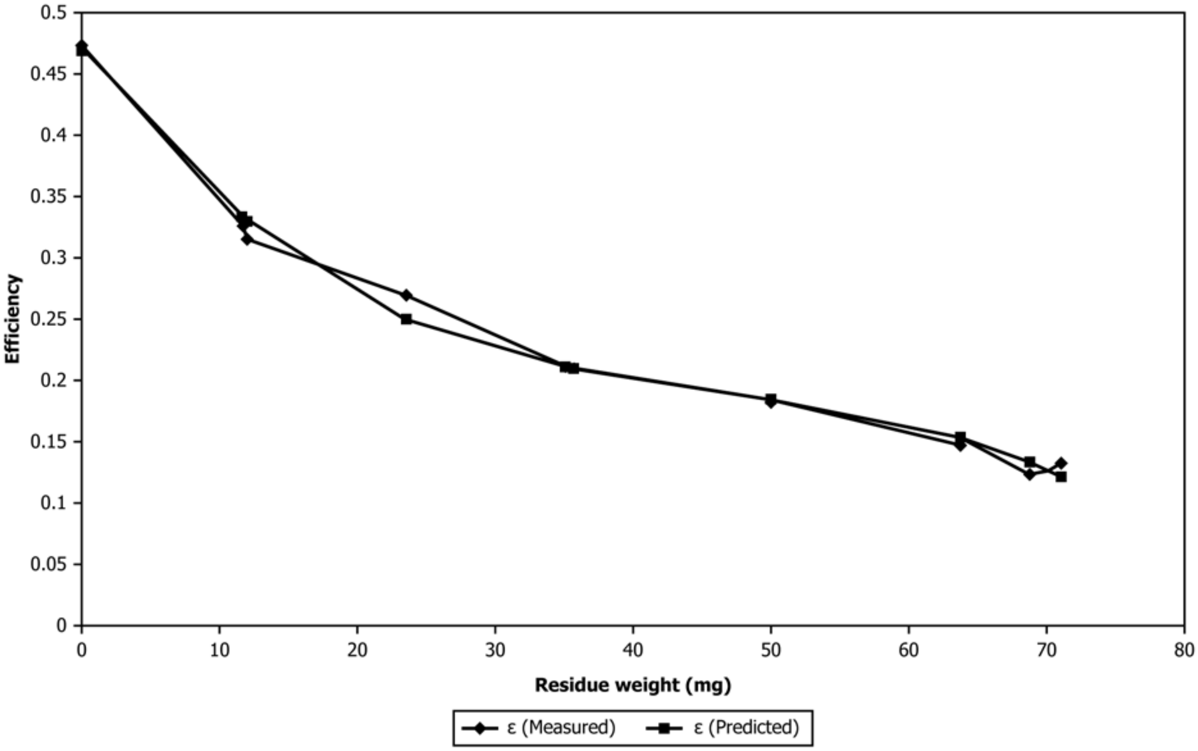

FIG. X1.1 Gas Proportional Counter Calibration

X2. GAS PROPORTIONAL COUNTER—ALPHA TO BETA AND BETA TO ALPHA CROSSTALK

X2.1 *General Considerations*:

X2.1.1 Alpha-to-beta crosstalk results when interactions of alpha particles in the detector are falsely registered as beta decays. Refer to Reference (**1**) for additional information.

X2.1.2 Beta-to-alpha crosstalk results when interactions of beta particles in the detector are falsely registered as alpha decays.

X2.1.3 Crosstalk is specific to the nuclides present in a sample and depends on the method of alpha/beta separation in a counter. For commonly used alpha/beta separation by a pulse height, the alpha-to-beta crosstalk results predominantly from the secondary radiations associated with alpha decay, such as X-rays, low-energy gamma rays, conversion and Auger electrons. The alpha-to-beta crosstalk due to mass attenuation of alpha particles is small. The beta-to-alpha crosstalk is caused by too low discriminator threshold and pile-up. With proper setting of the discriminator threshold and moderate activity level, this crosstalk can be nearly completely eliminated. Both crosstalks can also be caused by a fixed timing-discriminator threshold, in systems where alpha/beta separation is achieved by a pulse shape.

X2.1.4 When calibrating for a test such as Gross Alpha or Gross Beta, or Total Radiostrontium, one assumes the presence of a specific nuclide or mixture of nuclides. Associated crosstalk corrections must be performed assuming the same radioisotopic make-up of the samples.

X2.1.5 The radioisotopic make-up of the WCS should match that of the samples as closely as practical. When the identity and make-up of nuclide(s) in the sample are well-known, very accurate crosstalk corrections are possible. However, when, the radioisotopic makeup of the sample varies from that used for the calibration, as is often the case with environmental Gross Alpha and Beta testing, crosstalk corrections, and thus the reported results, may be grossly inaccurate. This would be a known limitation of the test, a fact that should be documented and reported in association with results.

X2.1.6 Crosstalk may vary according to sample test source configuration. The composition and geometry of the sample test source and the sample test source mount, and the distance from the active volume of the detector used for calibration should match that of samples as closely as possible.

X2.1.7 If both alpha and beta analytes are being determined simultaneously (that is, Gross Alpha/Beta), it may be appropriate to establish and perform corrections for both beta-to-alpha and alpha-to-beta crosstalk.

X2.2 Results must be corrected for crosstalk whenever an STS may contain alpha and beta emitters such that the magnitude of crosstalk evaluated for expected operating conditions will produce a statistically significant impact in test results. This may be assessed by calculating the count rate attributable to crosstalk over the range of conditions expected to be encountered for the technique. If, under these conditions, the calculated crosstalk count rate exceeds the associated uncertainty in the count rate or the decision level count rate, or both, crosstalk corrections should be performed.

X2.2.1 Crosstalk calibration data may be gathered and processed simultaneously with efficiency calibrations (see Section 16) assuming that the calibration source is known to be radioisotopically pure and does not contain decay progeny which will interfere with the determination (for example, ^{238}U sources are quickly compromised by the ingrowth of beta-emitting ^{234m}Th and ^{234}Pa and should not be used for a direct determination of the alpha-to-beta crosstalk without prior purification of the uranium). The source should be the radionuclide requiring calibration but may not necessarily be a pure alpha or pure beta emitting source (for example, ^{90}Sr, ^{90}Y). The point is not necessarily to characterize the intrinsic response of the detector, but rather to produce an empirical correction registered in the minor channel in the presence of a given radionuclide in a sample.

X2.2.1.1 The alpha-to-beta crosstalk, X_α, is defined as:

$$X_\alpha = \frac{R_\beta}{R_\alpha + R_\beta} \tag{X2.1}$$

where:

R_α = respective background corrected count rate (s^{-1}) in the alpha channel, and

R_β = respective background corrected count rate (s^{-1}) in the beta channel where in both cases (R_α and R_β) the counts are measured with an alpha-emitting source.

(1) The source(s) used for alpha self-absorption or efficiency curves are generally well-suited to perform a simultaneous crosstalk calibration if they are free of beta emitting decay progeny that would interfere with the measurement of the beta signal (increase the R_β count). The geometry and the chemical and physical makeup of the source must match the samples to be measured as closely as possible.

(2) Calculate the combined standard uncertainty $u_c(X_\alpha)$, using the equation defined in the laboratory QM or with example Eq X2.3.

$$u_c(X_\alpha) = \frac{\sqrt{\dfrac{(X_\alpha \times R_\alpha)}{t}}}{R_\alpha + R_\beta} \tag{X2.2}$$

where:

$u_c(X_\alpha)$ = combined standard uncertainty of the alpha-to-beta crosstalk, and

t = duration of count.

(3) The beta-to-alpha crosstalk, X_β, is defined as:

$$X_\beta = \frac{R_\alpha}{R_\alpha + R_\beta} \tag{X2.3}$$

where:

R_α = respective background corrected count rate (s^{-1}) in the alpha channel, and

R_β = respective background corrected count rate (s^{-1}) in the beta channel where in both cases (R_α and R_β) the counts are measured with a pure beta-emitting source.

(4) The source(s) used for beta self-absorption or efficiency curves are generally well-suited to perform a simultaneous crosstalk calibration if they are free of alpha emitting progeny that would interfere with the measurement of the alpha signal. The geometry and the chemical and physical makeup of the source must match the samples to be measured as closely as possible.

(5) Calculate the combined standard uncertainty $u_c(X_\beta)$, using the equation defined in the laboratory QM or with example Eq X2.6.

$$u_c(X_\beta) = \frac{\sqrt{\dfrac{(X_\beta \times R_\beta)}{t}}}{R_\alpha + R_\beta} \tag{X2.4}$$

where:

$u_c(X_\beta)$ = combined standard uncertainty of the beta-to-alpha crosstalk, and

t = duration of count.

X2.2.1.2 The relationship between X_α and X_β is given below:

$$R_{r\alpha} = R_{d\alpha} - (R_{d\alpha} \times X_\alpha) + (R_{d\beta} \times X_\beta) \tag{X2.5}$$

(1) Eq X2.7 states that the observed alpha count rate (s^{-1} corrected for background), $R_{r\alpha}$, consists of the actual alpha count rate (s^{-1}), $R_{d\alpha}$, (the total alpha count rate (s^{-1}) in both the alpha and beta channels due to only alpha interactions), minus those alpha interactions recorded in the beta channel (alpha-to-beta crosstalk counts), plus those beta counts recorded in the alpha channel (beta-to-alpha crosstalk counts).

$$R_{r\beta} = R_{d\beta} - (R_{d\beta} \times X_\beta) + (R_{d\alpha} \times X_\alpha) \tag{X2.6}$$

(2) Eq X2.8 is the equivalent of Eq X2.7 for beta counts. It states that the recorded beta count rate (s^{-1}, corrected for background), $R_{r\beta}$, consists of the actual beta count rate (s^{-1}), $R_{d\beta}$, (the total beta count rate (s^{-1}) in both the alpha and beta channels due to only beta interactions), minus those beta interactions recorded in the alpha channel (beta-to-alpha crosstalk counts), plus those alpha counts recorded in the beta channel (alpha-to-beta crosstalk counts).

(3) Solving the equations simultaneously for $R_{d\alpha}$ and $R_{d\beta}$ gives:

$$R_{d\alpha} = \frac{R_{r\alpha} - X_\beta \times (R_{r\alpha} + R_{r\beta})}{1 - X_\alpha - X_\beta} \tag{X2.7}$$

$$R_{d\beta} = \frac{R_{r\beta} - X_\alpha \times (R_{r\alpha} + R_{r\beta})}{1 - X_\alpha - X_\beta} \tag{X2.8}$$

(4) It should be noted that the values for $R_{r\alpha}$ and $R_{r\beta}$ represent the net count rate (s^{-1}) for their respective channels (that is, the gross count rate (s^{-1}) for each channel minus the respective background count rate (s^{-1})).

X2.2.1.3 The associated combined standard uncertainty for the net, crosstalk-corrected alpha count rate (s^{-1}), $u_c(R_{d\alpha})$, is:

$$u_c(R_{d\alpha}) = \frac{[u^2(X_\alpha)R_{d\alpha}^2 + u^2(X_\beta)(R_{d\alpha} - R_{r\alpha} - R_{r\beta})^2 + u^2(R_{r\alpha})(1 - X_\beta)^2 + u^2(R_{r\beta})X_\beta^2]^{1/2}}{[1 - X_\alpha - X_\beta]} \tag{X2.9}$$

(1) Similarly, the associated combined standard uncertainty

for the net, crosstalk-corrected beta count rate (s^{-1}), $u_c(R_{d\beta})$, is:

$$u_c(R_{d\beta}) = \frac{\left[u^2(X_\beta)R_{d\beta}^2 + u^2(X_\alpha)(R_{d\beta} - R_{r\alpha} - R_{r\beta})^2 + u^2(R_{r\beta})(1 - X_\alpha)^2 + u^2(R_{r\alpha})X_\alpha^2\right]^{1/2}}{\left[1 - X_\alpha - X_\beta\right]} \tag{X2.10}$$

(2) Since crosstalk factors vary with radionuclide, additional uncertainty components may be applied if the identities of the alpha and beta emitting radionuclides are unknown.

X2.2.2 Final crosstalk values are determined as follows:

X2.2.2.1 If a single point calibration is being performed, calculate the mean and standard deviation (see Practice D4375) of the crosstalk values obtained from the three calibration samples in Eq X2.1 or Eq X2.4. The standard deviation may be combined with other standard uncertainties to determine the standard uncertainty of the crosstalk. The estimated relative standard deviation (coefficient of variation) should be compared to and meet limits established in the laboratory QM but should not exceed 25 %.

X2.2.2.2 If a self-absorption or attenuation curve is performed, calculate and plot the crosstalk values calculated for each WCS against its residue mass.

X2.2.2.3 Determine the best-fit equation of each crosstalk calibration curve using all of the crosstalk values 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. 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.

X2.2.2.4 Compare the observed crosstalk value to the crosstalk value calculated from the calibration curve at that test source residue mass. A variety of software packages, including basic spreadsheet applications, are available to optimize the calibration coefficients in order to minimize the difference between observed values and the fitted values obtained from the calibration.

X2.2.2.5 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 crosstalk value for each data point should deviate 15 % or less of the value calculated from the fitted curve for that given sample test source residue mass. If all data points meet the acceptance criterion, the curve is acceptable and the analyst should proceed to X2.2.3. Single data points that exceed the ±15 % acceptance criterion 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. In no case should more than 20 % of the data points be removed. Decisions to exclude outliers should be clearly documented. After removing the selected data points the analyst must return to the beginning of X2.2.2.3, 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.

X2.2.2.6 If the difference of a datum value from the mean of the paired values (paired WCS) is greater than 15 %, delete the data pair. Return to X2.2.2.3 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 15 % of the value of the fitted curve. The loss of one replicate of a pair should not be cause for rejecting the calibration.

X2.2.2.7 The standard deviation of the fitted curve calculated by the regression software in X2.2.2.3 is used with the other known uncertainties to produce the standard uncertainty of the crosstalk calibration curve. The crosstalk calibration uncertainty shall be included in the combined standard uncertainty of the test source sample result.

X2.2.3 Count data generated for purposes of calibration verification in Section 16 should be evaluated to verify the adequacy of crosstalk calibrations, where applicable.

X2.2.3.1 If a single point calibration is performed, the observed crosstalk value for the calibration verification sample 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. If the sample result does not agree with these limits, the cause should be determined and corrected and the test performed again, or the system should be recalibrated.

X2.2.3.2 If a self-absorption or attenuation curve is performed, the observed crosstalk value for the three calibration verification samples 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. If the sample result does not agree with these limits, the cause should be determined and corrected and the test performed again, or the system should be recalibrated.

X2.2.3.3 A blank sample should also be analyzed with the verification sample(s). Compare the blank sample result to the laboratory's performance criteria as defined in its QM.

X2.2.3.4 When the criteria in X2.2.3.1 or X2.2.3.2, and X2.2.3.3 are met, the crosstalk calibration is acceptable. The crosstalk constant determined in X2.2.3.1 or the crosstalk calibration curve determined in X2.2.3.2 is used for calculation of sample test source results.

X3. MARINELLI BEAKER CALIBRATION USING MIXED STANDARD

X3.1 The Marinelli beaker standard used was a commercially prepared mixed gamma standard. It was counted on a p-type germanium detector to accumulate 10 000 counts or more per photopeak. The relative efficiency at 1.33 MeV, ^{60}Co, was 23 %. (See Fig. X3.1.)

X4. Ni-63 CALIBRATION

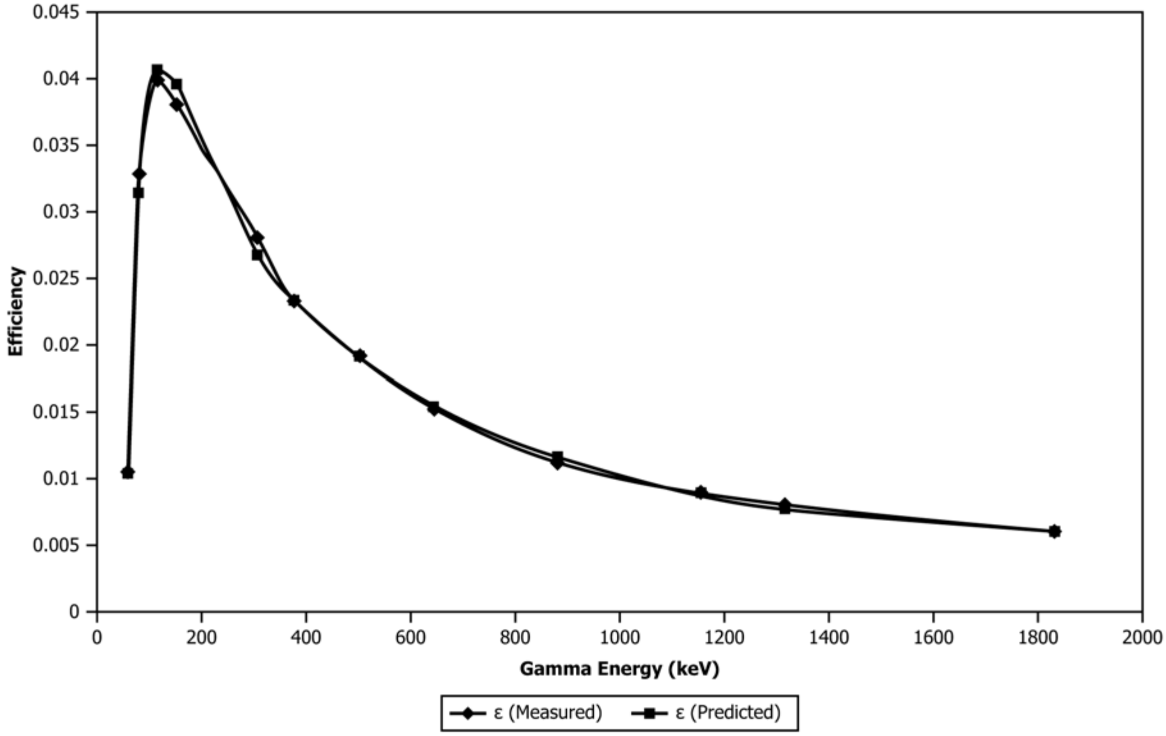

FIG. X3.1 Germanium Detector Marinelli Beaker Efficiency

X4.1 A ^{63}Ni CS, nickel carrier and an appropriate amount of quenching agent were diluted to 5 mL with reagent water to which was added 15 mL liquid scintillation cocktail. The solution was counted with a liquid scintillation counter. (See Fig. X4.1).

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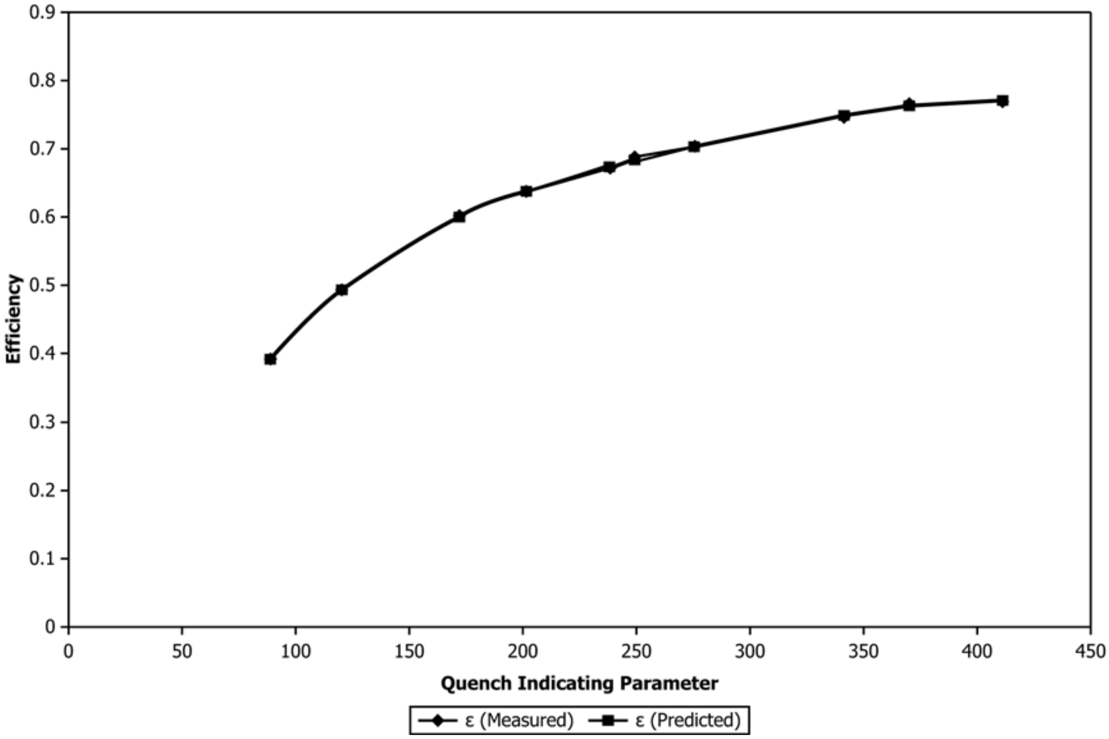

FIG. X4.1 Liquid Scintillation Counter Calibration

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