Designation: ~~D7282 – 14~~ D7282 – 21

Standard Practice for ~~Set-up,~~Setup, 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~~ setup, 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~~ Setup establishes 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.~~instrument—for example, voltage or discriminator settings. Calibrations determine the instrument's response characteristics—for example, its counting efficiency or gain. Quality control ensures that the performance of the instrument remains acceptable for its intended use and consistent with the performance at the time of calibration.

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

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

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

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1129 Terminology Relating to Water
D3648 Practices for the Measurement of Radioactivity
D7283 Test Method for Alpha and Beta Activity in Water By Liquid Scintillation Counting
D7902 Terminology for Radiochemical Analyses
~~D4375~~E2586 Practice for ~~Basic Statistics in Committee D19 on Water~~Calculating and Using Basic Statistics ~~(Withdrawn 2018)~~

[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.
Current edition approved ~~June 1, 2014~~May 15, 2021. Published ~~July 2014~~December 2021. Originally approved in 2006. Last previous edition approved in ~~2006~~2014 as D7282 – ~~06.~~14. DOI: ~~10.1520/D7282-14.~~10.1520/D7282-21.

[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.

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~~definitions of ~~other~~ terms used in this ~~practice,~~standard, refer to ~~Terminology~~Terminologies D1129 and D7902 and Practice E2586.

3.2 *Definitions of Terms Specific to This Standard:*

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

3.2.1.1 *Discussion*—

See Eq ~~10~~14 in ~~16.2.13~~16.2.15.

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

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

3.2.3.1 *Discussion*—

Instrument calibrations may include calibrations for counting efficiency, gain, and resolution.

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

3.2.4.1 *Discussion*—

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

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

3.2.5.1 *Discussion*—

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

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

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

3.2.8 *instrument check source (ICS), n*—a radioactive source, not necessarily traceable to ~~a national standards body,~~ any standard, that is used to ~~confirm the continuing satisfactory operation of an~~ test the response of a nuclear instrument.

[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.

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

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

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

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

3.2.12.1 *Discussion*—

See Eq ~~7~~12 in ~~16.2.11~~16.2.13.

~~3.2.11 *mean, n*—see Practice D4375.~~

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

3.2.13.1 *Discussion*—

See Eq ~~4~~10 in ~~16.2.9~~16.2.11.

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

3.2.15 ~~*national standards body,*~~*normalized residual, ζ_i, n*—~~an organization such as National Institute of Standards~~quotient of a *residual,* e_i, and ~~Technology (NIST) or another~~its combined standard uncertainty, ~~*similar*~~ u_c ~~national (*body*~~ e_i ~~that provides standards traceable to BIPM (Bureau International des Poids et Mesures (International Bureau of Weights and Measures)).~~)

3.2.15.1 *Discussion*—

~~Traceability is~~See Appendix X5 ~~accomplished with guidance from ANSI N42.22.~~for the calculation and use of ζ_i.

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

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

3.2.17.1 *Discussion*—

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

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

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

3.2.19.1 *Discussion*—

See Practice ~~D4375~~E2586 ~~and 16.2.7~~.

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

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

3.2.21 *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.~~or sample aliquot prepared or configured for measurement of its emitted radiation.

3.2.22 ~~*standard deviation,*~~*tolerance limit, n*—~~see Practice~~a limit ~~D4375~~.established to evaluate the acceptability of a monitored process parameter.

3.2.23 *working calibration source (WCS), n*—a calibration source (see 3.2.4~~), including those~~) diluted or prepared by ~~chemical procedure for the purpose of calibrating an instrument.~~the laboratory from radioactive reference materials.

3.3 *Acronyms:*

3.3.1 *ADC*—~~analog-digital conversion~~analog-to-digital converter

3.3.2 *AVR*—acceptable verification ratio

3.3.3 *BIPM*—Bureau International des Poids et ~~Mesures. Also known as~~ Mesures (English: International Bureau of Weights and ~~Measures.~~Measures)

3.3.4 *BSC*—background subtraction count

3.3.5 ~~*CS*—~~*CCS*—certified calibration source

3.3.6 *DF*—decay factor

3.3.7 *FWHM*—full width at half maximum

3.3.8 *ICC*—instrument contamination check

3.3.9 *ICS*—instrument check source

3.3.10 *KV*—known value

3.3.11 *LCS*—liquid scintillation counter

3.3.12 *MV*—measured value

3.3.13 *MQO*—measurement quality objective

3.3.14 *NIST*—National Institute of Standards and Technology

3.3.15 *NMI*—National Metrology Institute

3.3.16 *QC*—quality control

3.3.17 *QM*—quality manual

3.3.18 *RSD*—relative standard deviation

3.3.19 *STS*—sample test source

3.3.20 *WCS*—working calibration source

4. Summary of Practice

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

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

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

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

5. Significance and Use

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

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

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

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

6. Hazards

6.1 The ~~vendor supplied~~ 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~~aware of hazards involved with all materials and processes employed, and comply with any and all applicable health and safety procedures, plans, and regulations. Safety ~~Data Sheets~~data sheets are a source of information.

INSTRUMENT ~~SET-UP~~SETUP

7. Scope

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

8. Significance and Use

8.1 Successful ~~set-up~~setup of an instrument and its subsequent routine use depend, at least in ~~part;~~part, on how well the ~~manufacturer's~~manufacturer's instructions are written and followed. Thus, the ~~manufacturer's~~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~~ Setup Procedures

9.1 *Gas Proportional ~~Counting Initial Instrument Set-up:~~ Counter Setup:*

9.1.1 Upon initial ~~set-up,~~setup, after major repair or service, or when QC results indicate the need to ~~reestablish~~adjust operating parameters for an instrument, measure a suitable ~~calibration source (that is, ICS or WCS)~~ radioactive source as specified in the laboratory QM ~~and/or manufacturer's~~or manufacturer's protocol to confirm that the instrument responds according to QM or ~~manufacturer's~~manufacturer's specifications. The instrument ~~set up~~ setup 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~~ Set the appropriate instrument ~~operational~~operating parameters for the intended measurements. For example, acquire voltage plateaus and establish the alpha or beta, or both, plateau operating voltages, and alpha or beta, or both, discriminator settings (that is, adjust for crosstalk). ~~Instrument set up and~~ The instrument configuration should be optimized for the intended applications. For example, when measuring evaporated sample solids deposited in a 50.8 mm (2-inch) diameter planchet, it may be desirable to perform voltage plateaus and optimize discriminator settings using a ~~distributed source or a specific radionuclide~~ geometry and radionuclide similar to those that will be used for subsequent measurements (for example, a ~~50.8-mm (2-in.)~~50.8 mm diameter ^{230}Th source ~~as opposed to~~ rather than 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~~ Po). If setup procedures deviate from those recommended by the manufacturer, the ~~configuration and procedure to be used~~ procedures shall be specified in detail in the laboratory QM. Operating parameters should be ~~established~~set to produce consistency in ~~detection characteristics~~ performance across multiple detectors used for a common application. When the instrument ~~operational~~operating parameters ~~are satisfactorily~~have been established, record the "as-left" instrument settings for future reference.

9.2 *~~Gamma Spectrometry Initial Instrument Set-up:~~ Gamma-Ray Spectrometer Setup:*

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

9.2.2 If the instrument being configured has previously been used to generate sample test source results, the "as-found" instrument settings (that is, detector bias, amplifier gain, ~~analog-to- digital~~ 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~~Set the energy range for the spectrometer to include all gamma emission energies of interest to the laboratory. Adjust the amplifier gain, ADC range, or equivalent digital spectrometer ~~settings,~~settings to ~~establish~~produce the desired energy per channel relationship. When the instrument ~~operational~~operating parameters are satisfactorily established, record the instrument settings for future reference.

9.3 ~~*Alpha Spectrometry Initial Instrument Set-up:*~~ *Alpha-Particle Spectrometer Setup:*

9.3.1 Upon initial ~~set-up,~~setup, after major repair or service, or when QC results indicate the need to ~~reestablish~~adjust operating parameters for an instrument, measure a suitable ~~calibration source (that is, ICS or WCS)~~ radioactive source as specified in the laboratory QM ~~and/or manufacturer's~~or manufacturer's protocol to confirm that the instrument responds according to QM or ~~manufacturer's~~manufacturer's specifications (for example, bias voltage setting, ~~full-width at half maximum~~ full-width-at-half-maximum resolution, detector efficiency and background). The instrument ~~set-up~~setup 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~~operating parameters are satisfactorily established, record the instrument settings for future reference.

9.4 *Liquid Scintillation* ~~*Counting Initial Instrument Set-up:*~~ *Counter Setup:*

9.4.1 Upon initial ~~set-up,~~setup, after major repair or service, or when QC results indicate the need to ~~reestablish~~adjust operating parameters for an instrument, measure a suitable ~~calibration source (that is, ICS or WCS)~~ radioactive source as specified in the laboratory QM ~~and/or manufacturer's~~or manufacturer's protocol to confirm that the instrument responds according to QM or ~~manufacturer's~~manufacturer's specifications (for example, detector efficiency, background for region of interest for beta or alpha applications). The instrument ~~set up~~ setup 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~~Set the instrument ~~operational~~operating parameters for the intended measurements according to the ~~manufacturer's~~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~~operating parameters are satisfactorily established, record the instrument settings for future reference.

INITIAL INSTRUMENT QUALITY CONTROL ~~TESTING~~

10. Scope

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

11. Significance and Use

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

11.2 ~~Quality~~Instrument 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~~results, a tolerance ~~limit~~chart may be used. The laboratory QM shall indicate the appropriate tool, ~~quality~~ control chart or tolerance ~~limit,~~chart, for monitoring each ~~continuing instrument quality control~~ performance parameter.

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

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

12. Establishing the Control Chart

12.1 Using the appropriate ICS or ICC, perform at least 7 measurements of the ~~particular instrument quality control parameter,~~performance parameter to be monitored, 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~~ These measurements may be performed sequentially over a short period of ~~time,~~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.~~ 24 h period. In each case, the ICS or ICC ~~being used~~ should be removed from the instrument between measurements and re-inserted so that the control chart reflects variability in sample positioning.

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

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

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

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

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

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

$$\hat{C} = \frac{\sum_{i=1}^{n} C_i}{\sum_{i=1}^{n} DF_i} \tag{1}$$

$$\hat{\mu}_C = \hat{C} \cdot DF \tag{2}$$

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

where:

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

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

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

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

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

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

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

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

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

12.5 ~~Calculate the mean and standard deviation (see Practice D4375) of the measured parameter.~~ Create a control chart with the ~~measurement~~observed result on the vertical axis and the observation number ~~(or measurement date)~~ or date on the horizontal axis. Draw a horizontal line or a sloping (decay-corrected) curve on the chart to represent the predicted mean of the ~~measurement value. Additional horizontal lines set as "warning limits" and "control limits,"~~ observed values. Draw lines or curves for the control limits at three standard deviations above and below the mean. Additional lines or curves for "warning limits" should also be drawn, typically at two ~~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.~~ standard deviations above and below the mean. The quality control data should be evaluated to ~~establish that it is normally distributed, although very low counts are more likely to~~check that they follow the expected distribution—for example, Poisson or normal—and that there are no outliers. Appendix X6 ~~have a Poisson distribution. In this case the data could be tested against the Poisson model.~~ describes procedures that may be used to test the assumption of Poisson counting statistics. Reference (**1**) includes a discussion for pursuing ~~root cause~~ root-cause analysis of excursions (departures from the expected condition). Practices D3648 and Reference (**1**), Chapter 18, present information on the preparation and interpretation of control charts.

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

13.1 The purpose of ~~the~~ tolerance ~~limit~~charts is to ~~provide a comparison of measured~~ compare observed instrument performance to acceptable ~~instrument performance, and~~ performance limits. A tolerance may be expressed as a percent (%) deviation of ~~a mean performance measure.~~ an observed parameter from a nominal value, which might be an estimated mean, calibrated value, or other assumed target value. There may be different tolerances for values above and below the nominal value. The basis for the ~~tolerance limit~~ tolerances 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~~Tolerance limits differ from control limits in that they are not based on statistical measures, but instead are based on acceptance criteria appropriate to the method and scope of work. (The QM shall define the basis and manner by which tolerance limits are established for each performance ~~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.~~ parameter). A tolerance chart, similar to a control chart, is a graphical tool that can be used to evaluate instrument performance and trending of instrument parameters. In Reference (**1**), Chapter 18, several examples are given for the use of tolerance limits, one of which is monitoring the resolution of a high-purity germanium detector. In addition, it may be appropriate to establish "warning limits" when using a tolerance chart to ~~insure~~ensure appropriate actions are taken before a tolerance limit is crossed.

13.3 For each performance parameter to be charted, establish the tolerance ~~limit.~~limits. The tolerance ~~limit~~limits should be selected ~~such~~so 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~~ Account for radioactive decay as appropriate when evaluating parameters based on short-lived ~~radionuclides it may be necessary to account for radioactive decay when evaluating quality control data.~~radionuclides.

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

13.5 Create a tolerance chart with the ~~measurement~~observed result on the vertical axis and the observation number ~~(or~~

~~measurement date)~~ or date on the horizontal axis. Draw a horizontal line on the chart to represent the ~~mean~~ nominal value of the ~~measurement values,~~observed parameter, and draw ~~a~~ horizontal ~~line at~~lines for the tolerance ~~limit~~limits above and below the ~~mean. These lines are called "tolerance limits."~~nominal value. It can also be informative to draw horizontal lines for the 3-sigma statistical control limits, although these 3-sigma limits will not be used to accept or reject observed parameter values. The 3-sigma limits may be used instead to provide early warnings of trends that might eventually impact data quality.

13.6 Many instruments are provided with operation and analysis software which may include performance check capabilities. It is not necessary that the software use exactly the same terminology or graphical features. However, if the software is to be used for continuing instrument tolerance ~~measurements, it is necessary that the software~~ checks, it must be able to compare individual ~~performance measurements with~~ observations to the established tolerance limits and indicate ~~an out-of-tolerance condition. Stand-alone~~ out-of-tolerance conditions. Standalone 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.~~sources. The calibration ~~source~~sources shall have ~~a certified value (with uncertainty) that is traceable to a national standards body.~~values (with uncertainties) that are traceable to the SI via a national metrology institute. When working calibration sources are used, they shall be prepared from certified SI-traceable radionuclide standards.

15. Significance and Use

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

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

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

16. Gas Proportional Counter Instrument Calibrations

16.1 Refer to the guidance in Sections 7 to 13 for counting the ICS and ICC at ~~initial~~ instrument ~~set-up~~setup in preparation for ~~counting calibration sources (CS or WCS).~~ calibration. For those instruments already in use, count the ICS and ICC samples as prescribed in Section 22.

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

16.2.1 Instructions for a ~~single point~~ single-point efficiency calibration of a gas proportional counter are provided below. A ~~single point~~ single-point efficiency is used when the ~~sample test source residue mass varies little and the~~ efficiency change is ~~less than ±5 %~~ negligible over the expected mass range for the ~~test or there is a near-massless sample test source.~~test.

16.2.2 ~~To control the potential bias from a non-representative sample test source,~~ The guidance below assumes the use of working calibration sources (WCSs). To control possible bias due to non-representative calibration sources, the preparation method of the ~~calibration sources should be~~WCSs should produce sources that are as equivalent as ~~practical to that used to prepare~~ practicable to the sample test sources. Since ~~chemical processes are nearly always used to prepare calibration sources,~~ the preparation typically involves chemical procedures, with opportunities for loss of analyte, it is essential that the ~~process be carried out very~~procedure be designed and performed carefully to ensure its quantitative nature and ~~that measurements~~ to preserve traceability to the ~~appropriate national standard.~~SI. WCSs shall be prepared from certified SI-traceable radionuclide standards.

16.2.3 A minimum of three ~~calibration samples~~ WCSs (or one CCS) 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.~~ WCSs. The blank sample result should be compared to the ~~laboratory's~~ performance criteria ~~as defined in its~~ stated in the laboratory's QM.

16.2.5 ~~Select a WCS whose activity is sufficient~~ The activity of each WCS should be selected to produce a count rate not exceeding 5000 counts per second (s^{-1}). It is essential that the count rate of the WCS be ~~such~~ low enough to avoid instrument dead time that ~~will~~would 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~~manufacturer's specifications.

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

16.2.7 Correct the WCS activity ~~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~~7.

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

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

where:

~~ε_{WCS} = single point efficiency of WCS (counts per second per becquerel (s^{-1} Bq^{-1}),~~
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_a = count rate (s^{-1}) of WCS,
~~R_b = count rate (s^{-1}) of instrument background,~~
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).

16.2.7.1 Eq ~~1~~7 accounts for the total efficiency of the radionuclide even when the probability of alpha or beta emission per ~~decay, stated as a decimal fraction~~ decay is less than ~~1.0~~1.0 (less than 100 %).

16.2.7.2 ~~Calculate the combined standard uncertainty~~ Calculate the combined standard uncertainty $u_c(\varepsilon_{WCS})$, using the equation defined in the laboratory QM or with example Eq ~~2~~8.

$$u_c(\varepsilon_{WCS}) = \sqrt{\frac{\frac{R_a}{t_a} + \frac{R_b}{t_b}}{A_{WCS}^2 \times Y_{WCS}^2 \times DF^2} + \varepsilon_{WCS}^2 \times \left(\frac{u^2(A_{WCS})}{A_{WCS}^2} + \frac{u^2(Y_{WCS})}{Y_{WCS}^2} \right)} \quad (8)$$

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

where:

~~$u_c(\varepsilon_{WCS})$ = the combined standard uncertainty of the single point efficiency ε_{WCS},~~
$u_c(\varepsilon_{WCS})$ = 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~~

$u(A_{WCS})$ = the standard uncertainty of A_{WCS},
~~$u(Y_{WCS})$ = the standard uncertainty of Y.~~
$u(Y_{WCS})$ = the standard uncertainty of Y_{WCS}, and
φ_G = relative standard deviation of the efficiency due to source-to-source variability.

NOTE 2—The other ~~terms~~symbols are as defined for Eq ~~1~~7.

16.2.7.3 Correction for decay during counting may be made by multiplying DF by the value, DF_a, obtained using Eq ~~3~~9.

$$DF_a = \frac{1 - e^{-\lambda t_a}}{\lambda t_a} \tag{9}$$

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

where:

λ = decay constant of the radionuclide, and
t_a = duration of count.

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

16.2.8 ~~The uncertainty of the efficiency measurement is combined with other associated standard uncertainties to determine the efficiency calibration uncertainty. See Practice~~See Appendix X5 ~~D4375~~for guidance on 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~~ calculation of a weighted average and its uncertainty, and for assessing the fit of the calibration data. The total calibration uncertainty shall be included in the combined standard uncertainty of ~~the~~each sample result.

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

16.2.10 ~~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~~ stated in the laboratory QM.~~16.2.5~~.

16.2.11 Calculate the verification sample activity, MV, using the equation defined in the laboratory QM or with example Eq ~~4~~10.

$$MV = \frac{R_a - R_b}{\varepsilon \times Y \times DF} \tag{10}$$

$$MV = \frac{R_a - R_b}{\varepsilon \cdot Y \cdot DF} \tag{10}$$

where:

MV = measured value (Bq) of the verification sample,
~~R_a = count rate (s^{-1}) of 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),~~
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),~~