Radiochemistry Expert Committee (REC)
Meeting Summary

April 26, 2017

1. Roll Call and Minutes:

Bob Shannon, Chair, called the meeting to order at 1:00 pm Eastern on April 26, 2017 by teleconference. Attendance is recorded in Attachment A – there were 10 members present. Associates: Jim Chambers, Brian Miller, Carolyn Wong and Joe Pardue.

The March 22, 2017 minutes were reviewed by email and posted on the website.

2. Assessment Checklist

Larry commented that the Checklist has been completed and is now being reviewed. Items 95-115 complete the checklist. They are the new entries that need to be reviewed by the committee. Larry thinks this section is almost verbatim from the Module and is pretty straightforward.

Bob asked that everyone look at these new entries and give comments to Larry in two weeks. If there are no big concerns, the committee can finalize the checklist at the May meeting.

3. Small Laboratory Handbook (SLH)

Dave sent a revised version of the SLH to the committee by email. Dave got comments from Carolyn and Bob. He turned off track changes from the previous version and turned it back on for the most recent changes. The changes being tracked are only the changes since the last meeting.

Carolyn noted that a comma is needed in 1.5.4.

Candy asked if the document will go through one final review after the committee thinks it is done. Bob noted that this will happen and Ilona will go through an editorial review also.

The committee reviewed all red text in the document and made updates as appropriate. The committee started with Section 1.5.2.

1.5.2 - In Keypoints – change capability to validation.

Bob noted that Keith was one of the authors of a document EPA just published on the topic of radiochemistry detection limits and verification of detection limits. This was published

this month. This is taking the place of the MDLs which labs have long run but were statistically inappropriate. Keith thinks it is a guidance document. The text was was pulled out of the RAD ATP document. It presents the calculations for DL and chi-squared tests for verifying that the SDWA required detection limit can be met.

The committee reviewed and tweaked the Draft of the SLH down to section 1.7.2. Changes were made directly into the document and the document will be distributed after the meeting for everyone to review. Changes are noted in Attachment D.

It has been a very productive meeting. If this level of effectiveness of review can be maintained, Bob sees that the SLH can be finished soon. The committee decided to make the May meeting earlier to work on the SLH. Ilona cannot make this meeting date, but the meeting will be recorded to prepare minutes from.

Bob asked that everyone continue to do a high level review by email.

4. New Business

None.

5. Action Items

A summary of action items can be found in Attachment B.

6. Next Meeting and Close

The next meeting is scheduled for May 10, 2017 at 1pm Eastern.

A summary of action items and backburner/reminder items can be found in Attachment B and C.

The meeting was adjourned at 2:36pm Eastern.

Attachment A
Participants
Radiochemistry Expert Committee

Members	Affiliation		Contact Information	
			Phone	Email
Bob Shannon (Chair) (2019) **Present**	QRS, LLC Grand Marais, MN	Other	218-387-1100	BobShannon@boreal.org
Tom Semkow (Vice Chair) (2019) **Present**	Wadsworth Center, NY State DOH Albany, NY	AB	518-474-6071	thomas.semkow@health.ny.gov
Sreenivas (Vas) Komanduri (2019) **Present**	State of NJ Department of Environmental Protection Trenton, NJ	AB	609-984-0855	Sreenivas.Komanduri@dep.state.nj.us
Marty Johnson (2019) **Present**	US Army Aviation and Missile Command Nuclear Counting Redstone Arsenal, AL	Lab	865-712-0275	Mjohnson@tSC-tn.com
Dave Fauth (2018) **Present**	Consultant Aiken, SC	Other	803-649-5268	dj1fauth@bellsouth.net
Keith McCroan (2018) **Present**	US EPA ORIA NAREL, Montgomery AL	Lab	334-270-3418	mccroan.keith@epa.gov
Larry Penfold (2018) **Present**	Test America Laboratories, Inc; Arvada, CO	Lab	303-736-0119	larry.penfold@testamericainc.com
Ron Houck (2018*) **Present**	PA DEP/Bureau of Laboratories	AB	717-346-8210	rhouck@pa.gov
Yoon Cha (2020) **Present**	Eurofins Eaton Analytical	Lab	213-703-5800	YoonCha@eurofinsUS.com
Candy Friday (2020) **Present**	CdFriday Environmental, Inc.	Lab	713-822-1951	candy@fridayllc.com
Ilona Taunton (Program Administrator) **Present**	The NELAC Institute	n/a	828-712-9242	Ilona.taunton@nelac-institute.org

Attachment B

Action Items – REC

	Action Item	Who	Target Completion	Completed
75	Prepare copy of Standard annotated with summary document language.	Carolyn	On hold	
83				
84				

Attachment C – Back Burner / Reminders

	Item	Meeting Reference	Comments
5	Form subcommittee of experts in MS and other atom counting techniques to see that these techniques are adequately addressed in the radiochemistry module.	9/24/14	

The TNI Standard: Guidance for Small labs

- Generally, the activity range must include performance at zero activity since most radiochemical methods generate results that include zero activity.
- In the case of reference methods, performance data published in the method may be used in lieu of method validation at the laboratory. Where performance data is not available, or if the reference method is modified, the laboratory must generate this method performance data based on the final method used at the laboratory (e.g., by validating the method).
- Analysis of historical internal quality control data may be used to generate some or all of the performance validation data needed to satisfy validation requirements.

- The validation records must be maintained for the life of the method and be readily retrievable.

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Discussion:

- The standard requires all methods be validated including reference methods regardless if they are used within or outside the scope of the method.
- Methods as published in literature or developed by the laboratory can be used, but must be fully validated. Clients must be informed and agree with the laboratory on the selected method.
- Introduction of laboratory-developed methods should be introduced following a plan.
- The following parameters should be considered for validating in-house developed methods: detection capability, precision and bias, selectivity, repeatability and/or reproducibility, and robustness.
- Exact validation experiments should be relevant to the sample and required information.
- Validation includes specification of the requirements and scope, determination of the characteristics of the methods, appropriate tests to prove that the requirements can be fulfilled by using the method and a statement on the validity.

Examples:

1. Both reference and non-reference methods must be supported with data on the method's detection capability, precision, bias, measurement uncertainty, and selectivity. Such method validation data is required for each analyte / quality system matrix combination. Whenever a laboratory develops a method, or modifies a method to meet different data quality objectives, the new method must be validated prior to use.

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2. Use external performance testing (PT) samples to verify laboratory performance.

3. The use of non-TNI accredited PT providers is strictly for method validation purposes, and not for laboratory accreditation.

1.5.2 Detection Capability

Detection capability refers to terms commonly used in radiochemistry such as Critical Value, Minimum Detectable Activity (MDA) or the Safe Drinking Water Act (SDWA) Detection Limit. See Appendix A for information on the key term, Minimum Detectable Activity. Methods and associated MDAs will vary as implemented from laboratory to laboratory. The Standard does not specify the procedure to use to determine the Detection Capability. It is left to the laboratory to select any method that they can defend as being technically sound as long as regulatory, method, contractual, or laboratory quality system requirements are met.

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Key Points:

- The laboratory detection capability must be verified initially as part of the method validation study for each matrix.
- The laboratory detection capability must be re-verified when there is a change in the method or when there are substantial changes to the instruments used. If no changes have been made to the method or the type of instrumentation used, there is no need to reverify the detection capability.
- The laboratory is required to document the procedure used to determine detection capability.

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- The method needs to be appropriate and relevant for the intended use of the data recognizing that project-specific or client-specific requirements may be unique.
- If software is used for the detection capability it must be clearly identified. For example, the name, tracking, control, or revision numbers of commercially or laboratory developed software should be documented.

The Standard requires that the detection capability be initially determined for each analyte in each matrix. All steps of the analytical process must be included in the detection capability determination and confirmation. The procedure a laboratory uses to determine the detection capability of a method must comply with the specific requirement of Volume 1, Module 6, Sections 1.5.2.1 and 1.5.2.2.

Some regulatory programs, such as the SDWA compliance program, may prescribe acceptable approaches for detection capability determinations. See Appendix B for more details on Detection Capability.

1.5.3 Evaluation of Precision and Bias

The laboratory needs to evaluate the precision and bias of a method for each analyte of concern for each quality system matrix. Precision and bias must be characterized across the range of activities that brackets those applicable in samples, including zero activity. This might be accomplished by analyzing test sources with activity ranging from zero (i.e., blank) to the highest activity the laboratory will process for a given type of sample.

Key Points:

- The laboratory must establish the laboratory precision and bias for all measurements and all matrix types.
- The initial demonstration of capability (DOC) does not replace the method validation where the precision and bias are determined.
- Acceptance criteria for performance should be based on one of the following:
  - -DQOs/MQOs
  - -Applicable regulations (e.g., SDWA)
  - -Published guidelines, such as MARLAP or FEM

Examples:

1. One approach might involve using LCS or other spiked sample performance data to generate precision and bias results.
2. A laboratory could also analyze replicate blanks and evaluate the results for absolute bias.

1.5.4 Measurement Uncertainty

All radiochemical measurement results needs to be reported with an estimate of uncertainty expressed either as a standard deviation or a multiple thereof.

Key Points:

- The laboratory is required to document its procedure for estimating uncertainty in its quality system documentation.
- The reported results must also explicitly specify the total uncertainty. The results of the precision evaluation need to be compared to the uncertainty estimates as a check on the validity of the uncertainty evaluation procedure.
- The intent here is that the laboratory will report total uncertainty unless they are specifically required to report counting uncertainty.
- Reports must specify the type of uncertainty reported (counting or total) and coverage (e.g., 95%, 1 sigma, or k=1).

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Refer to Appendix C for a discussion of uncertainty calculation.

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1.5.5 Evaluation of Selectivity

The laboratory needs to qualitatively evaluate selectivity, if applicable, by addressing the following sample and matrix characteristics: See Appendix D for more information.

- the effect of matrix composition on the ability of the method to detect analyte;
- the ability of the method to chemically separate the analyte from the interfering analytes; and
- spectral and instrumental interferences.

The evaluation of selectivity may be accomplished by testing matrix blanks, spiked matrix blanks, worst-case samples, or certified reference materials. If applicable, a qualitative selectivity statement needs to be included in the SOP.

1.6 Demonstration of Capability (DOC)

1.6.1 General

The laboratory analyst must have constant, close supervision until a satisfactory DOC has been completed.

Key Point:

- All DOCs need to be documented, retained and readily available at the laboratory.

1.6.2 Initial DOC

An initial DOC needs to be completed prior to using any method and at any time there is a change in instrument type, personnel, or method and any time that a method has not been performed by the laboratory or analyst in a twelve month period. The DOC is not a method validation. It serves to demonstrate that the analyst is capable of running a validated method. Generally, the validation is more extensive and provides enough detail to simultaneously meet requirements for the initial DOC for the analyst performing it.

Key Points:

- Performance requirements are generally defined by method, regulation, contract, or accreditation requirements.
- Documented DOC is performed for each unique method and quality system matrix combination.
- Each analyst must perform a DOC before analyzing any samples.
- A new DOC is required whenever there is a change in method, instruments, or personnel.

Discussion:

The laboratory needs to document each initial DOC in a manner such that the following information is readily available for each analyst:

- Analyst(s)
- Matrix
- Analyte(s), class of analyte(s), or measured parameters
- Identification of method(s) performed
- Identification of laboratory-specific SOP used for analysis, including revision number
- Date(s) of analysis
- Summary of analyses

If the method, regulation or contract does not specify an initial DOC, the following procedure would be one acceptable approach. It is the responsibility of the laboratory to document that other approaches to initial DOC meet applicable requirements.

1. Prepare 4 test samples consistent with Section 1.7.2.3 Positive Control and 4 method blanks of clean quality system matrix in which no target analytes or interferences are present.
2. Analyze the samples according to the method.
3. Calculate the mean recovery and standard deviation of the spikes.
4. Compare the data to acceptance criteria specified in the method/regulation or contract.

Where no acceptance criteria exist, the laboratory needs to compare the data with criteria established in the laboratory quality system.

When performing multi-elemental analysis by gamma spectrometry, the DOC need not involve every radionuclide. The standard specifically states the test sample needs to contain gamma-emitting radionuclides that represent the low, medium, and high energy range of the analyzed gamma-ray spectra.

1.6.3 Ongoing DOC

The laboratory needs to have a documented procedure describing ongoing DOC that includes procedures for how the laboratory will identify data associated with ongoing DOCs. The analyst(s) needs to demonstrate on-going capability by routinely meeting the quality control requirements of the method, regulation or contract, or as established by this Standard and by the laboratory's quality system. It is the responsibility of the laboratory to document that other approaches to ongoing DOC are adequate.

Key Points:

- Performance is generally defined by the method, regulation, contract or the Laboratory's quality system and relies on Performance Testing samples.
- Ongoing DOC is by method, analyst and matrix.
- If the method has not been performed by the analyst in a 12-month period, an initial DOC needs to be performed.

1.7 Technical Requirements

1.7.1 Instrument Set-Up, Calibration, Performance Checks and Background Measurements

The set-up, calibration, performance checks of instrumentation, and background determinations are all critical steps of an analytical process. If not done adequately, all subsequent analyses are suspect. Many reference methods, however, contain no or incomplete requirements. The laboratory may need to supplement the method to satisfy applicable program, regulatory, or contractual requirements, in addition to those specified in Module 6.

The structure of this section parallels the stages of the calibration life cycle

- Instrument set-up
- Initial calibration
- Calibration verification
- Instrument checks

The approach in the standard parallels that in ASTM D7282 – Standard Practice for Set-up, Calibration and Quality Control of Instruments Used for Radioactivity.

1.7.1.1 Initial set-up of Instrumentation

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Many of these requirements address procedures and documentation for set-up and configuration of instrumentation. They might be implicit in requirements for procedures and documents but they are routinely overlooked and impact the quality of results produced.

Key Points:

- The laboratory needs to maintain the required radiation measurement systems for each method it performs.
- The laboratory needs to maintain records documenting radiation measurement system configuration and values for hardware- and software- related operational parameters .
- The laboratory must ensure the continued integrity of system configuration and perform corrective actions to determine and ameliorate any potential impact if any changes are made or identified.

1.7.1.2 Initial Calibration

This section specifies the essential elements for initial calibration of radiation measurement systems. Although standards of varying activity are not needed to calibrate radiometric techniques, multiple points may be needed to correlate parameters other than activity. Here are six common examples:

i) channel-energy calibration of alpha or gamma spectrometers;

ii) energy-efficiency calibration of gamma spectrometers;

iii) mass-efficiency (mass-attenuation) calibration of gas-flow proportional or x-ray detectors;

iv) quench-efficiency calibration of liquid scintillation detectors;

v) mass-crosstalk calibration of gas-flow proportional detectors; and

vi) quench-crosstalk calibration of liquid scintillation detectors.

This section reiterates the need for physical calibration of instruments against traceable reference materials but opens the door for applying mathematical or statistical corrections based on mathematical techniques such as Monte Carlo simulations.

Key Points:

- The laboratory needs to establish and document in written procedures and in records the details of the initial calibration. Details needs to include , at a minimum:
  1. the type of calibrations to be performed;
  2. the number of calibration points required;
  3. a description of the calibration standards required;
  4. the preparation of the calibration standards;
  5. the counting of the calibration standards;
  6. the maximum permissible uncertainty for calibration measurements (e.g., a maximum relative uncertainty of the calibration parameter or a minimum number of counts collected); and
  7. all calculations.

- The laboratory needs to document the criteria for conditions that initiate (re)calibration in its SOPs.
- The laboratory needs to quantitate sample results only from the initial instrument calibrations unless otherwise allowed by regulation, method or contract.

Example - Corrections to calibration

The laboratory performed a calibration of a Marinelli beaker geometry for Ge gamma spectrometer, using a physical source containing mixed gamma reference standard (Sections 1.7.1.2c) and 1.7.2.6c)). The source consists of an acidic solution of density 1.015 g cm^{-3}. Then two LCS samples were prepared by spiking and homogenizing vegetation (Section 1.7.2.3) with densities of 0.5 and 0.9 g cm^{-3}. The density and coincidence (cascade)-summing corrections were calculated for these two samples using Monte Carlo program (Section 1.7.1.2d)). In the calculations, nominal Ge

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detector parameters were used as given by the manufacturer, Marinelli beaker dimension were measured, and the chemical composition was taken for an average vegetation. The LCS samples were quantified, the calculated corrections were applied, and the results verified the known values. The LCSs should bracket the range of densities 0.5-0.9. Ensure that the same size and shape containers are used.

Comment: The nominal detector parameters as well as average vegetation composition are acceptable when the corrected values agree with the known values across the range over which corrections will be made because the calculated corrections are not very dependent on uncertainties in these quantities. For analyzing real vegetation samples, the corrections were calculated between 0.5 and 0.9 g/ cm^3 in steps of 0.05. From these values, the corrections are interpolated for a given sample density in the range. This is much faster and nearly as accurate as calculating the corrections for every sample.

1.7.1.3 Calibration Verification

This section of the Standard establishes requirements for verification of initial calibrations prior to use for analyzing samples. Requirements for calibration verification were poorly differentiated from and frequently confused with instrument performance checks. Calibration verifications verify the integrity of initial method-specific calibrations relative to established criteria that is based on measurement of independently produced calibration verification sources.

Key Points:

- Initial instrument verifications must be performed prior to use of an initial calibration for analysis of samples
- Unless reference standards cannot be procured or obtained, the standard used must be from a source or lot independent of the reference standard used in the initial calibration.
- The laboratory must specify the maximum permissible uncertainty for calibration verification measurements (e.g., the minimum number of counts collected for each measurement) in their SOPs.
- The laboratory needs to specify verification acceptance criteria in their SOPs and when corrective actions are necessary.

EXAMPLE:

The laboratory performed initial calibration of Ge gamma spectrometer (Section 1.7.1.2b) ii)), using a reference mixed gamma standard (Am, Cd, Co, Ce, Hg, Sn, Sr, Cs, Mn, Y, Zn, Co) (Sections 1.7.1.2c) and 1.7.2.6c)). However, the vendor was not able to provide a reference standard of the relatively short-lived mixed gamma radionuclides from another lot for calibration verification (Section 1.7.1.3a).

Comment: Therefore, the laboratory performed calibration verification by quantifying a set of LCS samples (Sections 1.7.1.3a) ii) and 1.7.2.3) and ensured that the acceptance criteria were met.

1.7.1.4 Instrument Performance Checks

In previous versions of the standard, this section was titled Continuing Calibration Verification, a misleading term. Instrument performance checks measure and track the stability of key detector response-related parameters over time. The continuing validity of initial calibrations is established by demonstrating the stability of the detection system from the point of initial calibration to the time of the Test Source measurement, whether it be days, months or even years because it is based on demonstrated evidence of instrument stability over time.

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- Making sure that the source has not changed provides one of the most reliable ways of detecting small changes in instrument response. Ensure that your instrument performance checks meet all the requirements specified in Section 1.7.1.4 of Module 6.

EXAMPLE 1. Change of operational parameter

Laboratory established an initial conversion gain of 4096 channels for a full energy range of 2 MeV for a Ge gamma spectrometer (Section 1.7.1.1b)). The gamma energy calibration was then performed using $^{125}Sb/^{154,155}Eu$ mixed gamma source (Section 1.7.1.2b) i)). The initial efficiency calibration (Section 1.7.1.2b) ii)) was performed using a reference mixed gamma standard (Sections 1.7.1.2c) and 1.7.2.6c)). The calibration was verified (Section 1.7.1.3) and instrument performance checks were performed as scheduled (Section 1.7.1.4).

A specific project for measurement of fresh fission products required readjusting of conversion gain to 16384 channels for the same energy range (Section 1.7.1.1.c)). The laboratory recalibrated the energy using Sb/Eu source (Section 1.7.1.2b)i)). Subsequent performance checks did not indicate any change in efficiency or resolution.

Comment: No efficiency re-calibration was necessary because performance did not change. A new energy calibration did need to be performed.

Example 2 – Performance check failure

An analyst performed a daily instrument check on a solid-state scintillation detector (Section 1.7.1.4b)v)) and it had no counts. The analyst recognized that the high voltage was off. He turned it on and the repeated performance check passed (Section 1.7.1.4a)vi)).

Comment: Since zero counts did not enter the database, the analyst followed laboratory SOP (Section 1.7.1.4a)vii)) which did not require informing supervisor or write a corrective action in this case.

Example 3 – Performance check failure

An analyst performed an instrument check on a semiconductor gamma detector (Section 1.7.1.4b) i) 1). The performance check was outside 95% tolerance (Section 1.7.1.4a) vi)). The analyst repeated the measurement (Note to Section 1.7.1.4) and it was outside tolerance again. The analyst informed the supervisor per laboratory SOP (Section 1.7.1.4a) vii)). The supervisor determined that the check source was measured at a wrong position. The source was repositioned and subsequent performance check passed.

Comment: Since the out of tolerance results were entered into the database, a dated record in the detector manual was entered; however, no written corrective action was necessary. The outliers do not affect past or future tolerance charts because they are rejected by a Grubbs test in calculations.

Example 4 – Performance check deviates from expected value

After initial calibration of a liquid scintillation counter for tritium analysis, the laboratory performs recalibrations on an annual basis (Section 1.7.1.2). Performance check is performed using a factory sealed tritium check source (Section 1.7.1.4a)iii)). The performance check results are plotted on a tolerance chart (Section 1.7.1.4a)vi)) and include fitting of exponential decay of tritium (Section 1.7.1.4a)v)). In between recalibrations, the supervisor observes a steadily increasing deviation from the fitted exponential curve up to 0.5%, in spite of satisfying statistical tolerance chart.

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Comment: The supervisor determines that this discrepancy is caused by an ageing of the optical system in the liquid scintillation counter. However, since this deviation is much smaller than the uncertainty of the laboratory reported results (5% or more), supervisor decides that it is not necessary to either replace the detector system or initiate out of schedule recalibration. The next recalibration will accommodate this aging of the counter.

Example 5 – Exception to minimum frequency of performance check

An analyst performs daily performance check procedure for a gas proportional counter on Friday (Section 1.7.1.4b)iii)) and then submits another procedure containing a batch of 20 samples which will count till Sunday morning and then begins counting another batch of 20 samples . The analyst prepares another daily performance check procedure to be counted automatically and immediately after the sample procedure on Sunday, skipping Saturday.

Comment: Skipping Saturday is allowed according to Section 1.7.1.4c)ii). Measuring of performance check on Monday instead of Sunday would also be acceptable.

1.7.1.5 Subtraction Background Measurements

Subtraction background measurements are performed to assess and correct for contributions due to cosmic radiation, naturally-occurring radioactivity, electronic noise, impurities in the detector, shielding, and source mounting material, or other sources that are not affected by the analytical processes. Even a small amount of bias in background measurements may be significant when results are close to background since it can influence decisions about whether the measurement indicates the presence of analyte of not.

Numerous counting configurations may be used to determine subtraction background, depending on the detector and the method, including: counting an empty detector; counting an empty container or blank Test Source in a detector; or counting a container filled with a surrogate matrix material free of measureable levels of radioactivity.

Note: The frequency of subtraction background measurements may be increased from the ~~above~~ requirements listed below when there is low tolerance for lost data due to failure of a subtraction background measurement.

Key Points:

- The laboratory needs to maintain written procedures for performing and evaluating subtraction background measurements.
- Background counting time must be at least as long as the associated sample counting time and be representative of the background count rate.
- The subtraction background measurement needs to be accomplished in one of the following ways:
- Paired measurements in which the subtraction background measurement is counted before or after the Test Source measurement or batch of Test Source measurements.
- Measurements performed at a fixed frequency, in which Test Sources may be measured between successive background subtraction measurements. In this case, the laboratory needs to perform background subtraction measurements at the following minimum frequencies:
- Gamma-ray spectrometry systems: Monthly.
- Alpha-particle spectrometry systems: Monthly.
- Gas-proportional and semiconductor alpha/beta detectors: Quarterly.

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- Liquid scintillation detectors.
- Individual quenched background: Once per Preparation Batch.
- Quenched background curve: According to frequency specified in laboratory procedures.
- Solid-state scintillation detectors (e.g., zinc sulfide) used for non-spectrometric measurements:
  - Day of use.

1.7.1.6 Short-Term Background Checks

Short-term background checks performed between subtraction background measurements are quality control measures used to verify the integrity of subtraction background measurements, check for possible detector contamination, electronics noise and to monitor each detector for trends and deviations from Poisson statistics. These background checks may be shorter in duration, yet more frequent than the subtraction background measurements, and therefore they may not always effectively identify every discrepancy that could compromise Test Source measurements (e.g., low-level contamination).

Key Points:

- The laboratory needs to maintain written procedures for performing and evaluating short-term background checks.
- The laboratory needs to establish exceptions to minimum frequencies for short-term background checks.
  - When short-term background has changed since the previous determination such that significant background bias is imparted to intervening Test Source measurements, the laboratory needs to initiate a corrective action. If the bias cannot be resolved, the laboratory needs to qualify affected results.
- If subtraction background measurements are performed with sufficient frequency for a given method or detector type, such that they ensure background integrity and are capable of identifying detector contamination, these subtraction background measurements may be substituted for short-term background checks, in which case the short-term background checks are not required.
- For liquid scintillation detectors, the laboratory needs to check short-term unquenched backgrounds each day of use. Unquenched backgrounds are sealed background vials such as those supplied by instrument manufacturers. Although unquenched backgrounds do not match the geometry or the levels of quench observed in real samples and should never be used for subtraction, if a change is detected, all sample counts since the last background check are suspect and would normally need to be recounted.

1.7.1.7 Contamination Monitoring

The laboratory needs to have written procedures that address cases where radiation detectors have been contaminated, as determined by the subtraction background measurements, short-term background checks, or method blanks. Detectors may not be brought back into service until corrective actions are completed.

Key Point:

- Contaminated detectors may not be brought back into service until corrective actions are completed

1.7.2 Quality Control for Radiochemistry

The essential elements of quality control are the quality control tests and/or samples that must be utilized to properly document the quality and defensibility of the data being generated. These elements consist of positive and negative controls, detection capability, data reduction, quality of standards and reagents, selectivity, and constant and consistent test conditions. Negative controls are method blanks (laboratory reagent blank) and positive controls are laboratory control samples (laboratory fortified blank), while sample specific controls consists of matrix spikes and matrix spike duplicates, matrix duplicates, and surrogate spikes.

1.7.2.1 General

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Both reference and non-reference methods require validation. (See Appendix B for an example of method validation study.) his to be done for each quality system

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The validation must follow a documented procedure.
The validation must address detection capability, precision, bias, measurement uncertainty, and selectivity (consistent with published guidelines such as MARLAP, FEM, EUROCHEM) where possible the activity range shall include zero activity

The validation records must be maintained for the life of the method and be readily retrievable.

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To prevent the use of a detection capability that is unrealistically low, the confirmation needs to take into account any analyte losses during sample preparation.

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Precision and bias can be derived and monitored from the LCS performance data.

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The initial documentation of capability is generally considered to meet the requirement for DOC.

For non-reference methods, the Standard enumerates the method for establishing precision and bias.

Precision and bias can be derived and monitored from the LCS performance data.

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Acceptance criteria for performance should be based on one of the following:

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-Applicable regulations (e.g., SDWA)

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-Published guidelines, such as MARLAP or FEM

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needs to be analyzed to test

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See Appendix B for another example of Uncertainty Calculations

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1. The intent here is that the laboratory will report total uncertainty unless they are specifically required to report counting uncertainty.

2. Reports must specify the type of uncertainty reported (counting or total) and coverage (e.g., 95%, 1 sigma, or k=1).

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The initial calibration and calibration verification of equipment are the most important steps of an analytical process. If not done adequately, all of the subsequent steps are suspect. Unfortunately, many reference methods contain sketchy requirements for calibration and quality control. Laboratories performing radiochemical measurements must rely more on laboratory-developed methods than reference methods for any matrix other than drinking water.

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Essential elements of instrument performance checks are

The check source used for instrument performance checks need not be a reference standard as defined in this module.

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The laboratory needs to use the same check source for ongoing performance checks as the one in the preparation of the tolerance or control chart limits at the point of the initial calibration.

The laboratory needs to prepare, handle, seal and/or encapsulate check sources to prevent damage, loss of activity and contamination.

The laboratory needs to minimize the uncertainty of the check source count to allow detection of small changes in detector response relative to the acceptance criteria. The count duration and check source activity should be sufficient to provide adequate counting statistics over the life of the source.

Where significant, the radioactive decay in the check source needs to be taken into account when evaluating count-rate sensitive parameters such as efficiency.

The laboratory needs to monitor the results of instrument performance checks using control or tolerance charts to ensure that instrument performance has not changed significantly since the point of the initial calibration.

The laboratory procedure needs to

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specify what corrective actions are to be taken when performance check acceptance criteria are not met.