Radiochemistry Expert Committee (REC)
Meeting Summary

July 26, 2017

1. Roll Call and Minutes:

Bob Shannon, Chair, called the meeting to order at 1:02 pm Eastern on July 26, 2017 by teleconference. Attendance is recorded in Attachment A – there were 5 members present. Associates: Carl Kircher, Joe Pardue and Carolyn Wong.

Meeting minutes are distributed by email for comment/revision for a week and then posted on the TNI website.

2. NEMC

Bob asked who will be at the meeting in DC and no one on the call today is planning to be there. Last month Yoon mentioned she would be there. Ilona will try to provide Webex if there is an internet connection in the conference room. People should look for an email on Monday, 8/7/17.

The Small Laboratory Handbook could be an agenda item if it is not finished up today.

3. Small Laboratory Handbook

Bob thanked everyone for all the feedback he received since the last meeting. Bob placed the comments he received into the DRAFT version he sent to everyone.

Bob reviewed the entire document using Webex. Any changes made to the document were made through track changes and can be seen in Attachment D.

"Laboratory Quality System" capitalization will need to be consistent throughout the document.

Larry volunteered to check Bob's numbers in the example calculations and let him know about any needed changes.

Bob plans to provide all committee members with a copy of the SLH with the changes made today and ask people to vote to finalize the SLH. Ilona will make the suggested editorial changes.

Addition: A motion was made by Larry Penfold by email on 9/5/2017 to approve the SLH – v8 as sent on 7/26/17. The motion was seconded by Dave Fauth on 9/5/2017. Vote: yes-8

(Bob Shannon, Sreenivas (Vas) Komanduri, Marty Johnson, Dave Fauth, Keith McCroan, Larry Penfold, Yoon Cha, Candy Friday), no-0 (Ron Houck), abstain-1 (Tom Semkow).

The motion passed on 9/8/2017.)

6. New Business

None.

7. Action Items

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

8. Next Meeting and Close

The next meeting is scheduled for August 8, 2017 at 1pm Eastern in Washington, DC. The next teleconference will be in September.

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

The meeting was adjourned at 2:56pm 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) **Absent**	State of NJ Department of Environmental Protection Trenton, NJ	AB	609-984-0855	Sreenivas.Komanduri@dep.state.nj.us
Marty Johnson (2019) **Absent**	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 until 2:30**	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*) **Absent**	PA DEP/Bureau of Laboratories	AB	717-346-8210	rhouck@pa.gov
Yoon Cha (2020) **Absent**	Eurofins Eaton Analytical	Lab	213-703-5800	YoonCha@eurofinsUS.com
Candy Friday (2020) **Absent**	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	Send SLH to Ilona after final update from today so she can do editing and formatting.	Bob/Dave	6/10/17	7/5/2017
84	Ilona will send the SLH back to the QS committee for further review.	Ilona	6/28/17	

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 NELAC INSTITUTE

The TNI Standard

Guidance For Small Labs

1st Edition

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Comment [1]: 1.Structure:
a.Pages numbers are missing
b.Table of contents is missing
c.Small vertical lines at top left on all pages, except cover
d.Reference to Attachment 2 appears before Attachment 1
2.Acronym Definitions missing
a.MARLAP
b.FEM
c.NIST
d.NPL

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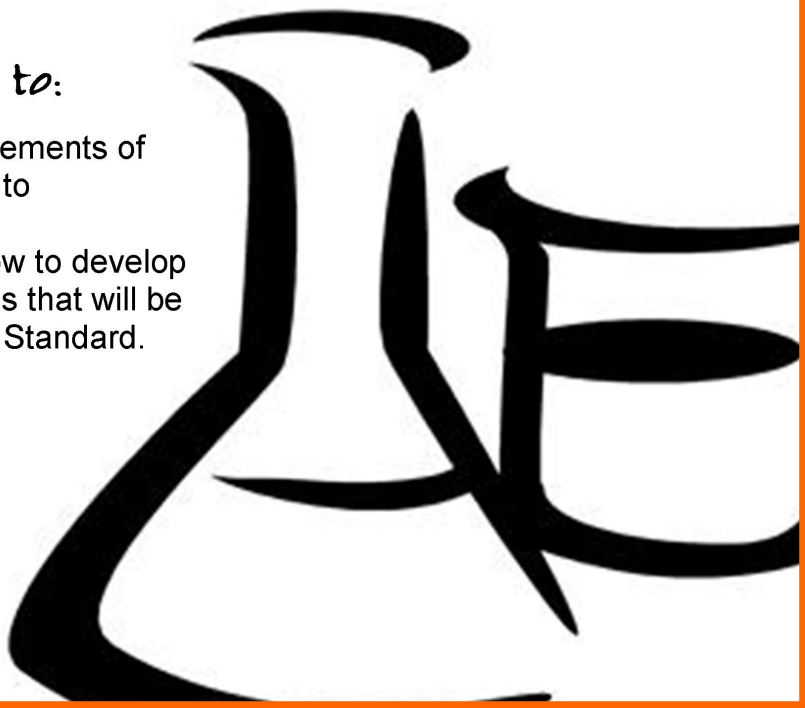
Section Break (Next Page)

Use this Guide to:

- Help explain the requirements of the TNI Standard, and to
- Obtain guidance on how to develop policies and procedures that will be in compliance with the Standard.



The elements of this module apply to techniques used for the purpose of measuring or monitoring radioactivity, or techniques used to demonstrate compliance with regulations pertaining to radioactivity. The laboratory needs to comply with the requirements of Chemical Testing (Module 4) in cases where technique-specific QA/QC is not defined in Radiochemical Testing (Module 6) (e.g., Mass Spectrometry [ICP-MS, TIMS] or Kinetic Phosphorimetry) or by the respective reference method (e.g., calibrations, calibration verifications, determinations of detection statistics, or method-specific quality controls). The laboratory needs to identify in its quality system how and when it is complying with the requirements and elements of Module 4 and Module 6, as applicable.

1.4 Method Selection

The TNI Standard generally assumes that radiochemistry laboratories use methods based on regulatory requirements (e.g. SDWA or Clean Water Act (CWA) compliance measurements). For those situations where a reference method is not specified by regulation or contract, any applicable method may be used. In all cases, the method used must be validated for that application. (see 1.5.1 for further discussion of method validation). In all cases, the client must approve method selection.

1.5.1 Validation of Methods

Both reference and non-reference methods require validation. Validation needs to be done for each quality system matrix.

Key Points Are:

- The validation must follow a pre-defined process that is consistent with Sections 1.5.2 through 1.5.5 of V1M2 of the TNI Standard.
- The activity range for validation must include zero activity whenever radiochemical methods will generate results that include zero activity that may be reported to the client (together with their associated uncertainty).
- 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 incomplete or not available, or if the laboratory modifies the reference method, the laboratory must generate this method performance data based on the final method used at the laboratory (i.e., by validating the method).
- For existing methods, analysis of historical internal quality control data is one strategy that may be used to generate some or all of the performance data needed to satisfy validation requirements.

- 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 of the Standard is to have laboratories report total uncertainty unless they are specifically required to report only 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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Example(s):

1 Refer to Attachment 3 for a discussion of uncertainty calculation.

1.6 Demonstration of Capability

1.6.1 General

Key Points:

- The laboratory analyst must have constant, close supervision of an experienced analyst until a satisfactory DOC has been completed.
- 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 12-month period. The DOC is not a method validation rather it demonstrates 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.

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 if overlooked, the quality of results produced may be negatively impacted.

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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:

1. channel-energy calibration of alpha or gamma spectrometers;
2. energy-efficiency calibration of gamma spectrometers;
3. mass-efficiency (mass-attenuation) calibration of gas-flow proportional or x-ray detectors;
4. quench-efficiency calibration of liquid scintillation detectors;
5. mass-crosstalk calibration of gas-flow proportional detectors; and
6. 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.

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Key Points:

- The laboratory needs to establish and document in written procedures and in records the details of the initial calibration including, 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 and a minimum number of counts collected (e.g., 1% or 10,000 counts); 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 most recent valid initial instrument calibrations unless otherwise allowed by regulation, method or contract.

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Example(s):

1 Mathematical Corrections to Calibrations:
The laboratory has performed a calibration of a Marinelli beaker geometry for a High Purity Germanium (HPGe) gamma spectrometer using a physical source containing a mixed gamma reference standard (Sections 1.7.1.2c) and 1.7.2.6c)). The calibration source consists of an acidic solution of density 1.015 g cm^{-3}. The laboratory intends to use a mathematical technique (i.e., Monte Carlo simulation) to correct the water equivalent efficiencies to count vegetation samples across a specified range of densities. This example presents one possible approach that might be used to validate the corrections prior to use.

Two LCS samples are prepared by spiking and homogenizing two vegetation matrices (Section 1.7.2.3) with densities of 0.5 and 0.9 g cm^{-3} and transferring them to Marinelli beakers that match the mixed gamma calibration standard ensuring that they are filled to the same height as the standard. Density and coincidence (cascade)-summing corrections are calculated for these two samples using a Monte Carlo program (Section 1.7.1.2d)). Calculations performed used nominal Ge detector parameters (i.e., detector characterization) provided by the manufacturer, Marinelli beaker dimensions measured by the laboratory, and the elemental compositions of the aqueous calibration standard and a typical or representative vegetation sample. The LCS samples are quantified, the calculated corrections are applied, and the results verified by comparing to the known values. Since the LCSs bracket the range of densities 0.5-0.9 cm^{-3}, this established the range of sample densities to which the corrections are applicable.

Comment: The nominal detector parameters as well as average vegetation composition are acceptable when the corrected values agree with the known values bracketing 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 can be calculated between 0.5 and 0.9 g cm^{-3} in steps of 0.05 g cm^{-3}. From these values, the corrections are interpolated for a given sample density in the range based upon bulk density generally determined by measuring the mass of sample that is needed to fill the counting container to the proper volume. 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. Often, requirements for calibration verification have been poorly differentiated from, and frequently confused, with instrument performance checks. Calibration verifications verify the integrity of initial method-specific calibrations by comparing measurements of independently produced method-specific calibration verification sources to established acceptance criteria. Instrument performance checks, in contrast, generally use a single source to verify the stability of an instrument's performance between the initial calibration(s) (there may be many initial calibrations) and the analysis of samples.

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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 reference 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., standard deviation of multiple determinations or 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(s):

1 The laboratory performs initial calibration of Ge gamma spectrometer (Section 1.7.1.2b)) 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, no vendor is available to provide a reference standard containing the relatively short-lived mixed gamma radionuclides from second or independent lot for calibration verification (Section 1.7.1.3a)).

Comment: Therefore, the laboratory can perform calibration verification by quantifying a set of LCS samples (Sections 1.7.1.3a and 1.7.2.3) in an older standard and to ensure 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. Rather, instrument performance checks use a single source to measure and track the stability of key detector response-related parameters over time. The continuing validity of all initial calibrations on the detector is established by demonstrating the stability of the detection system from the point of initial calibration through the time of the Test Source measurement, whether it be days, months or even years. This is thus based solely on demonstrated evidence of instrument stability.

Key Points:

- Since the acceptance criteria rely on the results of a specific check source, it is critical to ensure that the same source is used, and that it is not damaged or otherwise compromised. Using the same source over time is a very precise way of detecting small changes in instrument response. It is important, to ensure that all instrument performance checks meet all the requirements specified in Section 1.7.1.4 of Module 6.

Example(s):

1 Change of Operational Parameter: Laboratory establishes 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 is then performed using an $^{125}Sb/^{154,155}Eu$ mixed gamma source (Section 1.7.1.2b) i)). The initial *efficiency* calibration (Section 1.7.1.2b) ii)) is performed using a reference mixed gamma standard (Sections 1.7.1.2c) and 1.7.2.6c)). The calibrations are verified (Section 1.7.1.3) and instrument performance checks performed as scheduled (Section 1.7.1.4).

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

Comment: A new energy calibration must be performed but efficiency re-calibration is not necessary because the instrument performance checks verify that the efficiency has not changed.

2 Performance Check Failure: An analyst performs a daily instrument check on a solid-state scintillation detector (Section 1.7.1.4b)v)) and it shows no counts. The analyst recognizes that the high voltage was off. He turns it on and the repeated performance check passes (Section 1.7.1.4a)vi)).

Comment: In this case, the zero counts do not enter the database, so the analyst follows laboratory SOP (Section 1.7.1.4a)vii)) which does not require informing supervisor or writing a corrective action.

3 Performance Check Failure: An analyst performs an instrument check on a semiconductor gamma detector (Section 1.7.1.4b)i)1)). The performance check falls outside 95% tolerance (Section 1.7.1.4a)vi)). The analyst repeats the measurement (Note to Section 1.7.1.4) and it falls outside the tolerance again. The analyst informs the supervisor as required by the laboratory's SOP (Section 1.7.1.4a)vii)). The supervisor subsequently determines that the check source has been measured in the wrong position. The source is repositioned and subsequent performance check passes.

Comment: Since the out of tolerance results that enter the QC database are due to a known procedural non-compliance, the data should not be used to evaluate past or future control or tolerance. The record should not be deleted or obliterated, however. Instead, the laboratory may choose to flag the data point in the database as invalid ensuring that the rationale is documented (e.g., by entering a dated record in the detector maintenance logbook). There may or may not be need for a written corrective action depending on how the laboratory's SOP/quality system addresses this case.

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. Note that the standard does not require annual recalibration unless the laboratory decides to impose a frequency upon themselves. (Section 1.7.1.2). A 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)) which takes into account radioactive decay of the tritium check source (Section 1.7.1.4a)v)). After six months, the supervisor observes a negative deviation from the fitted exponential curve approaching 0.5%, in spite of satisfying statistical tolerance chart limits.

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Comment: The supervisor might determine that the trend is significant and take appropriate action such as maintenance and/or recalibration of the system. The supervisor, however, might also determine that this discrepancy is caused by aging of the optical system in the liquid scintillation counter and that the deviation is much smaller than the uncertainty required for the laboratory to meet its MQOs for reported results. If this is the case, supervisor may decide that it is not necessary to immediately replace detector system components or initiate an out-of-schedule recalibration. The next recalibration will accommodate the aging of critical component(s) of the counter. The supervisor should document the occurrence, for example, in the detector maintenance logbook, and the rationale for his decision.

5 Exception to Minimum Frequency of Performance Check: An analyst performs the daily performance check procedure for a gas proportional counter with an automatic sample changer on Friday (Section 1.7.1.4b)iii)) and then initiates counting a batch of 20 samples which will run until Sunday morning after which another batch of 20 samples will start counting. The analyst prepares a daily performance check to be counted automatically and immediately after the sample procedure on Sunday, skipping Saturday.

Comment: Skipping daily performance checks for up to 7 days during the counting of a batch of samples on an automatic sample changer is allowed according to Section 1.7.1.4c)ii) as long as a successful check is performed at the end of the batch. Since a second batch of samples is being counted following the first, a performance check is required before initiating the count of the next batch. If any check fails to meet acceptance criteria, any results obtained since the last successful check would generally be considered to be suspect and need to be recounted.

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, 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 an analyte or 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

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day-of-use may need to be interpreted as the day immediately prior to filling the Lucas cell with radon.

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.
  - When short-term background checks indicate that the background has changed since the previous determination such that significant bias is imparted to intervening Test Source measurements, the laboratory must take action to determine if the instrument is contaminated, and if previous sample results have been compromised. If any results have been compromised, the laboratory should take action to ameliorate the problem. It should also recount affected samples, or where this is not possible, qualify affected results.
- If subtraction background measurements for a given method or detector are performed with sufficient frequency, that they ensure background integrity and are capable of identifying detector contamination in a manner that is timely enough to identify potentially impacted results, these subtraction background measurements may be substituted for short-term background checks, in which case separate 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 must maintain written procedures that address cases where subtraction background measurements, short-term background checks, or method blanks may indicate that radiation detectors have been contaminated. Detectors may not be brought back into service until corrective actions are completed.

Key Points:

- If monitoring of instrumentation indicates contamination, the laboratory should refer to guidance from the instrument vendor for cleaning and decontamination to minimize the risk of damaging the instrumentation. To the extent possible, it is recommended that routine measures for decontamination be formalized in the laboratory's SOPs.
- Cleaning a detection system removes contamination that may have compromised prior sample measurements. It is important to keep in mind that short-term backgrounds tend to be shorter than, and thus less sensitive than ongoing sample measurements. Best practices would be to only clean the detection system after a background subtraction measurement. This ensures that the laboratory has data it needs to demonstrate that the measurement system was "in control" at all times samples are measured. If a detector is known to be contaminated, the laboratory may decide to reject data for all samples since the previous background subtraction count. In such cases, a background subtraction measurement would not be necessary.
- Contaminated detectors may not be brought back into service until corrective actions are completed, including determination of whether sample results have been impacted.

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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 consist of matrix spikes and matrix spike duplicates, matrix duplicates, and surrogate spikes.

1.7.2.1 General

It is important to recognize that many radiochemistry laboratories rely on *non-mandated* methods (e.g., laboratory-developed or modified methods). They frequently develop or modify (and validate) methods to address analytical needs. In cases where QC requirements are not specified by a source external to

the laboratory (e.g., regulation or contract), it is incumbent on laboratories to establish QC requirements. When external requirements are more stringent than the Standard, the more stringent requirements must be met. This provides flexibility while helping to ensure that the laboratory has a defensible basis for their QC requirements. It also allows assessors to ask about the basis for specific requirements and to reference MARLAP or other standards to explain the rationale for QC measures they select to use.

Key Points:

- The Laboratory needs to follow a documented QC program that monitors and assesses the performance of the laboratory's analytical systems. At a minimum, the QC program needs to incorporate requirements imposed by regulation, methods, and the TNI standard.
- The laboratory needs to process batch and sample-specific quality control samples to obtain empirical evidence to demonstrate that their analytical system is in control.
- The laboratory needs to employ either a Preparation Batch or a Radiation Measurement Batch (RMB) to determine the grouping of samples and assignment of batch QC.
- A Preparation Batch needs to be initiated where sample testing is performed that involves physical or chemical processing which affects the outcome of the test. Samples and associated QC assigned to a Preparation Batch need to be prepared together using the same processes, personnel, and lot(s) of reagents.
- Where testing is performed that does not involve physical or chemical processing which affects the outcome of the test (e.g., non-destructive gamma spectrometry, alpha/beta counting of air filters, or swipes on gas proportional detectors), an RMB may be initiated in lieu of a Preparation Batch. The samples and associated QC in the RMB need to share similar physical and chemical parameters, and analytical configurations (e.g., analytes, geometry, calibration, and background correction).
- Samples may be added to the RMB for fourteen (14) calendar days from the start of the first sample count, or until twenty (20) environmental samples have been counted, whichever occurs first. The 20 samples do not include laboratory QC samples.
- The laboratory may combine samples and associated QC within an RMB as long as they share a range of physical and chemical parameters, and analytical configurations (e.g., analytes, geometry, calibration, density) that conform to the ranges of physical and chemical parameters, and analytical configurations demonstrated by validation studies for the method and matrix (see Section 1.5).

- Laboratory procedures must document how method validation was performed, and records must document any corrections (e.g., for efficiency, density, cascade summing, and background) applied to physical calibrations.
- The laboratory QC program needs to document the frequency required for quality controls.
- The laboratory needs to process all batch QC samples together with and under the same conditions as the associated samples, and needs to use the same processes and procedures for preparation, analysis, data reduction and reporting of results.

> *Discussion:* Although samples in a Preparation Batch must be prepared together, they need not be analyzed concurrently on a single detector, rather they may be analyzed on different detectors as long as the detectors are calibrated for the technique in question and instrument quality controls indicate that the systems are in control. See also Attachment 5. Radiation Measurements Batch.

- The laboratory must ensure that is does not systematically or preferentially use specific detectors, equipment or glassware for the analysis of QC samples. This does not mean that laboratories should not identify and dedicate detectors, equipment, or glassware to minimize the risk of cross-contamination of samples or equipment. In general, this is considered a good contamination control practice as long as the criteria for segregation apply equally to QC samples and samples.
- The laboratory needs to assess the results of the QC samples against acceptance criteria documented in the QC program. Where there are no established criteria in regulations, the method, or contract, the laboratory needs to develop its acceptance criteria consistent with guidelines in MARLAP[3] or other consensus standards, or other criteria such as statistical control charts developed by the laboratory.
- The laboratory needs to track and trend the results of batch QC samples using statistical or tolerance control charts.
- The laboratory's QC program needs to document acceptance criteria for batch QC samples, sample-specific QCs, and for the evaluation of long-term trends and the methods used to establish these criteria.
- The laboratory needs to investigate to determine the cause when results do not meet acceptance criteria. They must take corrective actions to eliminate the source or minimize the magnitude of the problem. The laboratory should to consider samples associated with a failed QC parameter as suspect and, wherever possible, it should reprocess such samples. Where reprocessing is not possible, the laboratory must report results with appropriate data qualifiers. The laboratory needs to note the occurrence of a failed QC sample and any associated actions in the laboratory report.

Example(s):

1 All samples must be processed in a QC batch, of which there are two types: Preparation batches and Radiation Measurements Batches.

a) Most samples will be processed in preparation batches. Preparation batches apply to samples that undergo physical or chemical processing that could affect results. Examples of analyses requiring preparation batches are: gross alpha/gross beta in water (evaporation); tritium in water (distillation and mixing with cocktail); or total strontium in air filters (chemical separation).

The typical preparation batch consists of up to 20 environmental samples prepared together along with a method blank (MB), a laboratory control standard (LCS), a matrix duplicate (MD), and, if required, a matrix spike (MS). For samples with little or no activity, a matrix spike duplicate or LCS duplicate may be prepared in lieu of a matrix duplicate. Preparation of all samples within a preparation batch must be started within a 24-hour period. All samples in the preparation batch, including the quality control samples, are prepared together using the same processes, equipment, personnel, and lot(s) of reagents. Samples in a preparation batch may be counted on a single detector, or on multiple detectors as long as all detectors are calibrated and associated QC is in control. It is important to remember when setting up counts that samples should be organized for counting in such a manner that does not systematically or preferentially result in using or avoiding specific detectors to count any sample in the batch.

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b) For samples that do not involve physical or chemical processing that affects the outcome of the test, a Radiation Measurement Batch (RMB) may be used. Most frequently, this involves non-destructive testing such as gross alpha/beta or gamma spectrometry of air filter or swipe samples where the sample is not altered prior to analysis. Rather, the sample is placed directly in a planchet or counting container and counted. Samples may be added to an RMB for up to 14 days to a maximum of 20 samples.

All samples and QC samples added to an RMB, however, must share similar physical and chemical parameters, and analytical configurations. These should conform to the ranges of physical and chemical parameters, and analytical configurations that were used for validation studies for the method and quality system matrix (see Section 1.5). Put more simply, all samples should be analyzed for the same test and analytes, in the same

In fact, either approach will work as long as we require that statistical performance (e.g., observed ± 3 sd) will always be good enough to defend using our method to meet the project-required MQOs. One effective solution might be to create a hybrid that incorporates both statistical and tolerance limits in a single chart. We would require that upper and lower statistical limits always be tighter than the tolerance limits. We can accept results outside *statistical performance* as long as they meet our required ±25% tolerance limits. This will ensure that we stop the process as soon as statistical limits move outside the tolerance limits.

The Standard requires that control charts be reviewed for trends for the batch QC sample results. This is an extension of the same approach being used for control charting which identifies unlikely one-point events (i.e., a single point outside 3 sd control limits where the probability is ~3/1000) and possibly two-point trends (2 consecutive points in the warning zone where the probability of a result is ~ 2/1000).

Barring external requiremetns, it is up to the laboratory to establish in their procedures which decision rules they will use to trend data and which actions an identified trend will trigger. Although there are many improbable situations that could be identified as trends, not every trend is necessarily problematic or value-added. It is generally advisable to select a small subset of rules that indicate that data are already compromised, or that point to a need to take action soon to avoid compromising future data.

1.7.2.2 Negative Control – Method Performance: Method Blank (MB)

The MB assesses the process of handling, preparation chemical separation and analysis for cross-contamination and low-level analytical bias. Even for methods with minimal physical treatment or no chemical processing (e.g., drying, grinding and homogenization of solid samples, or preparation of sample Test Sources for swipe or air filter samples for non-destructive gamma spectrometry or alpha-beta counting), an MB could be used to assess the potential impact of sample handling and the analytical process. Absolute bias may result from contamination, changes in reagents or media, instability of the instrument background, or issues with subtraction backgrounds.

Key Points:

- Where a non-destructive gamma-ray spectrometry measurement is made using a multi-point energy/efficiency calibration curve which covers the energy range of the analyte(s) of interest:

  - a radionuclide with similar gamma energies as those of the analyte(s) of interest may be used (e.g., ^{133}Ba may be used in place of ^{131}I); or

  - the LCS needs to contain gamma-emitting radionuclides that, at a minimum, represent the low (e.g., ^{241}Am) and high (e.g., ^{60}Co) energy range of the analyzed gamma-ray spectra. Commonly a medium energy radionuclide is also included in the LCS (e.g., ^{137}Cs). The nuclides need not exactly bracket the calibration energy range or the range over which radionuclides are identified and quantified.

- The laboratory needs to evaluate results of the batch LCS using a statistical technique such as the percent recovery or z-score (see MARLAP Manual Chapter 18) that allows comparison to acceptance criteria documented in the laboratory QC program.

Example(s):

1 The media used for the MB (e.g., deionized water for aqueous samples, unused air filters, soil and vegetation from an uncontaminated area) may be used as matrix material for the LCS. Alternately well-characterized performance test sample material, or purchased spiked samples may be used as an LCS.

2 For RMBs a calibration source in the same media and geometry as the samples may be used. If a calibration source is used, it should be from a different lot as the standard used for calibration. This may be accomplished by calibrating the instrument with a new calibration source and using an old calibration source as the LCS. Alternately, two separate calibration sources from two separate lots/vendors may be purchased and one used for calibration and the other for QC.

3 The LCS must be spiked at an activity level which ensures that the uncertainty of the measurement will be less than one-third the acceptance criteria range. For example, if the results for the LCS must be within 15% of the known value, the LCS must be spiked at a level high enough that the relative standard uncertainty (k=1) of the measurement is less than 5**%** (1/3 of 15%).

When establishing levels of activity for spiking, it is important to keep in mind that the uncertainty is both a function of the standard's activity and the

2 The Duplicate Error Ratio (DER) is used to evaluate whether duplicate results agree with each other within the stated uncertainties. DER provides meaningful evaluations of statistical agreement at any sample activity but will not necessarily ensure that requirements for relative precision will be met. Generally, warning and control limits are established at 2 and 3. It is important to note that the standard uncertainty (k=1) is used to calculate DER. Thus, a 1.96σ uncertainty (k=1.96) would need to be divided by 1.96 prior to performing the calculation.

$$DER = \frac{|A_s - A_{Dup}|}{\sqrt{u^2(A_s) + u^2(A_{Dup})}},$$

where

A_S – sample result (as reported),
A_{Dup} – duplicate result (as reported),
$u(..)$ – standard uncertainty (k=1) of the quantity in parentheses, and
$u^2(..)$ – the square of the standard uncertainty (k=1) of the quantity in parentheses.

3 Requirements for duplicate analyses may be met in one of the following ways:

- For non-destructive analyses where a RMB is used, a single sample may be counted twice. For low-activity samples (less than three times the MDA) the LCS may be counted twice. If multiple detectors are use the duplicate must be counted on a different detector than the original count.
- Otherwise, a second aliquot of a sample must be taken through the total analytical process.
- When sample activity levels are expected to be low (less than three times the MDA), a MSD may be used at the discretion of the laboratory.
- If there is insufficient sample to perform a duplicate or matrix spike duplicate a LCS may be processed in duplicate.

Chemical Tracers or Carriers

1 The selected chemical tracer or carrier should have identical chemical properties as the analyte of interest. This is typically a different isotope of the analyte of interest. For example ^{242}Pu is commonly used as a tracer for the analysis of ^{238}Pu or ^{239}Pu. Stable strontium is used for the analysis of ^{89}Sr and ^{90}Sr.

2 The selected chemical tracer or carrier should not interfere with the analyses. If it is not possible to select a chemical tracer or carrier which does not interfere with the analyses the interference should be quantifiable and

Attachment 1:

Minimum Detectable Activity

Radiochemical data are often reported to include minimum detectable activity (MDA) or minimum detectable concentration (MDC) with sample results.[3] The MDA, as an *a priori* parameter, should be used to select a method that will be able to meet a Measurement Quality Objective (MQO) for detection capability (i.e., a Required MDA).

Laboratories frequently misuse the MDA concept by employing MDAs to decide whether a measurement indicates that activity is present in a sample. This practice is incorrect and should be avoided. The TNI standard and MARLAP recommend using the Critical Value (a.k.a. Critical or Decision Level) for detection decisions.

Radiochemical data are often reported in association with a sample-specific MDA. The sample-specific MDA reflects the specific analytical factors used to calculate a sample result. It indicates how well the measurement process is performing under varying real-world measurement conditions when sample-specific characteristics (e.g., interferences) may affect the detection capability. The MDA must *never* be used instead of the Critical Value as a detection threshold.

A number of specific analytical factors can affect the measurement process. Inadequate sample volume, short counting time, low detection efficiency, poor chemical yield, all can affect the detection capability of a method. The laboratory must have procedures in place for determining and documenting the detection capability even when such criteria are not found in the method, regulation or contract. Additionally, projects involving cleanup of contaminated sites often include requirements in contract specifications to report sample-specific MDAs. The laboratory needs to comply with the contract specifications.

There are multiple formulations used to calculate MDAs and critical values. Several variants of nearly the same formula may all satisfy the definition of MDA and critical value included in the Standard depending on details of the measurement. The discussion below provides an example for the determination of Critical Value and MDA.

[3] The MDC is the MDA expressed in terms of activity concentration instead of activity. For the purposes of the TNI Standard and the discussion that follows, both concepts will be referred to as MDA.

Note: MDA or MDC and SDWA DL are very different concepts. See Attachment 1 for a discussion on the MDA/MDC.

How is the DL affected by limited sample volume or shorter counting intervals? Too often, all laboratories find themselves having less than 1 L of sample or perhaps one of their instruments suddenly goes down requiring tight control over count time for the functioning equipment(s).

Let us assume that the laboratory has limited sample. An aliquot of 0.5 L is only available for the test. We assumed 1 L in our example. How will the reduced volume impact our DL? By substituting 0.5 L in the above equation, we find the DL is now 0.32 pCi/ L. Although the DL has just doubled, it is still low enough to meet the RDL of 3 pCi/L.

It is possible in advance to calculate DL for optimum counting time, or sample volume, or both. Can the laboratory count the sample and background for only 1 hour? All other parameters being the same, the DL will now be 0.63 pCi/L for a 1 hour count, which still falls below the RDL. Being able to optimize count times in advance is advantageous for laboratories with limited resources of equipment and manpower, and when additional challenge of higher than normal workload is received by the laboratory.

DETECTION LIMIT STUDY:

The SDWA DL calculation assumes that the only contributor to the uncertainty of the background is the random nature of radioactive decay (i.e., counting uncertainty). In a perfect world, the counting uncertainty would be approximated by a Poisson distribution where the square root of the number of counts is a good estimator of the standard deviation of the counts. In reality, however, there may be additional uncertainty from other sources.

Thus, drinking water laboratories may be required to perform detection limit studies to demonstrate that the detection capability of the methods, as run, is sufficient to meet SDWA program requirements. Describing this study in detail goes beyond the scope of this document. Instead, we will point readers to a recent EPA document, *Procedure for Safe Drinking Water Act Program Detection Limits for Radionuclides (EPA 815-B-17-003),* which describes in detail a process that can be used to statistically demonstrate the detection capability of the method is adequate to meet the SDWA RDL.

B) PRECISION & BIAS (ACCURACY) STUDY:

$$u_{cC}(AC) = \frac{\sqrt{C_S / t_S^2 + C_B / t_B^2}}{K_1 \times K_2 \times \ldots \times K_n} \quad (5)$$

and the total combined standard uncertainty is given by:

$$u_c(AC) = \sqrt{\frac{C_S / t_S^2 + C_B / t_B^2}{K_1^2 \times K_2^2 \times \cdots \times K_n^2} + AC^2 \times \left(\frac{u^2(K_1)}{K_1^2} + \frac{u^2(K_2)}{K_2^2} + \cdots + \frac{u^2(K_n)}{K_n^2} \right)} \quad (6)$$

MARLAP Section 19.4.3 discusses *Special Forms of the Uncertainty Propagation Formula*. MARLAP Example 19.10 presents an example based on Equation 19.16 that is very similar to one presented here.

To calculate uncertainties for more general types of activity equations, see the guidance in documents such as:

Guide to the Expression of Uncertainty in Measurement (available at http://www.bipm.org/en/publications/guides/gum.html),

NIST Technical Note 1297 "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results" (available at https://www.nist.gov/pml/nist-technical-note-1297), or

Chapter 19 ("Measurement Uncertainty") of the *Multi-Agency Radiological Laboratory Analytical Protocols (MARLAP) Manual* (available at https://www.epa.gov/radiation/multi-agency-radiological-laboratory-analytical-protocols-manual-marlap).

Attachment 5

Radiation Measurements Batch

A laboratory operates two germanium gamma spectrometers, GE1 and GE2. They are initially setup according to Section 1.7.1.1. An initial calibration is performed for a 1-L Marinelli beaker geometry, according to Section 1.7.1.2, and verified according to Section 1.7.1.3. The laboratory does performance checks twice weekly (Section 1.7.1.4.b.i)1)). The laboratory's subtraction background (Section 1.7.1.5) also serves as a short-term background check (Section 1.7.1.6.d).

The laboratory starts a Radiation Measurements Batch (Sections 1.3.1 and 1.7.2.1). The laboratory does one MB (Section 1.7.2.2.a), one LCS (Section 1.7.2.3.a)), and one MD (Section 1.7.2.4.b)iii). An MS is not required for gamma spectrometry (Section 1.7.2.4.a)ii)). The Quality Control samples are performed without preference for a detector (Section 1.7.2.1.f)). Water samples arrive randomly at the laboratory and are counted for 1000 minutes each. Therefore, only two samples can be accommodated each day.

The laboratory's schedule is as follows:

Day	GE1	GE2
Monday	Performance check	Performance check
	Sample 1	Sample 2
Tuesday	LCS	MB
Wednesday	Sample 3	Sample 4
Thursday	Performance check	Performance check
	Sample 5	Sample 6
Friday	Subtraction background	Subtraction background
Monday	Performance check	Performance check
	MD	None[1]
Tuesday	Sample 7	
Wednesday	Sample 8	
Thursday	Performance check	
	Sample 9	
Friday	Sample 10	
Monday	End of RMB[3]	

Footnotes:
1. GE2 was allocated to another urgent project, and removed from this RMB.
2. The RMB reaches 14 calendar days and must be terminated. Due to the long count times, fewer than the maximum number of 20 environmental samples / batch can be measured (Section1.7.2.1.c)iii)).

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1. Adherence to the Quality Systems Module 6 procedures, QC requirements specified by the reference method, regulation or project and the laboratory's Quality System[1] requirements need to be met by the laboratory.

2.

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3. The laboratory must meet Quality System Module 6 requirements and the laboratory's internal Quality System. While doing so, the laboratory may be responsible for complying with requirements established by regulations, contractual agreement, or in reference methods.]

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2. The laboratory re-measures an MB on GE1 to maintain the integrity of the RMB, which could be jeopardized due to a loss of GE2.
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3