Radiochemistry Expert Committee (REC)
Meeting Summary

May 10, 2017

1. Roll Call and Minutes:

Bob Shannon, Chair, called the meeting to order at 1:00 pm Eastern on May 10, 2017 by teleconference. Attendance is recorded in Attachment A – there were 7 members present. Associates: Patrick Bell, Terry Romanko, Jennifer Western and Carolyn Wong.

Patrick Bell is a new Associate Member. He spent 25 years in the counting room of Georgia Power and is now in management. He has attended TNI meetings in the past.

Jennifer Western is also a new Associate Member and is a laboratory manager for the St. Louis County Department of Health. She worked in a radiochemistry lab doing Drinking Water analysis. She started as an analyst in the lab, moved to Supervisor and now manages the lab in Missouri.

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

2. Small Laboratory Handbook (SLH)

Bob noted that TNI is trying to push up the schedule to make sure the SLH is finished before the end of the year because California will be implementing the 2016 Standard earlier and needs tools like this. Bob is hoping to finish the review today and get it to Ilona for cleanup. The document will then come back to the committee for a final review.

The committee started their review where they left off on Section 1.7.1.7. Attachment D includes the pages where updates were made during the meeting.

1.7.1.7 – Updates were made to the language as can be found in Attachment D.

Language was also added to Sections 1.7.2.1, 1.7.2.4 (example), 1.7.2.7 (example), 1.7.4 (example), and Appendix A. Deletions can be found in text boxes in Attachment D.

The cry for help to Tom in Appendix B should actually be to Vas. This is a section he was working on. Vas will have it back to Dave within the week.

There were no comments to review in Appendix C or D.

Bob asked that Dave update and cleanup the changes talked about in April and May and provided by Vas to Appendix B. It should then be forwarded to Ilona to do some editing

and formatting to the document. She will then hopefully get it back to the committee by the June meeting for review.

3. New Business

None.

4. Action Items

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

5. Next Meeting and Close

The next meeting is scheduled for June 28, 2017 at 1pm Eastern. There is no meeting scheduled for May 24th unless Bob finds a need to meet based on Dave's update.

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

The meeting was adjourned at 2:30pm 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*) **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	
84	Ilona will send the SLH back to the 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	

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.
- 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).

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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:

- 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 SOP.
- It is recommended that levels of contamination be confirmed by performing a background for subtraction prior to routine cleaning. An additional background measurement may not be needed if a detector is known to be contaminated.
- Contaminated detectors may not be brought back into service until corrective actions are completed, including determination of whether sample results have been impacted.

`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

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. Since QC requirements are often not specified by a source external to the laboratory (e.g., regulation or contract) it may be incumbent on laboratories to establish additional QC. When applicable, 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 point to MARLAP or other standards to explain the rationale for QC measures they select to use.

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- 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 needs to investigate the cause when results do not meet acceptance criteria and take corrective actions to eliminate the source or minimize the magnitude of the problem. The laboratory needs to consider samples associated with a failed QC parameter as suspect and needs to, wherever possible, reprocess such samples. Where reprocessing is not possible, the laboratory needs to 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.

Examples:

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 affects 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, 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 along with 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 result in systematically using or avoiding specific detectors.
   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. Rather, the sample is placed directly in a planchet 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 used for method validation studies (see Section 1.5). Put more simply, all samples should be analyzed for the same test and analytes, in the same counting geometry, and using the same process for calibration and background determinations. The same considerations regarding counting on multiple detectors and avoiding preferential use of detectors discussed for preparation batches apply here.

   Consider the following example: one air filter is collected and sent to the lab on a daily basis requiring gamma analyses. The laboratory may create an RMB beginning with the first sample and add samples to the RMB as they are received. Since only one sample is being collected daily, there will be 14 samples in the RMB. In addition to the samples in the RMB, the required quality control samples must also be counted during the 14 day period.

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2. Results for quality control samples are tracked and trended using statistical or tolerance control charts. Acceptance criteria are typically established by regulations, the method, or contract.

A statistical control chart might be appropriate when there is a need to characterize method performance or detect changes in performance over time that might indicate problematic performance. Statistical control charts, however, are not typically developed with the overall quality performance (bias and precision) parameters for an analytical method in mind. There are many valid approaches to statistical control charting that will yield valid results. One of the more frequently used approaches will be discussed here. Statistical control charts are usually based on a selected group of representative measurements for a given QC parameter, frequently 20 or more of the most recent observations. The mean value and standard deviation of the results are calculated, and warning and control limits, respectively, set at two (95.4%) and three (99.7%) standard deviations above and below the mean. Decisions to take some specific action are made when observed results occur at a higher frequency than would be expected. There is no single set of decision rules that meet all purposes, rather the rules applied should be appropriate for the process in question.

Let's assume that we have an LCS recovery result of 122%. Statistical control limits (3 sd) based on the 20 most recent measurements are 110 ± 42%. This tells us that we might expect the average result for the method to fall 10% above the true value and that we might expect to see 99+% of our LCS results fall within the range of 68 - 152%. Using this statistical control scheme, we would conclude that the LCS is in control since it is consistent with the observed performance of recent LCS results.

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It is important to keep in mind that statistical control charts are not generally sensitive to data quality requirements, rather they reflect observed performance of the parameter in question. Now let's also assume that we are working with a project that requires us to use a method for which LCSs fall within a tolerance of 25% of the known value. We would have to be concerned since we see that the observed performance of our method will regularly produce results outside the project's acceptable range.

We have a tolerance, so should we use a tolerance chart? ANSI N42.23 defines a tolerance chart as "A chart developed to evaluate the response of an instrument to a predetermined tolerance level as determined by an appropriate QC source. The predetermined tolerance level <…> is set with the overall quality performance (bias and precision) parameters for an analytical technique in mind." So we set up a tolerance chart with control limits set at ±25%. Using this tolerance chart, we would decide that our LCS meets project requirements but we have already seen that, in spite of this LCS performance, our method's performance is not adequate to meet project-defined MQO. It appears that the tolerance chart was not adequate for this purpose.

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

The standard also 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., any point outside control limits - probability ~3/1000) and possibly two-point trends (2 consecutive points in the warning zone (i.e., probability of a result ~ 2/1000). It is up to the laboratory to establish in their procedures the decision rules they will use to

- At a minimum, the laboratory needs to analyze one MD per Preparation Batch or RMB. For RMBs, the MD needs to consist of a second measurement of one sample. If the batch is counted on more than one detector, the MD needs to be performed on a second detector.
- When samples have low-levels of activity (less than approximately three (3) times the MDA) the laboratory, at its discretion, may analyze MS/MSD to determine reproducibility within a Preparation Batch in place of a MD.
- Based on specific project or program requirements or when there is insufficient sample available, the laboratory may choose to analyze a LCS in duplicate in place of a MD. The LCS and its duplicate will provide a measure of analytical precision. However, they will not provide information on matrix effects.

Chemical Yield Tracers and Carriers

- For those methods that employ a radioactive Tracer or a stable Carrier as a chemical yield monitor in the analysis, each sample needs to have an associated chemical yield calculated and reported. The chemical yield is one of the quality control measures to be used to assess the associated sample result acceptance.
- The selection of a Tracer or Carrier needs to not significantly interfere with the analyte(s) of interest nor cause bias in its measurements. When such a Tracer or Carrier is unavailable, the interference or bias caused needs to be quantifiable and appropriate correction applied to the sample results.
- The Tracer or Carrier used to monitor chemical yield needs to be added to the sample prior to performing any processes that affect the analyte of interest (e.g., chemical digestion, dissolution, ashing, separation, etc.) unless otherwise specified by the method.
- The chemical yield needs to be assessed against acceptance criteria specified in the method, regulation, contract or laboratory SOP. The laboratory needs to develop its criteria for data acceptance based on guidelines established in the MARLAP or other criteria such control charting developed by the laboratory. This assessment needs to meet established project or program measurement quality objectives.
- When the established chemical yield acceptance criteria are not met, the specified corrective action and contingencies needs to be followed. The occurrence of a failed chemical yield and the actions taken needs to be noted in the laboratory report.

Examples:

Matrix Spikes

1. The laboratory needs to document procedures for determining the effect of the sample matrix on the analytical results. This may be done by incorporating a MS in the preparation batch and/or a chemical yield tracer or carrier to every sample.
2. MSs, chemical yield tracers and carriers are not required for non-destructive methods.
3. For procedures which include a chemical yield tracers or carriers, the chemical yield tracer or carrier serves to determine if the matrix is interfering with the analytical processes, thus MSs are not required for these types of analyses.
4. The analytes in the MS should parallel those in the LCS. For example, if ^{230}Th is used to spike the LCS for gross alpha analyses, ^{230}Th should also be used for the MS.

1.7.2.6 Reagent Quality, Water Quality and Checks

In methods where the purity of reagents is not specified, reagents need to be analytical reagent grade or better. The quality of water sources needs to be monitored and documented and needs to meet method specified requirements. The QC program needs to establish and maintain provisions for radionuclide standards.

Key Points:

- Reference standards needs to be obtained from a national metrology institute (NMI), e.g. NIST in the USA or NPL in Great Britain, or from suppliers of NMI reference standards. Alternatively, reference standards may be obtained from an ISO/IEC Guide 34 accredited reference material provider, or an ANSI N42.22 reference material manufacturer.
- Reference standards needs to be accompanied with a certificate of calibration that meets the requirements of either ISO Guide 31, or ANSI N42.22, Section 8, Certificates and needs to include at least the following information: manufacturer, radionuclides calibrated, identification number, calibration method, activities or emission rates with associated uncertainties and the confidence limits, standard quantity, activity reference time (date or time as appropriate to the half-life of the radionuclide), physical and/or chemical description of the source, and radionuclide impurities.
- The laboratory needs to account for radioactive decay/ingrowth whenever decay/ingrowth has occurred between the Activity Reference Date and use that could impact use of the results.
- The laboratory needs to have written procedures for handling, storing and establishing expiration dates for reference standards.
- If there is no known provider of a particular standard (e.g., non-routine radionuclide or non-standard matrix), the laboratory may have no alternative but to use a standard with less rigorously established traceability. In this event, the laboratory needs to obtain from the provider the minimum information described the standards listed earlier. The laboratory needs to independently verify the activity of such standards prior to use and document the verification.
- If the laboratory's verification indicates a significant deviation from the original information from the provider, the standard should not be used unless the discrepancy can be resolved. If the standard is used for analysis of sample unknowns, the source and any other known limitations of the standard needs to be disclosed in the final report.

Example:

1. Because the half-life of tritium is only 12.32 years, the laboratory must decay correct the activity of the tritium standard when it is used to prepare an LCS so that the true activity of the LCS can be calculated.

1.7.2.7 Constant and Consistent Test Conditions

The laboratory needs to assure that test instruments consistently operate within the specifications required of the application for which the equipment is used. Labware needs to be cleaned to meet the sensitivity requirements of the method. Any cleaning and storage procedures that are not specified by the method needs to be documented in the laboratory's quality system and records. Note that some applications may require single-use glassware.

Key Points:

- The laboratory needs to maintain a radiological control program that addresses analytical radiological control.

- The radiological control program needs to explicitly define how low-level and high-level samples will be identified, segregated and processed to identify and minimize sample cross-contamination.
- The radiological control program needs to include the measures taken to monitor and evaluate background activity or contamination on an ongoing basis.

Example:

1. Laboratories should monitor trends in instrument background each day of use with the use of control or tolerance charts.

1.7.3 Data Evaluation and Reporting

Data acceptance and corrective actions requirements must be established for data review. The criteria may be established by the method, regulation, or by the laboratory. The laboratory should have specific protocol established for evaluating quality control samples that includes re-analysis of the samples, reporting sample data with qualification, or rejection of data. Corrective actions must be documented.

1.7.3.1 Negative Control – Method Performance: Method Blank

MB results needs to be evaluated for long term trends, absolute bias, possible contamination, or interferences that may affect results for samples in the batch.

Key Points:

- If acceptance limits are not met, corrective actions need to be taken to investigate the source of contamination or other bias. If sample activity levels are greater than five (5) times the activity found in the MB, lacking other requirements, it is acceptable to report qualified results for the samples associated with the blank. Otherwise, reprocessing and reanalysis of the associated samples needs to be required.
- When sample results associated with a failed MB are reported, the failure and associated corrective actions, or inability to complete corrective actions, needs to be noted in the laboratory report.
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Examples:

1. A method blank needs to be performed at a frequency of one per preparation batch. The results of this analysis are one of the quality control measures to be used to assess batch acceptance. Corrective actions must be taken when 1) the MB result is significantly different from zero (criteria defined by the lab) and associated sample results are less than five (5) times the MB activity, or 2) when a MB result may impact the analytical results. The corrective actions to be taken must be defined by the laboraotry. Often, laboratories re-prepare and reanalyze all affected sample in the batch. The occurrence of a failed method blank acceptance criterion and the actions taken needs to be noted in the laboratory report.
2. The batch method blank result may not be subtracted from sample results in the associated preparation or RMB. The laboratory may, however, subtract the average historical activity of method blank measurements to address a demonstrated bias. This correction must be applied to all analyzed samples, including quality control samples, and the uncertainty associated with the correction must be accounted for in the total uncertainty reported with the results.
3. When the aliquot size for the method blank varies from that used for routine sample, acceptance criteria needs to address the presumed aliquot size on which the method blank result is calculated and the manner in which the method blank result is compared to sample results of differing aliquot size.

1.7.3.2 Positive Control – Method Performance: LCS

LCS recoveries need to be evaluated to assess the performance of the entire analytical system independent of the sample matrix. LCS results needs to be calculated in percent recovery or other appropriate statistical measure that allows comparison to established acceptance criteria. The laboratory needs to document the calculation.

Key Points:

- An LCS that is determined to be within established acceptance limits effectively demonstrates that the analytical system is in control and validates system performance for the samples in the associated batch.
- Samples associated with an LCS that fails to meet acceptance limits are considered suspect and the samples needs to be reprocessed and reanalyzed.
- If samples cannot be reprocessed and reanalyzed, the failure and associated corrective actions or inability to complete corrective actions needs to be noted in the laboratory report.

Examples:

1. The laboratory shall spike the LCS at a level such that the uncertainty of the analytical result is less than one-third of the acceptance criteria. For example, if it is required that the LCS result be within +/- 30% of the known value, the laboratory shall spike the LCS at a level such that the uncertainty of the analytical result is less than or equal to 10%. When practical, the LCS should be spiked at a level comparable to the action level if known; or that of routine samples if the activities are expected to exceed ten (10) times the Decision Level (Critical Value).
2. The laboratory standards used to prepare the laboratory control sample needs to be from a source independent of the laboratory standards used for instrument calibration.

1.7.3.3 Sample-Specific Controls

Key Points:

Matrix Spike, Matrix Duplicates, Matrix Spike Duplicates

- MSs and MDs allow evaluation of the effect of matrix on the accuracy and precision of results.
- When results fall outside established criteria, corrective actions must be documented and the data reported with appropriate data qualifying codes. QC results outside acceptance limits must be noted in the laboratory report.

Tracers and Carriers

- Tracers or stable carriers monitor chemical yield in the sample with the results expressed as percent yield or other appropriate statistical measure that allows comparison to established method acceptance criteria.
- For alpha spectrometry, evaluation of Tracer acceptability needs to include evaluation of chemical yield (e.g., uncertainty, variability) and peak resolution.
- Samples associated with Tracers or Carriers that fail to meet acceptance limits are considered suspect, and the samples needs to be reprocessed and/or reanalyzed. If samples cannot be reprocessed and/or reanalyzed, the failure and associated corrective actions or inability to complete corrective actions needs to be noted in the laboratory report.

Key Points:

1. In general, MS need not be run unless a carrier or tracer is not used for chemical yield correction. The results of this analysis need to be one of the quality control measures to be used to assess batch acceptance. The matrix spike result needs to be assessed against the specific acceptance criteria when the specified matrix spike acceptance criteria are not met. The specified corrective action and contingencies will be followed. The occurrence of a failed matrix spike and the actions taken need to be noted in the laboratory report. The lack of sufficient sample aliquot size to perform a replicate analysis should be noted in the laboratory report.

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2. The laboratory standards used to prepare the matrix spike needs to be from a source independent of the laboratory standards used for instrument calibration.

1.7.3.4 Evaluation of Sample Results

Instrument raw data from energy spectral analysis needs to be evaluated to ensure that the target radionuclides are quantified consistent with laboratory procedures and applicable measurement quality objectives, and that target radionuclides in the spectra are evaluated for possible interferences. Results need to be reviewed for internal consistency, such as the presence of radionuclides consistent with known parent-progeny relationships and expected or likely decay series.

Key Points:

- Sample-specific estimates of uncertainty and MDA need to be evaluated to ensure that MQOs have been met.
- If objectives have not been met, then samples need to be reprocessed and/or reanalyzed.
- If samples cannot be reprocessed and/or reanalyzed, the failure and associated corrective actions, or inability to complete corrective actions, needs to be noted in the laboratory report.

1.7.3.5 Reporting Results

Following evaluation according to Section 1.7.3.4, results need to be reported as obtained with appropriate units, even if the results are negative. Results need to be reported with an appropriate number of significant figures and an estimate of uncertainty. The result needs to also include the Activity Reference Date in association with all radiochemical measurement results. The date listing may be a simple comment in the case narrative as long as it unambiguously defines the date for the reported results. Project or client specified reporting requirements can take precedence over the requirements in the Standard.

Note: Although the above criteria have a solid technical basis and rationale, specific regulations and programs may have requirements that would supersede them.

1.7.4 Sample Handling

While it may not be possible to physically verify all methods of preservation (e.g., addition of oxidizing or reducing agents), wherever practicable the laboratory needs to verify that samples have been preserved in compliance with all applicable requirements specified by regulation, method, or contract, or as established in the laboratory's quality system (if there are no established mandatory criteria).

Key Points:

- The laboratory needs to document the required timing, methods for performing measurements to verify preservation, the acceptance range, or any other conditions indicating acceptable preservation.
- Where thermal preservation of samples is required, the laboratory needs to verify the temperature of samples upon receipt.
- Where chemical preservation of samples is required, the laboratory needs to verify that samples have been preserved using readily available techniques such as pH measurement prior to sample preparation or analysis.
- If the results of the preservation verification do not satisfy established criteria, the laboratory needs to initiate corrective actions (i.e., notification of the client, preservation of the sample at the time of discovery), and qualify all impacted test results in the report to the client.

Examples:

1. The laboratory's written procedure for sample receiving needs to include a list of requirements for acceptable types of sample containers, minimum sample volumes, thermal and chemical preservatives, and maximum holding times for each radiochemical analysis the laboratory performs. The procedure should also indicate analytical parameters for which chemical preservatives should not be added to samples (e.g., acid preservation of carbon-14 in water).

2. The sample receiving procedure needs to detail how pH measurements are to be conducted and documented.

3. The sample receiving procedure needs to also describe the steps to be taken when sample acceptance criteria are not met, such as documentation of discussions with the client to either reject samples or proceed with analysis and qualify results on the final report.

APPENDIX:

A. MINIMUM DETECTABLE ACTIVITY

Radiochemical data is often reported to include minimum detectable activity (MDA) with sample results. The MDA is an a priori estimate of the detection limit for a method. It may be used to select a method that can meet Measurement Quality Objectives (MQOs) for detection capability.

Radiochemical data is sometimes reported in association with a sample-specific MDA which reflects analytical factors used to calculate the sample result. The sample-specific MDA should only be used to determine if the analysis, as run, meets the required Measurement Quality Objectives (i.e., required MDA).

A number of factors can adversely affect the MDA. Inadequate sample volume, short counting time, low detection efficiency all can affect the MDA individually or together. The laboratory must have procedures in place for meeting and reporting MDA. The 2009 TNI Standard requires that a laboratory establish criteria for reporting MDA when such criteria are not found in the method or a regulation. Additionally, projects involving cleanup of contaminated sites often include MDAs in the contract specifications. The laboratory needs to comply with the contract specifications.

There is no single formula for MDA. Several variants of nearly the same formula are in use in the industry. Following is an example of an MDA calculation.

A laboratory received a 1 L wastewater sample from one of its customers. The chain of custody indicated that it was a ground water sample from site near an operating nuclear power plant. The analysis required on the sample is Cs-137.

With the above information, the laboratory analyzed the wastewater sample using EPA 901.0 method. The method involved adding stable cesium carrier followed precipitation of Cesium-137 and gamma spectrometry using HPGE detection system. The identification of Cesium-137 and quantitation was via the 662 keV gamma-ray emission. The planchet geometry helped to achieve excellent results. The following data was gathered.

Sample volume: 1 L

Chemical Yield: 80%

Counting Efficiency, Cs-137: 25%

Sample counting time: 100 Min.

Reagent Blank (for Background) counting time: 100 Min.

Reagent Blank counts: 196 counts in 100 Min. MDA is calculated using paired measurements equation.

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$$\mathrm{MDA} = \frac{2.71 + 4.65\sigma_B}{CY \cdot EFF \cdot V \cdot 100 \cdot 2.22}.$$

Substituting the data into the formula, an MDA of 1.5 pCi/L would be obtained.

B. METHOD VALIDATION STUDY

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A laboratory is an accredited NELAP laboratory. The laboratory is seeking accreditation for Gross Alpha analysis in drinking water by co-precipitation method. The laboratory performed a method validation study and documented the results. Following is an excerpt from the study for illustrative purposes.

Method: Determination of for Gross Alpha Radioactivity in Drinking Water by
Reference Method: SM 7110 C, Co-precipitation Method
Applicable Matrix: DW

This study includes the following:

A) Detection Limit study,
B) Precision & Bias study,
C) Measurement Uncertainty,
D) Selectivity, and
E) Analysis of an external QC (or a PT) Sample.

A) DETECTION LIMIT STUDY:

When analyzing drinking water samples for compliance monitoring purposes under Safe Drinking Water Act (SDWA), the Alternate Test Procedure requires the DL for the method to be determined to ensure it meets the requirements of the SDWA.

[Note: Some laboratories continue to report minimum detectable activity concentration, (MDA or MDC) for all analysis including drinking water. Those laboratories must implement SDWA DL to be in compliance.]

The SDWA DL is defined in the 40 CFR Part 141.25(c) as 'that concentration which can be counted with a precision of ±100% at the 95% confidence level (1.96σ where σ is the standard deviation of the net counting rate of the sample)'.
A generalized form of the equation for SDWA DL[1] is as given below to be used for this example.

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$$\mathrm{SDWA\ DL}\left(\frac{\mathrm{pCi}}{\mathrm{L}}\right) = \frac{1.96^2}{2t_G} \cdot \frac{1 + \sqrt{1 + \frac{4t_G^2}{1.96^2} R_B \left(\frac{1}{t_G} + \frac{1}{t_B}\right)}}{2.22(\mathrm{Efficiency})(\mathrm{Volume})(\mathrm{Chemical\ Recovery})}$$

Where,

- Volume of the sample is in L. It is recommended to use 1.0 L for co-precipitation method.
- Chemical recovery refers to gravimetric recovery of the co-precipitate (radium-barium sulfate). We will assume 100% recovery for this example. In reality, a chemical yield of 90 – 95% is routinely achieved.
- 2.22 is conversion factor for DPM to pCi.