Radiochemistry Expert Committee (REC)
Meeting Summary – San Antonio, TX

August 6, 2013

1. Roll Call and Minutes:

Bob Shannon, Chair, called the meeting to order at 9am CT in San Antonio, TX. Attendance is recorded in Attachment A – there were 9 members present. Associate members present included: Terry Romanko, Virgene Mulligan, Brian Miller and Joe Pardue.

The minutes from the June 26, 2013 meeting were reviewed. Dave motioned to approve the minutes and Carolyn seconded the motion. Vote: For – 8 Against – 0 Abstain – 1 (Tom Semkow). The motion was passed.

Associate members need to let Bob and Ilona know they own a copy of ISO 17025 so they can be included in distributions of the draft working standard updates.

2. Terms

From: TNI SOP 1-101

2.5 Committee Members Terms of Appointment

2.5.1 Committee Members are appointed to three-year terms that are staggered so that members rotate off each year. Any member who has completed a first term may be nominated for a second three-year term, but no member may serve more than two (2) three-year terms consecutively. If a member resigns before his/her term of appointment is complete, the Committee Chair may nominate another individual from the same or another organization to complete the resigning member's term.

1 year: Richard, Tom, Carolyn

2 year: Keith, Larry, Dave

3 year: Marty, Bob, Todd, Vas, Nile

3. Standard Review

Bob Shannon distributed fifteen handouts by e-mail on August 3, 2013. He also distributed an updated copy of the standard and an agenda. The standard and handouts

were projected on the screen (and through Webex for members who called in). Changes discussed were made directly into the standard (base document) and an updated copy will be distributed to members after the meeting. This updated standard should be considered a summary of this meetings discussions and results and a copy can be obtained by contacting the Chair (Bob Shannon) if the inquirer owns a copy of ISO 17025. The information below summarizes the handouts originally submitted and some comment on the handouts.

V1M6 – Definitions in Working Draft (see handouts)

Measurement quality objectives. Paul agreed that this would be appropriately defined in Module 6 and not placed in Module 2. Bob will provide this text to the CSDP also to see if it might be of interest to the other modules. Tom highlighted that the Radiochemistry example might have to be removed or modified.

DQO is used in multiple standards. Paul Junio (Chair of Quality Systems Expert Committee) suggested a change to the definition. The second line should read "the directed planning process" instead of DQO. Bob will add this to the base document.

Paul commented that DQO should be defined in module 2 because other modules use this definition. Paul suggested that if the definition is needed now, a TIA should be looked at. This can be discussed in a Consensus Standards Development Program Expert Committee (CSDP EC) meeting. The Microbiology and Chemistry Expert Committees should be involved in this too.

V1M6 – Section 1.5.2 - Richard

Richard proposed the following language for 1.5.2 with the goal to put it in active voice:

The laboratory shall establish the detection capability for each method / matrix combination. Detection capability may refer to critical value, Minimum Detectable Activity (MDA), and SDWA DL (all terms are defined in Section 1.3.1) The laboratory shall document the procedure used to determine the detection capability. The laboratory shall record the quality system matrix used in the initial method validation and retain all supporting documentation for the initial study in a readily retrievable format for the lifetime of the method. The procedure a laboratory uses to determine the detection capability of a method must comply with the specific requirements of Sections 1.5.2.1 and 1.5.2.2. Method validation documentation must also include identification of software used for detection capability calculations and the software must conform to the requirements in Module 1 Volume 2 Section 5.4.7.2.

He also proposed an additional language change to 1.5.2.1 (b): The laboratory shall initially determine the detection capability of each method for the analysis of interest ….

Also 1.5.2.1 (c): The laboratory shall determine the detection capability each time there is a change …

To 1.5.2.2 (b): The word “approved” was added to methods.

1.5.2.2: Virgene asked why detection limit studies are being done in Radiochemistry. Larry commented it is a requirement under the Safe Drinking Water Act. Bob commented that a detection limit study would look at a distribution of results around zero. Richard commented that this section is Method Validation and he noted that it is important to understand initially what detection capability is possible. This is only done at the start-up of a method or when a method is modified.

PT – Larry and Tom

Language Proposed: For all methods, except reference methods, the laboratory shall analyze, as available, externally produced quality control samples from a national *metrology institute or a nationally or internationally* recognized source (e.g., accredited TNI PT Providers *or ISO 17043 accredited provider*) or from commercial vendors complaint with ANSI N42.22. The laboratory shall evaluate results of these analyses to determine its ability to produce acceptable data.

It was recommended that “or internationally” be added and ISO 17043 be added.

George Detsis asked about the term “external”. It is intended to indicate the source is not within the laboratory.

The language above will be added to the base document.

V1M6: Section 1.7.2 – Carolyn and Marty

Carolyn provided an update to this section at the meeting. Bob was able to bring the new document up on the screen for everyone to review.

The language submitted was reviewed and through discussion the following language was decided upon:

1.7.2 Quality Control for Radiochemistry

The laboratory shall have quality control procedures for reviewing and monitoring the validity of environmental tests undertaken as specified in this Section.

Results of the quality control samples shall be assessed against the acceptance criteria published in the mandated method or client specified criteria. Where there are no established criteria, the laboratory shall determine such internal criteria and document the method used to establish the limits in the laboratory report.

When the specified acceptance criteria are not met, the laboratory’s corrective action procedures shall be followed. Any affected samples associated with an “out of control”

quality control sample shall be reprocessed for reanalysis or the results reported with appropriate data qualifying codes.

The occurrence of a failed quality control sample and any associated actions shall be noted in the laboratory report.

It was asked if the committee will be looking at standardizing data qualifying codes. This is not something the committee plans to do.

1.7.2.1 Negative Control – Method Performance Method Blank

Carolyn and Marty added two additional paragraphs they would like to see added to the standard. The paragraphs were reviewed and the following language was determined:

Blanks are used in two different ways, depending on the method. Matrix-appropriate or reagent blanks are subtracted from the gross results to account for contributions from the total analytical measurement process. Batch method blank analysis results are assessed to evaluate the existence and magnitude of possible contamination problems.

The method blank result should be less than 1.65 times its CSU, or other established acceptance limits; if not, batch method blank results shall be evaluated for trends that indicate possible contamination or absolute bias.

Bob suggested putting it in the section on Background Subtraction to avoid confusion on which blank is being referred to. Move to Section 1.7.3.

A concern was raised about the term "net counts" and this was changed to "net activity".

Each time a blank is mentioned, the term needs to be clearly defined. It is causing confusion. Larry and Richard read the definition for Blank in the standard.

a) The method blank is used to assess the preparation batch for possible contamination during the preparation and processing steps or for other low-level bias. The method blank shall be processed along with and under the same conditions as the associated samples to include all steps of the analytical procedure. Procedures shall be in place to determine if a method blank result is significantly different from zero.
b) The method blank shall be analyzed at a minimum of one (1) per preparation batch. The method blank shall be prepared in a quality system matrix that is similar to the associated samples and is known to be as free of the analytes of interest as possible with an aliquot size similar to that of the routine samples for analysis.

d) In the case of gamma spectrometry a method blank shall be prepared using a calibrated geometry similar to that used for samples. The container of the

appropriate geometry should be filled to a similar volume as the samples to partially simulate gamma attenuation due to a sample matrix.

e) There shall be no subtraction of the method blank result from the sample results in the associated preparation or analytical batch.

Some concerns were raised about how blanks are presented in the module. Carolyn and Marty will take this concern and draft language to change.

V1M6: Section 1.7.2.3 – Nile and Vas

Vas and Nile prepared changes to the section as outlined below (italics). Accepted changes were made directly into the standard (base document):

1.7.2.3 Sample-Specific Controls

First paragraph: The laboratory shall document procedures for determining the effect of the sample matrix *on the analytical results* ~~method performance~~.

Second paragraph: Examples of matrix-specific QC include: Matrix Spike (MS); Matrix Spike Duplicate (MSD); and *sample* replicates.

Section a) Matrix Spike:

ii) The frequency of the analysis of matrix spikes are as specified by the method, *a regulation* or *it* may be determined as part of the contract review process**.** *Where there are no established criteria in the method, a regulation or contract, the laboratory shall develop its criteria for matrix spike analyses and document in the method SOP.*

iii) The components to be spiked shall be as specified by the mandated method or *regulation.* Any permit specified analytes, ~~as specified by regulation~~ or client requested analytes shall ~~also~~ be included.

iv) The lack of sufficient sample aliquot size to perform a matrix spike shall be noted in the laboratory report *for that sample. The laboratory shall make effort to include one matrix spike in a batch of 20 or less samples of a quality system matrix.*

v) The activity of the matrix spike analytes(s) shall be greater than five times the MDA *and it shall not be at the same level of LCS activity for that batch.*

Section b):

b) Matrix Spike Duplicates / Replicates / LCS Duplicates

i. Replicates are defined as ~~replicate~~ *multiple* aliquots of the same sample taken through the entire analytical procedure. The results from this analysis indicate the *measurement* precision of the *analyte* ~~results~~ for the specific sample using the selected method. *Replicate analyses* provide the most useful measure of precision when target analytes are ~~found~~ *present* in the sample chosen for replication.

ii. The frequency of the analysis of matrix replicates and duplicates are as specified by the method or may be determined as part of the contract review process. *Where there are no established criteria in the method, a regulation or contract, the laboratory shall develop its criteria for replicate and duplicate analyses and document it in the method SOP. The laboratory shall make effort to include one duplicate or matrix spike duplicate in a batch of 20 or less samples of a quality system matrix.*

~~iii. Replicates are performed on replicate aliquots of actual samples.~~

iv. For low-level samples (less than approximately three times the MDA) the laboratory may analyze a laboratory control samples *in* duplicate or a replicate matrix spike (matrix spike and a matrix spike duplicate) to determine reproducibility within a preparation batch in place of a sample replicate. In addition based on project or program requirements, the laboratory may analyze a laboratory control sample duplicate or a matrix spike duplicate in place of a sample replicate.

Section c):

c) Tracer

For those methods that employ a radioactive tracer *in the analysis,* ~~for yield determination,~~ each sample shall have an associated tracer yield calculated and reported. *The selection of a tracer shall be such that it does not interfere with the analyte(s) of interest nor cause bias in its measurements.*

Section d):

d) Carrier

For those methods that utilize a *stable (non-radioactive)* carrier for yield determination, each sample shall have an associated carrier yield calculated and reported. *The selection of the carrier shall be such that it does not interfere with the analyte(s) of interest nor cause bias in its measurements.*

V1M6: Sections 1.7.2.4, 1.7.2.5 and 1.7.2.6 – Larry and Dave

The following changes were proposed and reviewed during the meeting. The language was updated in the standard (base document).

Section 1.7.2.4:
Changes were made to this section and then all text including and below "Measurement Uncertainties …. " was proposed to be moved to Section 1.5.4. ISO Guide 98 was deleted and the following substituted: BIPMJCGM 100:2008.

Keep a) and add b) and c):

b) Detection levels (MDC, MDA, or MDL, as appropriate) shall be calculated as described in Section 1.5.2.
c) Measurement uncertainties shall be calculated and reported as described in Section 1.5.4.

Section 1.7.2.5:

Add "Reference Standards" to title of this section.

Section c): Make the following changes in italics:

i) Reference standards that are used in a radiochemical laboratory shall be obtained from NIST or suppliers *of* NIST standards or NIST traceable radionuclides. *Alternatively, reference standards may be obtained from suppliers outside the United States, provided that the standards are traceable back to each country's national standards laboratory.*

ii) Reference standards shall be accompanied with a certificate that includes at least the following information: manufacturer, radionuclides calibrated, identification number, calibration method, activities or emission rates with associated uncertainties and the confidence limits, calibration or reference date and time (if appropriate for the half-life of the radionuclide), physical and/or chemical description of the source, and radionuclide impurities (reference ANSI N42.22 – 1995, Section 8, Certificates).

iii) *Standards shall be verified prior to initial use.* Laboratories ...

iv) If there is no known provider of a particular standard (e.g., non-routine radionuclide or non-standard matrix) that is traceable to the International System of Units (SI), the laboratory may be forced to use a less traceable standard with less well established uncertainties. In this event, the laboratory will obtain from the provider the minimum information described in Section 1.7.2.5 c) ii), and will undertake to independently verify and document that information. If the laboratory's verification indicates a

significant deviation from the original information from the provider, the standard should not be used. If the standard is used for analysis of sample unknowns, the source and any other known limitations of the standard shall be disclosed in the final report.

Section 1.7.2.6: Other than bolding title, leave as is.

V1M6: Section 1.7.3 – Larry and Dave

Larry and Dave prepared the following changes to this section. The standard (base document) was updated as appropriate:

Section 1.7.3.3 a) ii): ... used to establish the limits *or utilize client specified assessment criteria.*

Section 1.7.3.3 b): There were some numbering and formatting issues corrected.

Section 1.7.3.3 b) ii): used to establish the limits *or utilize client specified assessment criteria.*

Add sections 1.7.3.3 c) and d) to keep in the format established for other QC. The statements about evaluation of tracers should be removed from 1.7.2.3 c) and the statements about evaluation of carriers should be removed from 1.7.2.3 d). The text below is meant to replace it. Nile and Vas also worked on language for 1.7.2.3 c) and d) (see above) and this was considered in the discussion when putting language into the base document.

c) Tracer (if used)

i) The results from tracers are used to monitor chemical yield in each sample. Results are expressed as percent recovery (%R) or other appropriate statistical technique that allows comparison to established criteria. For alpha spectrometry, evaluation of acceptability also includes evaluation of peak resolution, such as peak width at one-half peak height (FWHM). The laboratory shall document the calculation for %R, FWHM, or other statistical treatment.

ii) Results are compared to acceptance criteria as published in the mandated method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits or utilize client specified assessment criteria. Samples with a tracer result determined to be "out of control" shall be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate qualifying codes.

iii) The occurrence of a failed replicate and any associated actions shall be noted in the laboratory report to the client.

d) Carrier (if used)

i) The results from tracers are used to monitor chemical yield in each sample. Results are expressed as percent recovery (%R) or other appropriate statistical technique that allows comparison to established criteria. The laboratory shall document the calculation for %R, FWHM, or other statistical treatment.

ii) Results are compared to acceptance criteria as published in the mandated method. Where there are no established criteria, the laboratory shall determine internal criteria and document the method used to establish the limits or utilize client specified assessment criteria. Samples with a tracer result determined to be "out of control" shall be considered suspect and the samples reprocessed and re-analyzed or the data reported with appropriate qualifying codes

iii) The occurrence of a failed replicate and any associated actions shall be noted in the laboratory report to the client.

V1M6: Section 1.7.4 – Marty and Bob

All language in Section 1.7.4 was stricken.

V1M6: Section 1.7.1 – Tom, Bob, Vas

The section was re-written and distributed by Bob via e-mail. The entire section was forwarded, reviewed and changes were made as appropriate to the standard (base document).

4. Action Items

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

5. Next Meeting and Close

The next meeting will be scheduled by e-mail, but is expected to be Wednesday, August 28, 2013 at 1pm EST.

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

The meeting was adjourned at 5pm CT.

Attachment A
Participants
Radiochemistry Expert Committee

Members	Affiliation		Contact Information	
			Phone	Email
Bob Shannon (Chair) **Present**	QRS, LLC Grand Marais, MN	Other	218-387-1100	BobShannon@boreal.org
Tom Semkow (Vice Chair) **Present**	Wadsworth Center, NY State DOH Albany, NY	AB	518-474-6071	tms15@health.state.ny.us
Sreenivas (Vas) Komanduri **Present - Phone**	State of NJ Department of Environmental Protection Trenton, NJ	AB	609-984-0855	Sreenivas.Komanduri@dep.state.nj.us
Marty Johnson **Present**	US Army Aviation and Missile Command Nuclear Counting Redstone Arsenal, AL	Lab	865-712-0275	Mjohnson@tSC-tn.com
Dave Fauth **Present**	Consultant Aiken, SC	Other	803-649-5268	dj1fauth@bellsouth.net
Carolyn Wong **Present**	Lawrence Livermore National Laboratory Livermore, CA	Lab	925-422-0398	wong65@llnl.gov
Keith McCroan **Present**	US EPA ORIA NAREL, Montgomery AL	Lab	334-270-3418	mccroan.keith@epa.gov
Todd Hardt **Absent**	Pro2Serve, Inc. Oak Ridge, TN	Other	865-241-6780	HardtTL@oro.doe.gov
Nile Ludtke **Absent**	Dade-Moeller and Associates Oak Ridge, TN	Other	865-481-6050	nile.luedtke@moellerinc.com
Larry Penfold **Present**	Test America Laboratories, Inc; Arvada, CO	Lab	303-736-0119	larry.penfold@testamericainc.com
Richard Sheibley **Present**	Sheibley Consulting, LLC	Other (Former AB)	651-485-1875	RHSHEIB111@yahoo.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	Actual Completion
2	Richard will look at all of 1.5.2 (including 1.5.2.1) and propose some new language. He will review it with Nile before submitting to committee. (2/27/13: Carolyn and Tom also asked to review this before submission to the committee.)	Richard	2-26-13	Complete
3	Richard will prepare language update for 1.5.3 and submit to committee.	Richard	2-26-13	
10	Prepare definition for "activity" based on today's conversation.	Bob	5/22/13	
11	Complete and distribute language proposed for 1.7.1.	Bob Tom Vas	~~5/22/13~~ ~~To be finished for 6/26/13 meeting.~~ Next Meeting	In Progress
13	Does a PT Provider need to be a TNI PTPA approved provider? Language will be worked on and discussed at next meeting.	Larry Tom	6/26/13	Complete
15	Work on language for Section 1.7.2.	Carolyn Marty	Next Meeting	Complete
16	Work on language for Section 1.7.2.3.	Nile Vas	Next Meeting	Complete
17	Work on language for Sections 1.7.2.4, .5 and .6.	Larry Dave	Next Meeting	Complete
18	Work on language for Sections 1.7.3.	Larry Dave	Next Meeting	Complete
19	Work on language for Section 1.7.4.	Bob Marty	Next Meeting	Complete
20	Bob will update Standard/Base Document. All should review and comment to Bob.	Bob All	8/28/13	
21	Work on presentation of blanks in the module.	Carolyn Marty	8/28/13	

Attachment C – Back Burner / Reminders

	Item	Meeting Reference	Comments
1	Update charter in October 2013	n/a	
2	Issue of noting modifications to methods.	1/16/13	
3	Look at batching when QC is looked at.	1/16/13	
4	Look at need to reference year for any standard references– which version is being referenced. Is this necessary?	5/22/13	