# Radiochemistry Expert Committee (REC) Meeting Summary

# April 23, 2014

### 1. Roll Call and Minutes:

Bob Shannon, Chair, called the meeting to order at 1:05pm EST on April 23, 2014. Attendance is recorded in Attachment A – there were 8 members present. Associate members: Joe Pardue, Terry Romanko, Brian Miller, Jeffery Reed, Virgene Mulligan, Ron Houck, and guests, Martha Smith (NJ) and Sharon Robinson (NJ).

The April 9, 2014 minutes were reviewed. A motion was made by Dave to accept the minutes. The motion was seconded by Vas. Vote: 7 – For, 0 – Against, 1 – Abstain (Tom did not receive them). The motion was unanimously approved.

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. Standard

# 1.5.4 – Language for Uncertainty

Keith and Bob worked on the language after the last call:

- 1.5.4 Measurement of Uncertainty
- a) All radiochemical measurements shall be reported with an estimate of the uncertainty of each quantitative measurement result.
- i) All radiochemical measurements shall be reported with estimates of the combined uncertainty of measured results. The combined uncertainty is the uncertainty of a measured value expressed as either an estimated standard deviation, called the combined standard uncertainty, or a multiple thereof, called an expanded uncertainty. The procedure for determining the combined measurement uncertainty shall be documented and shall be consistent with BIPM JCGM 100:2008: Guide to the Expression of Uncertainty in Measurement (GUM) or the recommendations in the Multi-Agency Radiological Laboratory Analytical Protocols Manual Chapter 19 (MARLAP, Volume II, EPA 402-B-04-001B, July 2004).
- ii) For purposes of demonstrating compliance with the Safe Drinking Water Act, and consistent with requirements established by regulators, laboratories may report the counting uncertainty as defined in the appropriate reference method and documented in the laboratory SOP.

b) The report shall clearly specify the type of uncertainty reported. The report shall: i) indicate whether the uncertainty is the combined standard uncertainty (CSU) or only the counting uncertainty; and ii) for expanded uncertainties, indicate the coverage factor (k) or the level of confidence.

Tom asked if the laboratory has to report both uncertainties – combined and counting. Larry thought it could be only counting uncertainty.

The language above is supposed to keep the reporting options open depending on the individual requirements of each lab. It was written to satisfy any regulation — could be combined or counting or just counting or just combined. Tom commented that it is open for the assessor's interpretation. The word "may" is used. This could be a problem. Richard pointed to the language in the first sentence in b). The report specifies the uncertainty and this should not be a problem.

Keith suggested rewording to make it clear. He will work on the language and share it by email for finalization.

# Status on Language about Random Processing of Samples/QC Samples (Tom, Vas, Richard, Marty, Bob)

This was looked at and reviewed by email during the last two weeks. This is the last version sent by Bob:

1.7.2 ...The laboratory shall process all batch quality control samples together with, and under the same conditions as the associated samples, and shall use the same processes and procedures for preparation, analysis, data reduction and reporting of results. The laboratory shall have procedures to ensure that specific detectors, equipment or glassware are not used systematically or preferentially for the analysis of quality control samples. Where best laboratory practices recommend segregation of detectors, equipment or glassware to prevent cross-contamination of samples, such practices are allowed as long as the criteria for segregation applies equally to batch quality control samples and samples.

Vas did not like the word segregation in the last two lines. The definition was reviewed and it was decided that the term was the most appropriate. Bob thought it was important to make it obvious (use "segregation") because labs don't follow this.

### **Status 1.7.3.4 (Larry)**

Larry has not received any additional comments, but Tom received one comment from Tom by email.

Tom thinks there is an issue in language consistency – there is a duplication in definitions. Sometimes the term "sample duplicate" is used and other times "matrix duplicate" is used. It should be the same throughout the document. He prefers "matrix duplicate".

# 3. Discussion on Batches Following Louisville

Bob reviewed the information from the last meeting and provided a document in Attachment D to review his proposal. He walked the committee through the document.

2) ii) The 14 days are only a suggestion. Bob would like feedback.

Bob also noted that in Section 1.7.1 there is QC that runs in parallel.

Tom also provided a document included in Attachment D and he walked everyone through the different versions. Tom took what he thought was the best of each of the versions and provided the last proposal.

Tom asked what is "destructive analysis"? In his opinion grinding is not destructive analysis. It is only a way to achieve homogeneity in the sample. Sieving would be destructive because you would segregate certain fractions.

Vas disagreed and commented that all the items listed in the first paragraph (grinding, sieving, etc.,) are all destructive. Some sample preparation is involved. Calling grinding non-destructive is a stretch.

Terry commented that perhaps the words "destructive analysis" are not needed. Instead, something like: When sample preparation is performed that includes physical or chemical processing of the sample, such as grinding, sieving, evaporation or chemical separation, etc ....

An example was given where tritium analysis in concrete was requested. It was found that the tritium was being lost because of the heat while grinding the concrete. They had to devise a process to keep it cool.

Terry was concerned that contamination or potential loss of the analyte should be considered. He does not think the issue is whether it is destructive or non-destructive. Carolyn agreed that the wording suggested by Terry above should be considered. This is in her version in Attachment D.

Bob suggested removing reference to "destructive" and use language similar to Carolyn's. He also commented that grinding is not just putting the sample directly on the instrument. It is prep. There was further discussion on what is purely analytical and what is preparation. This affects the definition of analytical batch.

Terry will work with Bob on a revision of the language. Bob asked people to email him if they would like to participate on an extra call on this topic to prepare for the next meeting.

# 4. Review of Module 6 beginning with Section 1.7

Bob distributed this document by e-mail and did not receive any comments. People asked for more time. Bob asked that people send him comments before the next meeting for inclusion in the next call.

Larry asked if the committee could look at line 208: "... not exceeding in size ..." – what is the intent?

From the standard:

3) An individual test source, or a batch of sources not exceeding in size the preparation batch, analyzed using automated instruments such as liquid scintillation or gas proportional counters, may be uninterruptedly measured for a longer time than the routine interval between performance checks as long as the checks are done at the beginning and end of the test source or batch measurement and both checks meet all applicable acceptance criteria..

Tom commented that the language above allows a batch to finish before the next instrument check is done. The batch could take days to finish. Through discussion, it was commented that this refers to counting time – not number of samples. This language was a result of a concern on the earlier language that limited it by time.

This will be looked at when the batch discussion continues. Bob commented that there are other parts of the standard that still have time windows. This needs to be looked at for consistency and good practices.

There were no further comments. This section will continue to be discussed by email.

### 5. PT Committee Discussions

The subcommittee updating the procedures for establishing FoPT limits will be meeting in the next couple of weeks.

# 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 will be May 7, 2014 at 1pm.

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

The meeting was adjourned 2:22 pm EST. Motion: Larry Second: Vas Unanimously approved.

# Attachment A Participants

Radiochemistry Expert Committee

| Mambara Affiliation   |  |                      | Contact Information |   |  |
|---|--|----------------------|---------------------|---|--|
| Members   | Affiliation  |                      | Phone               | <u>Email</u>                            |  |
| Bob Shannon<br>(Chair)<br><b>Present</b>                      | QRS, LLC Grand Marais, MN                                | Other                | 218-387-1100        | BobShannon@boreal.org                   |  |
| Tom Semkow  | Wadsworth Center, NY State                               |                      |                     |   |  |
| (Vice Chair) Present  | DOH<br>Albany, NY  | АВ                   | 518-474-6071        | tms15@health.state.ny.us                |  |
| Sreenivas (Vas)<br>Komanduri                                  | State of NJ Department of<br>Environmental Protection    | AB                   | 609-984-0855        | Sreenivas.Komanduri@dep.<br>state.nj.us |  |
| Present   | Trenton, NJ  |                      |                     |   |  |
| Marty Johnson   | US Army Aviation and Missile<br>Command Nuclear Counting | Lab                  | 865-712-0275        | Mjohnson@tSC-tn.com                     |  |
| Absent  | Redstone Arsenal, AL                                     |                      |                     |   |  |
| Dave Fauth  | Consultant   | Other                | 803-649-5268        | dj1fauth@bellsouth.net                  |  |
| Present – First<br>Part of Meeting                            | Aiken, SC  | Other                | 803-049-3208        | djiradin@bellsodin.net                  |  |
| Carolyn Wong  | Lawrence Livermore National Laboratory                   | Lab                  | 925-422-0398        | wong65@llnl.gov                         |  |
| Present   | Livermore, CA  |                      |                     |   |  |
| Keith McCroan   | US EPA ORIA NAREL,                                       | Lab                  | 334-270-3418        | mccroan.keith@epa.gov                   |  |
| Present Todd Hardt  | Montgomery AL Pro2Serve, Inc.                            |                      |                     |   |  |
| Absent  | Oak Ridge, TN  | Other                | 865-241-6780        | HardtTL@oro.doe.gov                     |  |
| Nile Ludtke   | Dade-Moeller and Associates                              | Other                | 865-481-6050        | nile.luedtke@moellerinc.co              |  |
| Present Larry Penfold   | Oak Ridge, TN Test America Laboratories,                 |                      |                     | <u></u>                                 |  |
| Present   | Inc; Arvada, CO  | Lab                  | 303-736-0119        | larry.penfold@testamericai<br>nc.com    |  |
| Richard Sheibley  Absent                                      | Sheibley Consulting, LLC                                 | Other<br>(Former AB) | 651-485-1875        | RHSHEIB111@yahoo.com                    |  |
| Ilona Taunton<br>(Program<br>Administrator)<br><b>Present</b> | The NELAC Institute                                      | n/a                  | 828-712-9242        | Ilona.taunton@nelac-<br>institute.org   |  |

# **Attachment B**

# **Action Items - REC**

|    |   |  | Target     | Actual  |
|----|---|--|------------|---|
|    | Action Item   | Who                                    | Completion | Completion                                      |
| 36 | Prepare summary of comments on standard through Section 1.7.  | Tom                                    | 2/25/14    | Still waiting for input from committee members. |
| 41 | Section 1.7.2.3: comment by email before the next meeting   | All                                    | 4/22/14    | Complete  |
| 42 | Update language in 1.7.2.   | Carolyn, Vas,<br>Bob and Marty         | 4/22/14    | Complete  |
| 43 | Work on language in 1.5.4.  | Keith                                  | 4/22/14    | Complete  |
| 46 | Continue to work on batch language on a special conference call. Results to be discussed at the next meeting. | Terry, Bob, and others who contact Bob | 5/6/14     | Complete  |
| 47 | Send April 23 <sup>rd</sup> meeting minutes out for an email vote.  | Bob                                    | 5/7/14     |   |
|    |   |  |            |   |
|    |   |  |            |   |

# Attachment C – Back Burner / Reminders

|   | Item  | Meeting<br>Reference | Comments |
|---|---|----------------------|----------|
| 1 | Update charter in October 2014  | 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              |          |
|   |   |                      |          |
|   |   |                      |          |
|   |   |                      |          |

#### Attachment D - Batch Discussion Documents

#### From Bob:

- 1) Where sample preparation is performed that involves physical or chemical sample processing (e.g., destructive analysis, grinding, sieving, evaporation, chemical separation), the laboratory shall prepare a preparation batch containing quality control samples and evaluate results as described in sections 1.7.2, and 1.7.3 and there is no need to initiate an analytical batch.
- 2) Where samples are measured without prior physical or chemical sample processing (e.g., non-destructive analysis of air filters or swipes gamma spectrometry or alpha beta proportional counting), an analytical batch may be initiated in lieu of a preparation batch with the following requirements:
  - i) Up to twenty (20) environmental samples analyzed using an analytical configuration common to all samples (i.e., samples processed using an analytical configuration common to all associated samples, as defined in the method validation,) may be combined into a single analytical batch. Batch QC samples shall be assigned to the batch that are applicable for measurement using the analytical method. The samples and associated QC parameters shall be analyzed using a set of common analytical parameters (i.e., same quality systems matrix, counting geometry, analytes) and may be counted on any detection system calibrated as described in section 1.7.1
  - ii) Samples may be added to the analytical batch until 20 samples have been counted or the maximum time limit for the analytical batch is reached. The total time of the analytical batch processing (analytical batch period) is limited to the time required to measure all samples in the batch using detection systems available for counting samples. This time shall not extend beyond ????14 days????.
  - iii) At minimum, one (1) positive control (calibration verification count), one (1) negative control (batch contamination check), and one (1) matrix duplicate (second count of one sample) shall be counted in each analytical batch. Systematic or preferential selection of detectors for analysis of QC samples shall be avoided.

## Section 1.7.1 language:

- 1.7.1.d.3. An individual test source may be uninterruptedly measured after the routine interval between performance checks expires as long as the respective quality control check is promptly performed once the test source measurement has completed and prior to initiating the next sample test source count.
- 1.7.1.f.4. An individual test source may be uninterruptedly measured after the routine interval between background checks has expired as long as the respective short term background check is promptly performed once the test source measurement has completed and prior to initiating the next sample test source count.

## From Tom:

#### Bob's version with Tom's corrections and comments

1) Where sample preparation is performed that involves physical or chemical processing of the sample (e.g., destructive analysis including grinding, sieving, evaporation, chemical separation), the laboratory

shall initiate a preparation batch and evaluate quality control sample results as described in Sections 1.7.2 and 1.7.3.

- 2) Where samples are measured without prior physical or chemical sample processing (e.g., non-destructive gamma spectrometry or alpha/beta counting of air filters or swipes on gas proportional detectors), an analytical batch may (I replaced shall with may, because analytical batch is an option for labs who elect to choose it.) be initiated in lieu of the preparation batch. The analytical batch, when initiated, shall have the following requirements:
  - i) Up to twenty (20) environmental samples may be combined into an analytical batch.
    - a. All samples and QC samples in the analytical batch shall (I think we cannot have the exact same sample and QC in an analytical batch regardless whether calibration is experimental or computational. The way it was written does not need analytical batch, would over-QC non-destructive analyses, and it would put many labs to a halt.) share the set or range of characteristics and analytical configurations have characteristics and analytical configurations in the ranges similar (I used this language in order to allow some room in putting slightly different samples together in the analytical batch.) to those used for calibration of the method (e.g., analytes, geometry, calibration parameters, and correction parameters).
    - b. The samples and associated QC samples may be counted on any detection system calibrated as described in Section 1.7.1. The laboratory shall have procedures to ensure that specific detectors are not systematically or preferentially used for the analysis of QC samples.
    - c. Samples may be added to the analytical batch until 20 samples have been counted or until the time limit for the analytical batch is reached. The maximum time for processing an analytical batch (analytical batch period) shall not extend beyond 14 days from the start of the first sample count.
  - ii) At minimum, one (1) LCS (I do not think we need new defiitions.), one (1) MB, and one (1) MD shall be assigned to each analytical batch (see Sections 1.7.2 and 1.7.3).
    - a. The LCS shall consist of a prepared standard or a calibration standard. The laboratory may prepare the LCS a single time and reuse it with the subsequent batches of samples.
    - b. The MD shall consist of a second measurement of one sample. The second measurement shall be performed on another detector, if available.
  - iii) The sample results associated with the analytical batch shall be reported after obtaining acceptable analytical batch QC results.

## Bob's version with Carolyn's comments in red

- 1) Where sample preparation is performed that involves physical or chemical processing of the sample (e.g., destructive analysis including grinding, sieving, evaporation, chemical separation), the laboratory shall initiate a preparation batch and evaluate quality control sample results as described in sections 1.7.2, and 1.7.3. An analytical batch is not required.
- 2) Where samples are measured without prior physical or chemical sample processing (e.g., non-destructive analysis of air filters or swipes gamma spectrometry or alpha beta proportional counting), an analytical batch shall be initiated in lieu of the preparation batch with the following requirements:
  - iv) Up to twenty (20) environmental samples may be combined into an analytical batch (this will not work for production laboratories handling hundreds of samples a day).

- a. All samples and QC samples in the analytical batch shall share the set or range of characteristics and analytical configurations used for validation of the method in Section 1.5 (e.g., analytes, geometry, calibrations for energy-resolution, energy-efficiency, density-self-absorption, instrument settings, coincidence summing). (I think we say this for each of the QC types, e.g. "The laboratory shall prepare the method blank using the dame quality system matrix as samples in the batch..." and "The size of the aliquot used for calculation of the method blank result shall be similar to that of routine samples for analyses..."
- b. The samples and associated QC samples may be counted on any detection system calibrated as described in section 1.7.1. The laboratory shall have procedures to ensure that specific detectors are not systematically or preferentially used for the analysis of QC samples. (See language for No QC Preference.)
- c. Samples may be added to the analytical batch until 20 samples have been counted or until the time limit for the analytical batch is reached. The maximum time for processing an analytical batch (analytical batch period) shall not extend beyond 14 days from the start of the first sample count.
  - v) At minimum, one (1) positive control (analytical batch calibration verification (I believe this is a new term, I'm not sure it is necessary)), one (1) negative control (analytical batch blank), and one (1) analytical batch duplicate shall be assigned to each analytical batch (my lab would have problems with this. It is simply impractical to pull samples from a batch and count them on a different instrument. We do recount positive samples when requested).
- a. Analytical calibration verification (ACV) (If we include this it needs to address other types of counters as well as gamma spectroscopy. For alpha/beta counters are instrument performance checks are done with the calibration standards (single-point calibrations). Would we require the laboratory to prepare a source separate from the sources used for the attenuation curve?):
  - i. The laboratory may prepare an ACV, or use a calibration standard for the ACV as long as the calibration standard is from the different source that that used for calibration of the measurement system.
  - ii. The laboratory may prepare the ACV a single time and reuse the standard with subsequent batches of samples.
  - iii. The activity of the ACV shall be 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%.
  - iv. The duration of the ACV count may be shorter than that of associated samples.
- b. Analytical batch blank (AB):
  - The AB is a blank test source set up together with samples at the time of initiation of the analytical batch
  - ii. The AB shall not contain detectable levels of analyte or interfering analyte.
  - iii. The duration of the AB count shall as long or longer than the longest sample count in the associated batch. (I believe we state this already)
- c. Analytical duplicate (AD) (See my note above regarding counting samples on multiple detectors.)
  - The AD consists of a second count of one sample. If more than one detection system is used for the analysis, the AD shall be counted on a different detector that the original sample result.
  - ii. The duration of the AD count shall be the same as the associated sample.
  - vi) Acceptable analytical batch QC results shall be obtained prior to reporting any sample results associated with the analytical batch. Once analytical batch quality control results indicate that the detection system is in control (This is where I'm confused, the instrument performance checks demonstrate that the detection system is in control,

not the analytical batch quality controls,), and all other method requirements have been met, associated sample results may be reported. (This should be added to section 1.7.3.)

- a. QC sample results shall be processed and data reduced using the same analytical parameters and data reduction routines used for the associated samples.
- b. ACV results shall be evaluated using the criteria established for laboratory control samples in 1.7.3.2.
- c. ABB sample results shall be evaluated using the criteria established for blanks in 1.7.3.1.
- d. AD results shall be evaluated using the criteria for duplicates established in 1.7.3.3. b).

### **Original Tom's version**

- 1) The laboratory shall process samples in the same quality system according to a preparation batch (Volume I, Module 2, Section 3.1).
- 2) For samples requiring only mounting and measurements, and not requiring any physical or chemical sample processing (e.g., non-destructive counting or spectrometry), the preparation batch can be substituted with an analytical batch (Volume I, Module 2, Section 3.1) having the following requirements:
  - i) Up to twenty (20) [?] environmental samples from different quality systems (e.g., different sample matrix, different, counting geometry, or different detectors) shall be combined into a single analytical batch.
  - ii) The total time of the analytical batch processing (analytical batch period) is limited to three (3) [?] times the total time required to measure all samples in the batch.
  - iii) The samples can start at any time during the analytical batch period.
  - iv) At the minimum, one (1) LCS, one (1) MB, and one (1) MD shall be inserted randomly between the samples and measured once each on a randomly selected detector.