Summary of the NELAP Accreditation Council Meeting

December 1, 2014

The NELAP Accreditation Council (AC) met at 1:30 pm EDT on Monday, December 1, 2014, for another of its series of assessor conversations. Attendance was not taken, except to note that FL, KS, LA DEQ, MN, NY, OR, PA, UT and VA, plus OK, had representatives present, as well as Analytical Excellence, Dade Moeller, Sims & Associates, Richard Sheibley and Wade Consulting.

The AC's Chair, Aaren Alger of PA, introduced Stephanie Ostrowski of NY as the moderator of the discussion about Method Modifications. Stephanie had circulated a document of discussion points prior to the meeting; this is in Attachment 1.

Stephanie explained that NY seeks to document what constitutes a method modification and that NY finds that process to be complicated by the prep methods that are sometimes included and sometimes accredited separately, but sometimes not. Defining what actually constitutes a method and a modification is proving difficult.

The drinking water methods in 40 CFR 141, those from **Standard Methods**, ASTM methods and any methods published in NY regulations essentially cannot be modified. Non-potable water methods in 40 CFR 136 may be modified within explicit constraints. Alternate Test Protocols (ATPs) may be approved by the EPA region for potable and non-potable water, on a per-site/per-discharge basis, and occasionally in-house methods are developed for uncommon or unregulated analytes and submitted for accreditation. For ATPs and in-house methods, NY needs to establish procedures for identifying these in the lab's Field of Accreditation (FoA) as well as to identify them satisfactorily for recognition purposes in secondary accreditations.

However, solid and hazardous waste methods (SW-846) are designed to be guidance, rather than rigid protocols, so that labs are expected to customize methods from this source for their own purposes. Further complicating these performance-based methods is the Methods Innovation Rule (MIR) and the advice provided by the Office of Resource Conservation and Recovery's (ORCR's) Method Information Communication Exchange (MICE line). Communications from the MICE line are intended by ORCR to be helpful and are commonly accepted by labs but are not official Agency interpretations but rather advice from contract personnel, based on industry best practices. it is NY's position that MICE line advice has no regulatory standing and cannot be used to define QC requirements.

Defining what is an "allowable change" to a method clearly requires client agreement, in accordance with the TNI Standard, but the end user of the data – the regulatory agency – often remains uninformed about modifications that may have been made to the analytical method and accepted by the client. Additionally, the concept of amplification of a method (changing volumes but not analytical techniques) must be accommodated into the modification scheme, and should be documented in the approved and accredited SOP, rather than being found by an assessor, after the fact.

At this point in the discussion, the Program Administrator pointed out that there is a clear philosophical difference between the programs using "regulatory methods" and those using "performance based methods," and that performance-based methods of ORCR will not fit easily into the regulatory framework that is established by the drinking water program. Other regulators (pesticides, foods, and drugs) routinely enforce regulatory limits with performance based methods, but the EPA regulatory framework was set up to accommodate the drinking water program's strict method protocols as defined in regulation.

Matrix-specific method validations as well as client approval are clearly needed when modifications are made. Modifications to the prep methods must also be documented, and this is complicated by the different ways in which prep methods may be associated with the quantification part of the analysis.

One AB noted that it gets few requests for modification of prep methods. Several ABs noted that select ion monitoring (SIM) methods are generating many requests for method modifications, to accommodate new analytes. Different ABs require different levels of documentation before approving modifications, ranging from a full analytical data package for assessor review to confirmation of approval for the modification by the state regulatory agency and/or the permit writer. NY noted that it has authority to approve the method modifications, but seeks to bring in the end user as a necessary approval (in this case, NY Dept. of Environmental Conservation, DEC) in addition to the lab's client.

NY posed the question of how a deficiency is cited by other assessors, when a previously unapproved modification is identified during the on site assessment. One contract assessor replied that a variation from the method as documented in 40 CFR, if not specifically noted as such in the SOP, warrants a deficiency of "not following the method." One AB clarified that they do cite unapproved modifications as deficiencies, while noting that it accredits "prep techniques" (not prep methods) as prep for particular methods, but these are not listed in the lab's FoA.

Discussion revealed that virtually all ABs and assessors struggle to some extent with the SW-846 methods and how to adapt the scheme for strict protocol regulatory methods to accommodate the modifiable performance-based methods.

NY asked about modifications to methods from non-EPA sources, such as ASTM, NIOSH and **Standard Methods**. One AB stated they cite the modification as a deficiency, and leave it up to the lab to argue, while acknowledging that the lab could become accredited for the method yet have the regulatory agency refuse the data. This AB did clarify that automations of manual methods are allowed. Another AB noted that if a lab seeks accreditation for a modification, it must apply as if for a new method. For instance, in substituting reagents in a commercial kit, one or two might be acceptable but if there are three substitutions, it must become a new SOP. This AB also noted that they see very few NIOSH methods, and mostly as secondary accreditations only.

NY then asked how best to identify a genuine modification to a method, in the lab's scope of

accreditation, recognizing that it's important for other ABs to realize that it is a modified method when it comes to granting secondary accreditations. One AB responded that if the modification goes beyond what's allowed in the method as written, then the accreditation is issued for the specific method SOP. Another AB just indicates "(modified)" after the method, in the scope.

One contract assessor noted that when the lab's client is a consulting firm, it's up to the client to ensure that the method agreed upon meets the regulatory requirements (and not up to the lab.) Discussion indicated that regulators are "almost never" consulted about normal "RCRA stuff" (now the ORCR methods, the SW-846 series) but that Defense and other federal programs are more often consulted about whether the methods as employed (modified) are acceptable.

Stephanie acknowledged that NY must soon finalize its procedures for approving method modifications, and that it must meet the state's regulatory requirements without too far exceeding the programmatic requirements of other NELAP ABs. She thanked everyone for participating and sharing their ideas.

Attachment 1

What is a modification?
NYS Regulation 10NYCRR 55-2.1 'Approved Methods', 55-2.2 'Certificates of Approval'
Any alteration from the approved (published) method – is considered a modification in NYS

Potable Water:
NYS ELAP will not consider a method modification in potable water
40 CFR 141.27 – Alternate Test Procedures (ATP) – with EPA
According to state regulation 10NYCRR 55-2.5 – DOH can review and approved 'in-house' developed method (rare)

Non-Potable Water:
40CFR 136.6 – 'Method Modification and Analytical Requirements'

- iDOC (mid-level standard, 4 replicated, MDL study)
- must meet or exceed QC requirements: precision/accuracy, detection limit, spike recoveries
- in matrix to which modified method applied
- must notify permitting authority of intent to modify
- document modification in SOP
- if does not meet or exceed – is not 'equivalent' and must use approved method as published
- if 'equivalent' – must continue ongoing QC tests
  - sample matrix (analysis of matrix spike/ spike duplicate pair) on every 20 samples
  - ongoing precision and recovery (lab fortified blank or blank spike) with each batch of 20 or fewer samples
- if method user uncertain if modification is allowed – must notify regional ATP coordinator or permitting authority prior to use

(3) Restrictions:
May not modify for a method-defined analyte, or result in the measurement of a different form or species of analyte, or if changes alter the define chemistry

> *phenol method 420.1 or 420.4 defines phenolics as ferric iron oxidized compounds that react with 4-aminoantipyrine (4-AAP) at pH 10 after being distilled from acid solution. Because total phenolics represents a group of compounds that all react at different efficiencies with 4-AAP, changing test conditions likely would change the behavior of these different phenolic compounds*

May not modify sample collection, preservation, or hold time
(Concept may conflict with Low Volume Initiatives (e.g., 3510C) in NW)

(4) Allowable changes: (i – x):
Chromatographic columns, temperature programs, automated vs manual sample prep, changes to pH adjustments, buffer reagents order of reagents added (so long as does not generate interference)

40CFR 136.5 – 'Approval of Alternate Test Procedures for Limited Use'

Solid and Hazardous Waste:
Methods Innovation Rule (MIR) (70 FR 34547, June 14, 2005) allows for the use of any appropriate SW 846 method for RCRA applications. BUT - the SW-846 methods are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application.

Use of MICE: http://www.epa.gov/osw/hazard/testmethods/mice.htm
Methods Information Communication Exchange (MICE) Service: "Provides timely answers to method-related questions and takes comments on technical issues regarding the EPA Office of Resource Conservation and Recovery (ORCR) methods manual known as *Test Methods for Evaluating Solid Waste: Physical/Chemical*

Methods (SW-846), Acts as an effective means to directly educate the public regarding inherent SW-846 method flexibility and to clarify whether a method is required by a RCRA regulation."

Not EPA, not a regulatory authority, always advised to check with permitting authority prior to use

No standardized process for validation and/or ongoing QC:

- Some methods have limited guidance on modification QC (eg. Method 3580A)

- Some methods do:

Example: Method 3546 (Microwave Extraction):
Sec. 1.5 Method validate using the solvents listed. Other solvents may be used.
Sec. 7.4 must demonstrate adequate performance for analytes of interest at levels of interest (regulatory limits), at a minimum with an initial DOC **using a clean matrix** – refer to Method 3500, Sec. 8.2

Method 3500, Sec. 8.2 : Demonstration of Proficiency:
Proficiency with each prep and determinative method combination – in a clean reference matrix, minimum of 4 replicates

Section 8.4: procedure in place for documenting and charting effect of matrix on method performance

- Neither account for matrix interferences – real world sample matrices

What are allowable changes?

- **Those listed in 40CFR 136.6? –good enough for non-potable water then good enough for SW?**
- **Allowable 'Amplifications' – listed in NELAC (2003) 5.5.4.5.2 and 2009 TNI V1M4 1.5.1**
  LOQ, LOD, Precision & Bias, Selectivity
- **Client agreement: NELAC (2003) 5.5.4.4 – Non-Standard methods, and 2009 TNI V1M4 1.5.1?**

"*When it is necessary to use methods not covered by standard methods, these shall be subject to agreement with the client and shall include a clear specification of the client's requirements and the purpose of the environmental test. The method developed shall have been validated appropriately prior to use*."

Outstanding questions:

- Informal poll in summer 2014: *** 8 of 14 ABs certify for prep methods***
- What about other methods not outlined above: ASTM, NIOSH, Standard Methods?
- How does recognition work between state ABs? - flag certificates? require state-specific approvals?
- Does this conflict with regulatory expectations of labs performing 'approved methods' or formally seeking method validations?
- What happens when client is not the end-user of the data? (environmental consulting firm vs. regulatory agency)