

TNI Stationary Source Audit Sample Expert Committee (SSAS) Meeting Summary

October 15, 2018

1. Roll call and approval of minutes:

Chair, Tom Widera, called the TNI SSAS Executive Committee meeting to order by teleconference on October 15, 2018, at 2:00 pm Eastern. Attendance is recorded in Attachment A – there were 6 committee members present. Associate Members present: Matt Sica (ANAB), Mike May (Chester LabNet), Khoi Nguyen (EPA Region 9), and Eugene Chen (EPA Region 9).

The minutes will be reviewed by email. Ilona needs notes from Tom to put together the August meeting minutes in New Orleans.

2. Method 8 Audit Samples

Tom sent an email out to the labs. He is trying to get input on the methods they are using. He got 6 or 7 responses. Some of the responses were to dilute in 80% IPA as this is how the stack samples come in. Some of the responses were to dilute in DI water if running by IC and 80% IPA if running by titration. Others didn't have an opinion.

There shouldn't be an issue doing the matrix matching. No waste issues. There should also be no problems with columns, but this depends on column being used. You should be able to dilute and not have a column problem.

Tom plans to have his company matrix match and will let Sigma know what is happening too. He is hoping this will improve the failure rate. This can't be required.

3. SOP 6-100 – SSAS Table Management

The group reviewed DRAFT SOP 6-100 that Sheri has been working on. See track changes in Attachment D for work reviewed during today's meeting.

Ned Schappley will be contacted about PTs and lower concentration limits. Just to be sure there isn't something the committee is missing.

Tom will review the definitions to be sure they are the same as the TNI Glossary and will send new definitions to Ken Jackson and Bob Wyeth.

4. New Business.

- None.

5. Action Items

The action items can be found in Attachment B.

6. Next Meeting

The next meeting will November 19 at 2pm Eastern by teleconference. *(Addition: The November meeting was canceled. The next meeting was December 17, 2018.)*

Action Items are included in Attachment B and Attachment C includes a listing of reminders.

Tom adjourned the meeting at 3:30pm Eastern.

Attachment A

Participants

TNI

Proficiency Testing Program Executive Committee

Members	Rep	Affiliation	Contact Information
Tom Widera (2020) CHAIR Present	Other	ERA (Provider)	twidera@eraqc.com
Ilona Taunton, Program Administrator Present			ilona.taunton@nelac-institute.org
Ed MacKinnon (2019*) Present	Other	TRC Env Corp (Stationary Source Tester)	emackinnon@trcsolutions.com
Gregg O'Neal (2020*) Absent	AB	NC DAQ	gregg.oneal@ncmail.net
Katie Gattis (2021) Absent	Lab	Element One Inc.	katie.strickland@e1lab.com
Michael Klein (2020*) Present	AB	NJ DEP	michael.klein@dep.nj.gov
Mike Hayes (2019*) Absent	Other	Linde (Provider)	mikeh@spectragases.com
Michael Schapira (2021*) Present	Lab	Enthalpy Analytical LLC	Mike.schapira@enthalpy.com
Sheri Heldstab (2022*) Present	Lab	Chester LabNet	sheldstab@chesterlab.net

Attachment B

Action Items – TNI PT Executive Committee

	Action Item	Who	Date Added	Expected Completion	Completion
2	Find out which group in EPA is helping the Microbiology FoPT Subcommittee crunch numbers for limits.	Ilona	2/12/18	3/19/18	Need to hear back from Jennifer Best.
6	Contact Ken about what notifications are needed to continue Standard development.	Ilona	4/23/18	5/21/18	
7	Send Sheri and Tom copies of TNI SOP on SOPs and PTPEC's SOP on Table Management.	Ilona	4/23/18	5/1/18	
8	Send Tom mailing lists with relevant stakeholders.	All	4/23/18	5/8/18	
9	Prepare general summary of what the committee plans to change in the current Standard and why. First DRAFT.	Tom	4/23/18	5/21/18	
10	Send ideas on Storage Condition issue to Tom so he can summarize them for an agenda item in July.	All	6/18/18	7/15/18	
11	Update Notice of Intent language and send to committee for last comments and then to Ken Jackson	Tom	7/16/18	8/15/18	
12	Discuss matrix matching with Sigma.	Tom	10/15/18	11/15/18	
13	Contact Ned Shappley about PT concentrations to complete SOP 6-100.	Tom	10/15/18	11/15/18	

	Action Item	Who	Date Added	Expected Completion	Completion
14	Contact Ken Jackson and Bob Wyeth about glossary definitions to make sure there are no conflicts with SOPs being worked on.	Tom	10/15/18	11/15/18	
15	Provide Ilona with notes from New Orleans meeting so August minutes can be completed.	Tom	10/15/18	TBD	

pilot study. At least five laboratories must be willing to absorb the costs of analyzing the Audit samples for the pilot study, to generate the data by which the acceptance limits of the pilot study concentrations shall be determined.

5.2 Vested Party Approval

The SSAS Chair must obtain, in writing (electronic or hardcopy) approval to participate in the pilot study from the Provider and a minimum of five laboratories. Approval must include a statement that the vested party understands that the financial burden of participation in the study shall be borne solely by the vested party. Approval to participate must be obtained from each participating laboratory for each shipping batch of pilot study samples. A non-disclosure agreement may be utilized by either the Provider or the laboratory.

5.3 Production of Pilot Study Audit samples.

The PTP interested in creating an expanded concentration range shall manufacture, at their cost, one or more PE samples in the pilot study concentration range, using the same protocols used in general manufacture of PE samples to include homogeneity and stability testing. The PTP shall, at their cost, ship a minimum of four and a maximum of 10 samples in one batch to the laboratories participating in the study. No more than two batches per year may be shipped to a given laboratory.

5.4 Analyses of pilot study range PE samples.

The laboratories who have agreed to participate in the study shall analyze the pilot study range PE samples in a manner consistent with their protocols for analyzing PE samples. The samples must be prepared and analyzed using the same preparatory and analytical techniques as used when analyzing PE samples for submission with field samples.

5.5 Reporting the Results

The PTP shall have a means of reporting the results that is separate from routine PE samples. The PTP shall have this reporting system functional prior to the shipment of samples to the laboratory.

The laboratory shall understand how to use the reporting system and agree to use only that reporting system designated by the PTP for the pilot study concentration range study. The laboratory shall submit the results within 60 days of receipt of the samples.

5.6 Statistical Analysis

The PTP shall aggregate the data in a manner such that statistical analyses may be easily performed on the data.

5.6.1 Applicability

5.6.1.1 The underlying assumptions for these calculations are that laboratory measurements for analyte concentrations follow a normal (Gaussian) distribution.

5.6.1.2 The linear regression model was used by US EPA for determining acceptance limits prior to externalizing the PT program and is used by many environmental laboratories in calibrating test equipment. If higher-order regressions, segmented regressions, or other models are used, then acceptance criteria based on correlation coefficients and statistical outlier removals based on standard errors of the estimate may not be applicable.

- Tom Widera 10/15/2018 1:13 PM
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5.6.1.3 For those fields of proficiency testing that have acceptance limits specified in US EPA regulations, the acceptance limits used by the TNI program shall not be wider than the US EPA limits. [ed note: are any of our acceptance limits set by EPA?]

5.6.2 A Remove obviously erroneous data as follows for each data set of aggregated results for a particular analyte:

5.6.2.1 1 Display original data from lowest Assigned Value to highest Assigned Value, listing AV, PM, MR, RSD and N.

5.6.2.2 2 Review the data set for anomalies and PT Provider reporting errors. Remove all sets with $N < 10$ [ed note: this is not in agreement with the "collect 20 data points" mentioned in Section 4.9 of Rev 1.0 SSAS Table Management SOP]. Review apparent outliers in MR and RSD in the ordered list. Flag, at a minimum, sets where the $MR < 10\%$ or the $MR > 200\%$. Also, flag data sets where the $RSD > 50\%$. If the majority of data sets contain $RSD > 50\%$, there is no need to flag these data.

NOTE: IN ALL CASES where data is removed from the aggregate data set, the final number of remaining pilot study data points (N) used for determining the acceptance limits, after outlier removals, must be ≥ 10 .

5.6.2.2.1 ii Record the reasons for all data set removal;

5.6.2.2.2 iii Retain all data sets that can't be justified for removal, and that do not exceed the above limits.

5.6.3 Determine linearity of aggregate data set:

5.6.3.1 4) Display graphically the AV (horizontal, x axis) vs. PM (vertical, y axis).

5.6.3.2 7) Display graphically the AV (horizontal, x axis) and SD (vertical, y axis).

5.6.3.3 5/8) Perform linear regression analysis to determine slope (a, c), Y-intercept (b, d), the square of the correlation coefficient R^2 , (Standard Deviation), and SER for each graph.

5.6.3.4 6/9) Look for nonlinearity in the plot, since that is an indication of a need for segmented regression or a higher order regression model.

5.6.4 Evaluate initial linearity of aggregate data set:

5.6.4.1 10/16) If R^2 (Mean) is ≥ 0.90 , R^2 (Standard Deviation) is ≥ 0.75 , and $N \geq 10$ data points, then the regression is acceptable.

5.6.4.1.1 12) Calculate the PT acceptance limits over the concentration ranges using a, b, c, d. [ed note: this is exactly as written in 4-101. There is no actual formula(e) for how to calculate the acceptance limits, just direction to do so.] Display graphically the derived acceptance limits (y axis) with pilot study sample concentrations (x axis).

5.6.4.1.2 13) Check visually for convergence (points at the lowest ends of the concentration range where the acceptance limits cross). If this occurs, examine the data again for a need for segmented regression or for influential data-set points (high or low) that could influence the

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regression in a way that causes the convergence. Display graphically AV vs. PM, AV vs. SD, and AV vs. RSD, along with the regression lines to help determine such influential points.

NOTE: Influential points may lie at the opposite end of the testing range from where the convergence occurs, and are best identified with analysis of the residuals (differences between observed and expected points, based on the proposed linear regression equation). Points are best checked for influence by conducting the analysis with and without the point (or points) in question and determining the effect. There are several accepted statistical procedures for this, but for the purposes of this procedure the initial data censoring rejects data points outside +/- 2.0 SER for both the PM vs. AV regression and the SD vs. AV regression.

- 5.6.4.1.3 **14)** Check for single points far from the body of points, to see if the points belong in the same relationship as the others. For example, if the next highest concentration point is less than 70% of the highest concentration point along the x axis, the highest concentration point exerts a disproportionate influence on the regression results. In such a case, a reduction in the concentration range by eliminating the highest concentration point may be needed.
- 5.6.4.1.4 **16)** If no PT data points are removed from the data set, proceed to Section 5.6.5.
- 5.6.4.2 **11)** If there is poor correlation on regressions between the PM's and the AV's, or between the SD's and the AV's, then either:
 - 5.6.4.2.1 Repeat Section 5.6.4.1 using participant means and calculated limits for SD; or
 - 5.6.4.2.2 Remove further suspect data as follows:
 - 5.6.4.2.2.1 **17)** Remove all values farther from the line than + 2.0 SER for the regression of PM versus AV.
 - 5.6.4.2.2.2 **18)** Rerun the linear regression of PM versus AV, obtaining new values for a, b, and R² (mean) and SER.
 - 5.6.4.2.2.3 **19)** Remove all values farther from the line than + 2.0 SER for the regression of SD versus AV.
 - 5.6.4.2.2.4 **20)** Rerun the linear regression of SD versus AV, obtaining new values for c, d, R² (standard deviation), and SER.
 - 5.6.4.2.3 **15)** If any influential points are removed for being distantly separated, or creating "convergence," then eliminate those points by proceeding with the analysis beginning as in Section 5.6.4.2.2. Document the reason(s) for any data-point removals (e.g., +/- 2 SER to eliminate data convergence at the low end, + 1 SER to achieve acceptance criteria for the square of the correlation coefficient).

5.6.5 Verify Acceptance Limits and Regression: