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

March 16, 2020

1. Roll call and approval of minutes:

   Chair, Sheri Heldstab, called the TNI SSAS Executive Committee meeting to order by teleconference on March 16, 2020, at 2pm Eastern. Attendance is recorded in Attachment A – there were 4 committee members present. Guest(s): Stan Tong.

2. Announcements

   Sheri asked people to PLEASE look over the SES newsletter blurb and the FAQ that goes with the Poster, even though the SES conference has been 'postponed' until next year. Both documents are very similar, and Sheri is concerned that we could contradict ourselves if the documents aren't proofed/corrected/changed at the same time. The due date for comments is 3/25/20.

   Sheri thanked Michael Klein for his input on these documents.

3. SOP 6-100

   The Committee continued discussion of SOP 6-100 and worked on wording (see track changes in 6-100 in Attachment D).

   The committee decided after continued discussion that the ANOVA test recommended by Carl Kircher was not the right tool for Pilot Study data given our (most likely) small data set due to needing labs to volunteer to run samples for free, and the need to find labs who know the methods.

4. New Business.

   None.

5. Action Items

   The action items can be found in Attachment B.

6. Next Meeting

The next meeting will be April 6, 2020 at 2:30pm Eastern. Ilona will send out a WebEx invitation the day of the meeting.

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

Sheri adjourned the meeting at 3:30pm Eastern. (Motion: Mike S. Second: Gregg. Unanimously approved.)

Attachment A

Participants
TNI
Stationary Source Audit Sample Expert Committee

Members	Rep	Affiliation	Contact Information
Sheri Heldstab (2022*) CHAIR **Present**	Lab	Chester LabNet	sheldstab@chesterlab.net
Tom Widera (2023) VICE-CHAIR **Present**	Other	ERA (Provider)	twidera@eraqc.com
Ilona Taunton, Program Administrator **Present/Recording**		TNI	Ilona.taunton@nelac-institute.org
Ed MacKinnon (2022) **Absent**	Other	TRC Env Corp (Stationary Source Tester)	emackinnon@trcsolutions.com
Gregg O'Neal (2020* **Present**	AB	NC DAQ	gregg.oneal@ncdenr.gov
Katie Gattis (2023) **Absent**	Lab	Element One Inc.	katie.gattis@e1lab.com
Michael Klein (2020*) **Absent**	AB	NJ DEP	michael.klein@dep.nj.gov
Michael Schapira (2021*) **Present**	Lab	Enthalpy Analytical LLC	Mike.schapira@enthalpy.com

Attachment B

Action Items – Stationary Source Audit Sample Expert 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. [1/21/20: Eric Smith (PTEC) said that Chemistry FoPT subcommittee working on calculations] [2-18-20: Shawn did not have formulae, but agreed to stay in touch with me to ensure consistency between 6-100 & 4-101]
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	In progress. [1/21/20: On hold until SOP 6-100 & 6-101 completed]
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	How to word storage conditions. Leave open. [1/21/20: On hold until SOP 6-100 & 6-101 completed]
18	Update SOP 6-100 based on review during meeting.	Tom	1/22/19	2/24/19	In Progress
37	Put current Charter up on the TNI website.	Tom/Bob Wyeth	2/18/20	3/2/20	

	Action Item	Who	Date Added	Expected Completion	Completion
38	Comment on Sheri's SES Poster for presentation.	All	2/18/20	3/2/20	
39	Consider contacting PT Providers about providing Audit Samples after more procedures and modules are complete.	TBD	2/18/20	TBD	
40	Send an example action table to Sheri from another committee.	Ilona	3/2/20	3/16/20	
41	Meet with Carl Kircher and Shawn Kassner to discuss statistics in SOP 6-100.	Sheri	3/2/20	3/16/20	
42	Comment on SES newsletter article and FAQ.	All	3/16/20	3/25/20	

Attachment C

Backburner / Reminders
Stationary Source Audit Sample Expert Committee

	Item	Meeting Reference	Comments

Attachment D - SOP 6-100

SOP TITLE:	**Conducting Pilot Studies for New Concentration Ranges and Acceptance Limits for Source Sampling Audit Samples.**
SOP NO.:	**6-100**
REVISION NO:	**0**
PROGRAM	**SSAS**

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SOP Approval Dates

	Initial Approval	Last Revision Date	Last Review Date
Committee: SSAS Expert			
Program: SSAS			
Policy Committee			
TNI Board of Directors Endorsement			
SOP Effective Date			

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1.0 Purpose and Applicability

1.1 This SOP provides the procedure for expanding the concentration range(s) for existing SSAS Table analytes. The expansion may include either increasing or decreasing the concentrations for SSAS Audit samples. This SOP is also applicable to new method(s) and/or analyte(s) requested to be added to the SSAS Table.

1.2 This SOP is applicable to the determination of new acceptance criteria and/or concentration ranges for method/analyte combinations in the SSAS Table. This SOP is also applicable to the determination of acceptance limits for new method(s) and/or analyte(s) requested to be added to the SSAS Table.

1.2.1 This SOP is applicable to the determination of acceptance criteria determined assuming 100% recovery (relative to the made-to Assigned Value).

1.2.2 This SOP is applicable to the determination of acceptance criteria based on participant results used to calculate a consensus target value (robust mean).

1.2.3 For other methods of determination, refer to TNI SOP 4-101, Revision 3.1, Section B.

2.0 Summary

One or more Pilot Study Audit sample(s) are manufactured by an Accredited Audit Sample Provider, at their expense. A minimum of five (5) volunteer laboratories shall be provided with at least one (1) Audit sample from each manufacturing lot within the Pilot Study. The laboratories, at their expense, shall analyze the Pilot Study samples and report their findings to the Audit Sample Provider using a reporting format determined in advance by the Audit Sample Provider, and not utilizing the reporting system for reporting Audit samples for compliance or regulatory purposes. The results shall be aggregated into one data set per concentration per method per analyte. The theoretical acceptance limits shall be determined.

The SSAS Expert Committee shall be presented with the results of the statistical analyses following the protocol in SOP 6-101, Revision 2.0. After a review of the statistical analyses by the SSAS Expert Committee, the new concentrations and/or acceptance limits shall be voted upon by the SSAS Expert Committee. If the vote passes, the Chair shall forward the modified SSAS Table to the PTPEC for review.

The procedure for changing the table is documented in SSAS SOP 6-101.

3.0 Related Documents

3.1 TNI SOP 6-101, SSAS Table Management. Revision 2.0.
3.2 TNI SOP 4-101, Calculation of Acceptance Limits for Chemical, Radiochemical, and Microbiological Components of Proficiency Tests. Revision 3.1.
3.3 TNI SOP 1-100 Format Guidelines for Standard Operating Procedures (SOPS) of The NELAC Institute (TNI), Revision 1.2.

4.0 Definitions

a	Slope of the linear regression line for $\bar{X}_{PR}$ vs. AV.
Acceptance Limit	The range of values that constitute acceptable performance for a laboratory participating in an audit sample or PT study.
ASRL	Audit Sample Reporting Limit. A statistically derived value that represents the lowest acceptable concentration for an analyte in an Audit sample.

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AV	Assigned Value. Value attributed to a particular property of a proficiency test or audit sample.
b	Y-intercept of the linear regression line for $\bar{X}_{PR}$ vs. AV.
c	Slope of the linear regression line for AV (or $\bar{X}_{PR}$) vs. SD.
Central Database	Repository for data related to audit performance and any field sample concentration measurements that are being evaluated in accordance with the TNI SSAS Program.
d	Y-intercept of the linear regression line for AV (or $\bar{X}_{PR}$) vs. SD.
DL	Detection Limit.
Gaussian Distribution	Also called normal distribution. A statistical function where the probability that a result falls within one standard deviation of the mean value is 0.683.
LoQ	Limit of Quantitation. The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence.
Manufacturing Lot	A definite amount of material produced during a single manufacturing cycle, and intended to have uniform character and quality.
n	Number of participant results used to determine $\bar{X}_{PR}$ and SD.
Pilot Study	A round robin study whose sole purpose is to obtain data for the potential expansion of the concentration range and/or determination of acceptance limits for analytes contained on the SSAS Table or analytes and/or methods to be added to the SSAS table.
Pilot Study Audit Samples	Audit samples whose sole purpose is conducting the Pilot Study. These are not compliance samples and shall not be evaluated for compliance.
PTPEC	Proficiency Testing Program Executive Committee. The committee responsible for the administration and maintenance of the SSAS program.
Provider	Organization providing Audit samples for the Audit Sample Pilot Study.
Robust Mean	The average of all data points for a given concentration returned in a Pilot Study, prior to outlier removal.
Robust Standard Deviation	The standard deviation of all data points for a given concentration returned in a Pilot Study, prior to outlier removal.
RSD	Relative Standard Deviation, expressed as percent.
R	Correlation coefficient, or Pearson Product Moment of Correlation for the regression used.
R^2	"R-squared", the square of the correlation coefficient for the regression used.
SD	Standard Deviation of participant results (robust or after outlier removal).
SER	Standard Error of Regression (synonymous with Standard Error of the Estimate).

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Shall	Denotes activities, procedures, or elements from which no deviation is allowed and is synonymous with "must" as opposed to "may" or "should".
Should	Indicates that an associated element is recommended but not mandatory and is synonymous with "may".
SOP	Standard Operating Procedure. A written document that details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks.
SSAS	Stationary Source Audit Sample. May refer to the Program or the Expert Committee.
SSAS Table	Stationary Source Audit Sample Table. Table in which the methods, analytes, and acceptance limits for audit sample materials are defined.
Vested Party	An entity with financial burden in the Pilot Study (e.g., Provider or laboratory).
$\bar{X}_{PR}$	Mean of participant results, either robust or after outlier removal.
$\bar{X}_{Rec}$	Mean recovery of participant results, either robust or after outlier removal.

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5.0 Procedure for Expanding the Concentration Range and Acceptance Limits of SSAS Audit samples or creating a new audit sample method/analyte combination.

5.1 Determination of Need

The SSAS Expert Committee is responsible for determining, by consensus, if there is a need for a new concentration range for an existing Audit sample or a need for a new method and/or analyte to be added to the SSAS Table.

At least one Provider must be willing to absorb the costs associated with producing Audit samples for the 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.

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5.2 Vested Party Approval

The SSAS Expert Committee Chair must obtain, in writing (electronic or hardcopy), approval to participate in the Pilot Study from the Provider and a minimum of five laboratories, prior to the beginning of the study. 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 Provider(s) interested in participating in the Pilot Study shall manufacture, at their cost, one or more samples in the Pilot Study concentration range, using the same protocols used in general manufacture of Audit samples to include homogeneity and stability testing. The Provider shall, at their cost, ship a minimum of four and a maximum of 10 samples in one shipping batch to the laboratories participating in the study. Shipping batches must be composed of the same number of samples from the same

manufacturing lots, and must be shipped to all laboratories participating in the Pilot Study. No more than two batches per year for any given sample type (e.g. total fluoride in impinger solution) may be shipped to a given laboratory.

5.4 Analyses of Pilot Study Audit Samples.

The laboratories who have agreed to participate in the Pilot Study shall analyze the Pilot Study Audit samples in a manner consistent with their protocols for analyzing routine samples. The laboratory shall initially prepare the Pilot Study Audit sample at the dilution ratio specified by the Provider's instructions.

The diluted samples must be prepared and analyzed using the same preparatory and analytical techniques as used when analyzing field samples.

5.5 Reporting the Results

The Provider shall have a means of reporting the results that is separate from routine Audit samples. The Provider shall have this reporting system functional prior to the shipment of samples to the laboratory. This separate reporting system must be designed to prevent the possibility of pilot study data being inadvertently submitted to the Central Database.

The Provider reporting system must include the capability for the lab to report, at a minimum, the name of the laboratory, Pilot Study Sample Analyte(s) Results, Detection Limit (DL), Limit of Quantitation (LOQ), Reference Method and Analytical Technique (e.g. ICP, ICP-MS).

The laboratory shall understand how to use the reporting system and agree to use only that reporting system designated by the Provider 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 Provider shall aggregate the data in a manner such that statistical analyses may be easily performed on the data. Appendix A contains the calculations necessary to perform the required statistical analysis described below.

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 is the same as that used by the US EPA for determining acceptance limits prior to their externalizing of the Audit Sample 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 determined using correlation coefficients and statistical outlier removals based on standard errors of regression may not be applicable.

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

5.6.2.1 Display original data from lowest Assigned Value to highest Assigned Value, listing AV, $\bar{X}_{PR}$, $\bar{X}_{Reg}$, RSD and n for each set of returned results at each Assigned Value.

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<#>Using a computer program capable of performing a one-way F test, such as Microsoft Excel or other statistical program, calculate the F-value and the p-value. Based on degrees of freedom, look up the F Critical value on an F test table.↵
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<#>Note: MSExcel does not come with the "DataAnalysis" menu option pre-installed. Instructions for installing the "Analysis ToolPak" Add-In are easily found on the internet.↵
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<#>If the returned F value is >F-crit and the p-value is <0.001, the intralaboratory variance is not significantly different from the interlaboratory variance, and the entire robust data set may be used. Carl, is this correct/reasonable/warranted?↵
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<#>If the returned p-value is >0.01, consider whether this is the due to one laboratory's returned results being significantly different enough from the other laboratories' returned results to warrant removal of a single set of returned data. Carl, F-test challenge failed on spreadsheet.¶

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5.6.2.2 Review the data set for anomalies and Provider reporting errors.

5.6.2.3 Remove all sets with n <20.

5.6.2.4 Review apparent outliers in $\bar{X}_{Rec}$ and RSD in the ordered list. Flag, at a minimum, sets where the $\bar{X}_{Rec}$ <10% or the $\bar{X}_{Rec}$ >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.

5.6.2.5 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 ≥20, and no single laboratory's results may exceed 25% of the final number of remaining Pilot Study data points (n).

5.6.2.5.1 Record the reasons for all data sets removed;

5.6.2.5.2 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 robust data set:

5.6.3.1 Display graphically the AV (horizontal, x axis) vs. $\bar{X}_{PR}$ (vertical, y axis).

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

5.6.3.3 Perform linear regression analysis to determine slope (*a*, *c*), Y-intercept (*b*, *d*), the square of the correlation coefficient (R^2), and standard error of regression (SER) for each graph. For the AV vs. $\bar{X}_{PR}$ graph, the slope is *a*, and the Y-intercept is *b*. For the AV vs. SD graph, the slope is *c*, and the Y-intercept is *d*.

5.6.3.4 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 robust data set:

5.6.4.1 If R^2 ($\bar{X}_{PR}$) is ≥0.90, R^2 (SD) is ≥0.75, and n ≥20 data points, then the regression is acceptable.

5.6.4.1.1 Calculate the Audit sample acceptance limits over the concentration ranges using *a*, *b*, *c*, *d*. Display graphically the derived acceptance limits (y axis) versus Pilot Study sample concentrations (x axis).

5.6.4.1.2 Check visually for convergence (points at the lowest end 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 regression in a way that causes the convergence. Display graphically AV vs. $\bar{X}_{PR}$, 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

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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 × SER) for both the $\bar{X}_{PR}$ vs. AV regression and the SD vs. AV regression.

5.6.4.1.3 This section is not applicable when the Provider is attempting to lower the concentration range for an existing analyte/method combination.

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 If no Audit sample data points are removed from the data set, proceed to Section 5.6.6.

5.6.4.2 If there is poor correlation on regression between the $\bar{X}_{PR}$ and the AV, or between the SD and the AV, then either:

5.6.4.2.1 Repeat Section 5.6.3 using mean ($\bar{X}_{PR}$) and standard deviation (SD), then remove all values farther from the line than ±(2 × SER) for the regression of SD versus AV.; **or**

5.6.4.2.2 Remove further suspect data as follows:

5.6.4.2.2.1 Remove all values farther from the line than ±(2 × SER) for the regression of $\bar{X}_{PR}$ versus AV.

5.6.4.2.2.2 Remove all values farther from the line than ±(2 × SER) for the regression of SD versus AV.

5.6.4.2.2.3 Rerun the linear regression of $\bar{X}_{PR}$ versus AV, obtaining new values for *a*, *b*, R^2 (mean), and SER (mean).

5.6.4.2.2.4 Rerun the linear regression of SD versus AV, obtaining new values for *c*, *d*, R^2 (standard deviation), and SER (SD).

5.6.4.2.2.5 Remove all values farther from the line than +(1 × SER(SD)) for the regression of SD versus AV.

5.6.4.2.2.6 Rerun the linear regression of $\bar{X}_{PR}$ versus AV, obtaining new values for *a*, *b*, R^2 (mean), and SER (mean). Rerun the linear regression of SD versus AV, obtaining new values for *c*, *d*, R^2 (SD), and SER (SD).

5.6.4.2.3 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(SD)) to achieve acceptance

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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. ... [6]

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criteria for the square of the correlation coefficient).

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5.6.5 Verify Acceptance Limits and Regression:

5.6.5.1 Repeat Section 5.6.3 and 5.6.4 again at this point to consider the adequacy of the resulting regression equations and to confirm the suitability of the derived acceptance limits. If such considerations and confirmations are deemed satisfactory, then proceed to Section 5.6.7. Otherwise, if the results are unsatisfactory, then one or more of the following may need to be implemented:

5.6.5.1.1 Alternate statistical analyses, regression models, data distributions, and acceptance criteria need to be used.

5.6.5.1.2 The Audit sample acceptance limits for the SSAS Table should be recommended based on the robust mean and 2 robust standard deviations in each individual Pilot Study.

5.6.5.1.3 The Pilot Study concentration range or analyte/method combination should not be recommended for inclusion or continuation in the Audit Sample Program.

5.6.6 Test for Fixed Limits:

5.6.6.1 To test whether the AV can be used as a target value, examine whether *a* is between 0.95 and 1.05, and whether the absolute value of *b* is less than 5% of the lowest AV concentration being used (i.e., the lowest point in the concentration range).

5.6.6.2 To test for the use of fixed acceptance limits, examine whether the absolute value of *d* is less than 5% of the lowest AV being used for the equation.

5.6.6.3 If *a*, *b*, and *d* meet the above criteria, then consider recommending a fixed acceptance limit of (AV ± (2 × *c*)) for the SSAS Table. Fixed limits should be "rounded" to logical values (i.e., 12.6% could be rounded to 10%, 12%, or 15%, depending on the Expert Committee's judgment).

5.6.7 Verify that the proposed Audit sample acceptance limits are consistent with test method quality characteristics for accuracy that are routinely in use at environmental testing laboratories.

5.6.7.1 Calculate the Audit sample acceptance limits over the applicable concentration ranges using *a, b, c, d.* Display graphically the derived acceptance limits (y axis) along with submitted concentration ranges (x axis).

5.6.7.2 Compare the Audit sample acceptance limits for the SSAS Table with method Quality Control (QC) limits, if applicable. Audit sample acceptance limits narrower than laboratory QC limits should prompt re-evaluation of the proposed Audit sample acceptance limits. Check to see if the rejection of too many Pilot Study data points could have been responsible for this occurrence.

6.0 Documentation and Submittal for Consideration:

6.1 The Provider must document the manufacture and assignment of the assigned value of the Pilot Study concentration samples in a similar manner to the documentation maintained for routine Audit samples.

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6.2 The Provider must document the statistical analyses performed and the results of the analyses in a concise and unambiguous manner to be submitted to the SSAS Expert Committee for review. The results shall be anonymized such that all references to the laboratories have been removed.

The data that must be submitted from the Provider to the SSAS Expert Committee for each analyte in each Pilot Study manufacturing batch are as follows:

6.2.1 verified Assigned Value (AV),
6.2.2 total number of participant results submitted by the laboratories (n),
6.2.3 number of participant results (n) used to determine acceptance limits,
6.2.4 results prior to and, if used, after removal of outliers,
6.2.5 robust mean ($\bar{X}_{PR}$),
6.2.6 mean ($\bar{X}_{PR}$) used to determine acceptance limits,
6.2.7 robust Standard Deviation (SD),
6.2.8 standard deviation (SD) used to determine acceptance limits,
6.2.9 lower and upper acceptance limits,
6.2.10 graphical representation of data as described in Sections 5.6.3, 5.6.4, and 5.6.5,
6.2.11 the ASRL, calculated as (AV – (2 × SD)) and calculated as 0.1 × (lowest analyte concentration in the concentration range),
6.2.12 total number of participant laboratories,
6.2.13 number of participant laboratories whose data was used to determine acceptance limits,
6.2.14 the percentage of total number of participant results used to calculate the acceptance limits that would have failed the analyte based on the calculated acceptance limits, and,
6.2.15 range of the Detection Limits (DLs) and Limits of Quantitation (LoQs) for the participating laboratories as described in 6.2.12.

6.3 The laboratory must document all Pilot Study samples' receipt, log in, preparation, and analysis following their normal protocols for those activities. This documentation must be made available to the Provider upon request.

6.4 The Provider shall submit a SSAS Table Change Request Application (CRA) per TNI SOP 6-101 and attach all documentation from Section 6.2 to the CRA for consideration by the SSAS Expert Committee.

7.0 Review of Pilot Study concentration ranges and acceptance limits. The SSAS Expert Committee shall consider the following:

7.1 The number of data sets originally considered for this evaluation relative to the number of data sets that remained for the final determination of the acceptance limits. If over 33% of the available Pilot Study data are rejected as outliers, then the SSAS Expert Committee must consider seriously the implications of rejecting so many data points just to make correlation coefficients and acceptance limits achieve desired criteria. Reasons for all rejections of outliers shall be documented, in case reconsiderations are necessary.

7.2 The ASRL for the Pilot Study concentration ranges as determined by (AV – (2 × SD)). This value is the lower Audit sample acceptance limit when the Assigned Value is at the lowest analyte concentration in the concentration range. If the Audit sample acceptance limits are based on the $\bar{X}_{PR}$, the ASRL may be set based on one-tenth the lowest analyte concentration in the concentration range.

7.3 How this ASRL relates to Detection Limits (DLs) and Limits of Quantitation (LoQs) expected in environmental laboratories for various analytical technologies that are in routine use. Reconsideration of the Audit sample acceptance limits may be necessary if some less-sensitive common laboratory technologies cannot achieve the proposed ASRL. Some technologies that

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Deleted: Method …etection Limits (M…Ls) and Limits of Quantitation (LoQs) expected in environmental laboratories for various analytical technologies that are in routine use. With the proper sample preparation and analysis regimen, laboratories accredited for a particular FoA should be able to achieve a LoQ below the ASRL for the corresponding Table (which uses the same ... [23]

could pose difficulties in this regard are IC and titration (as in Method 6 and Method 8).

7.4 Acceptance limits relative to applicable concentration ranges: Examine the graph of the Audit sample acceptance limits. Consider the span of the acceptance limits between the lower limit and upper limit, relative to the analyte concentration range. As an example, to illustrate the problem, if the Audit sample acceptance limits are 50-150% of assigned value and the concentration range is narrow, e.g., 100-200 µg/L, then the resultant Audit sample provides no suitable challenge to laboratories. The laboratory conceivably could report 150 µg/L each Audit for the analyte and never fail it. Thus, for problematic analytes where the audit sample acceptance limits are wide, the concentration range should be looked at critically to verify that any outlier data did not also truncate the concentration range that could have been used for the concentration range.

One procedure to ensure suitable challenge to the laboratories is to take the logarithm of the ratio of the highest and the lowest concentrations in the concentration range, then divide that by the logarithm of the ratio of the upper to lower Audit sample acceptance limits. If the ratio is ≥2, then the Audit is suitable for achieving challenge to the participants. As an example, if the Audit sample acceptance limits are 66.6 – 133% and the concentration range is 20-200 µg/L, then the calculation works out as:

log (200/20) / log (133/66.6) = 1.00 / 0.301 = 3.3 (suitable challenge).

For the example in the first paragraph, the ratio is <1.0 (useless as an Audit).

7.5 As indicated with all the considerations in this section, the recommendations for Audit sample acceptance limits may not fulfill all the desired acceptance criteria. Compromises are made to achieve as many of the desired acceptance criteria as possible for the SSAS Table. The appropriate balance needs to be documented carefully, as to which criteria were achieved and which criteria could not be met, among the following:

7.5.1 At least 20 valid data points with at least 5 participant laboratories in the Pilot Study.
7.5.2 R^2 (Mean) is ≥ 0.90 after linear regression of $\bar{X}_{PR}$ versus AV.
7.5.3 R^2 (Standard Deviation) is ≥ 0.75 after linear regression of SD versus AV or SD versus $\bar{X}_{PR}$.
7.5.4 Audit sample acceptance limits are consistent with laboratory control limits in the reference method, if available, for test method accuracy.
7.5.5 ASRL is consistent with the requirements of Section 7.3.
7.5.6 Audit sample acceptance limits relative to the concentration range result in an Audit sample that provides suitable challenge to participants.

7.6 The above considerations shall, at a minimum, be documented on the "Checklist For Consideration of Pilot Study Data" found in Appendix B of this SOP.

8.0 References

8.1 M Thompson, SLR Ellison, and R Wood, "The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories," Pure Appl. Chem. Vol. 78, #1 (2006), pp.145-196.

8.2 ISO 13528, "Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparisons," International Organization for Standardization (2005).

8.3 ISO/IEC 17043: Conformity assessment - General requirements for proficiency testing (2010).

8.4 TNI SOP 4-101, Revision 1.2 "Calculation of Acceptance Limits for Chemical, Radiochemical, and Microbiological Components of Proficiency Tests".

9.0 SOP Approved Changes

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Commented [S13]: removal of duplication of "suitable"
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Revision No.	Effective Date	Description of Change
0	DRAFT	New Document.

Appendix A: Calculations

$$a \text{ or } c\ (\text{slope}) = \frac{(\sum y)(\sum x^2) - (\sum x)(\sum xy)}{n(\sum x^2) - (\sum x^2)}$$

$$b \text{ or } d\ (Y - \text{intercept}) = \frac{n(\sum xy) - (\sum x)(\sum y)}{n(\sum x^2) - (\sum x^2)}$$

$$\bar{X}_{PR} = \frac{\sum x}{n}$$

$$\bar{X}_{Rec} = \frac{\bar{X}_{PR}}{AV} \times 100$$

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (x_i - M)^2}{n - 1}}$$

$$RSD = \frac{SD}{\bar{X}_{PR}} \times 100$$

$$R \text{ (Pearson Product Moment of Correlation)} = \frac{n(\sum xy) - (\sum x)(\sum y)}{\sqrt{[n\sum x^2 - (\sum x)^2][n\sum y^2 - (\sum y)^2]}}$$

$$R^2 = R \times R$$

$$SER = \sqrt{\left(\frac{1}{n-2}\right)\left[\sum (y - M_y)^2 - \frac{[\sum (x - M_x)(y - M_y)]^2}{\sum (x - M_x)^2}\right]}$$

$$\text{Acceptance Limits}_{\text{Mean}}\ (a, b) = (a \times AV) + b$$

$$\text{Acceptance Limits}_{SD}\ (c, d) = (c \times AV) + d$$

$$ASRL = AV - (2 \times SD)$$

~OR~

$$ASRL = \frac{[lowest\ concentration\ in\ study\ range]}{10}$$

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Appendix A (continued): Calculations

Example Microsoft Excel Formulae for Statistical Calculations*		
Abbreviation	*Description*	*Microsoft Excel formula*
AV	Assigned Value	--
a	Slope of M vs AV	=slope(SeriesA_cell1:SeriesA_cellN,SeriesB_cell1:SeriesB_cellN)
b	Intercept of M vs AV	=intercept(SeriesA_cell1:SeriesA_cellN,SeriesB_cell1:Series_BcellN)
c	Slope of AV vs SD	=slope(SeriesA_cell1:SeriesA_cellN,SeriesB_cell1:SeriesB_cellN)
d	Intercept of AV vs SD	=intercept(SeriesA_cell1:SeriesA_cellN,SeriesB_cell1:SeriesB_cellN)
$\bar{X}_{PR}$ or $\bar{X}_{Ref}$†	Mean	=average(cell1:cellN)
SD	Standard Deviation	=stdev(cell1:cellN)
RSD	Relative Standard Deviation	=((stdev(cell1:cellN))/(average(cell1:cellN)))*100
R^2	Correlation Coefficient squared	=(correl(SeriesA_cell1:SeriesA_cellN,SeriesB_cell1:SeriesB_cellN))^2
SER	Standard Error of Regression	=steyx(SeriesA_cell1:SeriesA_cellN,SeriesB_cell1:SeriesB_cellN)

*This is not an endorsement of Microsoft Excel. Other programs may be used for the statistical calculation protocol detailed in Section 5.6.

Note: "cellN" refers to the final cell in the data series.

†May refer to the mean of data set prior to removal of outliers ("robust mean") or after removal of outliers.

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Moved down [2]: †May refer to mean of data set prior to removal of outliers ("robust mean") or after removal of outliers.

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Appendix B

Checklist for Consideration of Pilot Study Data

Reference Section	*Query*	*Yes*	*No*
7.1 # data points used vs. # data points returned	[ ]%: >67%?		
7.2 ASRL = AV – (2 × SD)	[ ] Is lowest AV compatible with this ASRL?		
7.2 ASRL = 0.1 × [lowest conc in range]	[ ] Is lowest AV compatible with this ASRL?		
7.3 ASRL vs DL and LoQ	Is ASRL compatible with DLs and LoQs reported by labs in Pilot Study?		
7.3 Need to reconsider acceptance limits due to technologies?	Is the difference between reported DLs and LoQs due to differing technologies?		
7.4 Consider the span of acceptance limits between the lower and upper limit relative to concentration range.	log (highest conc/lowest conc) ÷ log (highest acceptance limit/lowest acceptance limit) = [ ] Is result >2? (Provides sufficient challenge to lab?)		
7.5 Other considerations	Data set comprised of ≥20 data points?		
	Data returned by ≥5 laboratories?		
	R^2 (Mean) ≥0.90 using $\bar{X}_{PR}$ vs. AV?		
	R^2 (SD) ≥0.75 using SD vs. AV?		
	R^2 (SD) ≥0.75 using SD vs. $\bar{X}_{PR}$?		

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