TNI Stationary Source Audit Sample Expert Committee meeting at the Environmental Measurement Symposium, Washington DC
August 9, 2012, 1:30 pm EDT

Attendance:

Maria Friedman – Chair TestAmerica (Laboratory)	Committee member	present
Mike Hayes Linde (Provider)	Committee member	absent
Michael Klein New Jersey DEP (State government)	Committee member	absent
Theresa Lowe CCI Environmental	Committee member	absent
Paul Meeter Weston Solutions (Stationary Source Tester)	Committee member	present*
Gregg O'Neal, North Carolina DAQ (State government)	Committee member	present*
Michael Schapira Enthalpy (Laboratory)	Committee member	present*
Jim Serne TRC Solutions (Stationary Source Tester)	Committee member	present*
Richard Swartz, Vice-chair Missouri DNR (State government)	Committee member	present*
Stanley Tong EPA Region 9 (Federal government)	Committee member	present*
Ken Jackson TNI (Program Administrator)	Program Administrator	absent
Ty Garber Wibby (Provider)	Associate member	absent
Shawn Kassner ERA (Provider)	Associate member	present
Mike Miller (Member at large)	Associate member	present
Wayne Stollings (Triangle Env. Services)	Guest	present*
William Daystrom TNI (Webmaster)	Guest	present
Charles Simon (VOC Reporting)	Guest	present*

*present by teleconference

1) Introductions

The Committee Members, Associate Committee Members and guests on the telephone introduced themselves. Maria had those Committee members in the room and others present also introduce themselves.

Maria outlined the agenda, to include the SSAS Program Update, the SSAS Table update and a report from the Method 25 subcommittee regarding proposed updates to the method and a sampling guide.

2) Review of minutes from the teleconference on July 10, 2012.

There were no comments. However, since a quorum was not present at this time, Maria suggested soliciting any comments from absent Committee Members and then voting by e-mail.

3) SSAS Table Management SOP

The draft SOP (Attachment 1) was displayed on the screen. Maria said it is similar to the TNI FoPT management SOP. It would be needed to address changes required for future changes to the SSAS Table methods and analytes, and how the Committee will deal with new methods or analytes. When methods/analytes are in the table, providers will be able to offer them as audit samples (at least two providers will be required). Also addressed in the SOP is the task of how to define acceptance criteria and concentration ranges, etc., editorial issues, such as revision dates, and how proposed changes would be reviewed and adopted. Maria said the SSAS Table management subcommittee was chaired by Shawn Kassner, and its recommended changes would need to be approved by the Expert Committee and by EPA.

Maria showed the general procedure for modification of the SSAS Table. The EPA Final Rule on privatization of the SSAS program requires historical data to be used to update acceptance criteria and it must provide for 90% of laboratories passing. The SOP says that at least 20 data points will be needed. When new methods or analytes are added, it is not likely that historical data will be available. The Change Request Application (CRA) form to be completed for addition or deletion of analytes/methods was described. Charles Simon commented that it does not say anywhere that this is restricted to adding new analytes or removing analytes. Nothing restricts anyone from filing a CRA to modify existing methods or anything else in the table. Maria went on to say data could be used from a pilot study, from EPA, or from PT laboratories, but that is confined to new methods or analytes. Historical data must be used for existing methods. Maria referred to Section 5.2.3.6. This originally said for new methods and analytes we would set up a default acceptance range of 10% – 200% recovery, which is not acceptable to EPA or to regulators since it is so wide. The Section now describes sources that can be used for establishing acceptance criteria. As Charles has suggested, Maria said the Committee must consider if clarifying language is needed to say that for existing methods, data from audit samples must be used. She referred to an earlier e-mail message (Attachment 2). Gregg cautioned, if new audit samples for existing methods (e.g., added interferent) are to be added, there would not be historical data. However, Charles said there would be a pilot study for the data. Maria said we could add “as required in the EPA Final Rule, historical audit sample data must be used when considering requests to change acceptance criteria for existing methods and analytes.” So if adding new audit samples even for existing methods there will be no existing criteria and the new audits would be considered initial criteria and the pilot study could be used.

Maria asked if there were more comments on the SOP. Paul questioned (Section 5.2.1) whether the stated 21 days of the receipt of the request to perform a preliminary review is enough time, and suggested 30 days might be better. Maria said the Committee could hold a special meeting if necessary, but would change it to 30 days pending further discussion.

4) Recommendations from the Method 25 subcommittee

The subcommittee report (attachment 3) was displayed. Charles described each of the 13 recommendations.

I. Subcommittee Approved Method 25 Audit Procedure Recommended Changes

Recommendation 1. Charles asked Maria if it will be necessary to wait for the biennial review, required in the Final Rule, before such adjustment can be made. He added that, since this audit is a 2-step process (all others are a 1-step process), results are very varied with a lot of failures. Maria replied that because there is a SSAS central database, the data can be reviewed anytime and if necessary a CRA can be prepared to make an adjustment.

Recommendation 2. Charles said recommending methane as the tracer gas goes back to the old Method 25, when both methane and carbon dioxide were added. Maria asked, if methane is reported, does that mean it will be regulated? Charles said there will be the non-methane organics and the methane. The methane is not reportable in the real sample, but in the audit sample it should be reported as the tracer. Maria suggested that creates a discrepancy between real samples and audit samples. Charles responded there cannot be a requirement for the accuracy of methane. It just indicates if there is a problem in the field or in the laboratory. It was pointed out, however, if the methane fails it is unlikely the VOC will pass.

Recommendation 3. Maria asked, if applying blank subtraction to all samples, would you apply it to your calibration and control samples? Wayne responded no, because that process is not involved with the analysis. Charles said the reason for allowing but not requiring a blank is because if you are in a situation where you know all samples will be above (say) 500 ppmC, a 10 ppmC blank would not be significant. In response to a question by Maria, Charles said the reference EPA340/1-91-008 is a guidance document that coordinates several methods of VOC testing.

Recommendation 4. Maria pointed out that just allowing the protocol gas vendors to supply the samples without accreditation as a provider would not be acceptable to TNI. Charles responded that they are recommending the TNI requirement should be changed in this case.

Recommendation 5. Maria asked, since modification to the method is recommended, shouldn't they reference the CFR method rather than the guidance document? Charles said there are several ways of doing it, but the subcommittee was not tasked with the "how to". If asked, they could provide a recommended wording change to the CFR and submit that with these recommendations. Should they re-write the CFR or the guidance document, or both? Maria suggested the subcommittee should wait until the Expert Committee has considered whether changes should be made to the CFR or just the guidance document.

Recommendation 6. There were no questions or comments.

II. Subcommittee Approved Method 25 Field Procedure Recommended Changes

Recommendation 1. Paul said, regarding sources with >40% moisture, the method provides a guideline of multiplying % carbon dioxide with %moisture, and if the product is >100, the bias would be considered significant. He asked if the comment should be re-worded to include this thought process. Charles responded no, saying the subcommittee has addressed that potential interference in the 10 future discussion items. They will include data from a study that has been performed to justify their rationale.

Paul added that dry ice may not always be available to a field tester. He asked if water ice could be used instead, and if there is evidence that dry ice is better than water ice. Wayne responded the colder the better since it is physical entrapment.

Recommendations 2, 3, and 4

There were no questions or comments.

III. Subcommittee Approved Method 25 Laboratory Procedure Recommended Changes

There were no questions or comments.

Remaining topics.

There were no questions or comments.

5) Update on accreditation of providers.

A representative from A2LA confirmed they still have just one accredited provider (ERA). Some other providers have expressed interest, but none have applied. If they are an existing PT provider, this would be just an extension to their program and it could go quite quickly (e.g., 1 month). A new provider requires about 5 months on average. Maria suggested if other providers show interest and have questions, they could be directed to this Expert Committee.

It was reported that ACLASS has a similar time frame. They have submitted their documentation for TNI approval. Two prospective providers have shown interest.

Maria suggested the Protocol vendors could partner with accredited providers, and Charles said he is trying to encourage that. A gas provider (Air Gas) is currently accredited for breath alcohol by A2LA as a PT provider, though not under the TNI program. Their scope of accreditation is on the A2LA website, and it was suggested it may be worth reaching out to them. However, Wayne said he has already spoken to them, and so far they are not interested.

5) Next Steps

The next call will be August 20. Maria said the Committee would start with SASS table management SOP, and would then vote on each recommendation in the Method 25 subcommittee report before the subcommittee drafts the formal language. It will also be discussed whether amendments should be proposed to the guidance document or to the CFR. Mike Miller said it could take several years to modify the CFR so wouldn't it be quicker to amend the guide? Stan said, although EPA is currently reviewing the methods, the deadline for comments closed on March 9. He will check if there is an extension, but he reminded everyone that a change to the method (CFR) can only be done through the Federal Register and guidance documents are not subject to that requirement.

6) Adjournment

The meeting was adjourned at 3:15 pm EDT.

Attachment 1 Draft SSAS Table Management SOP

1.0 Purpose and Applicability

This Standard Operating Procedure (SOP) delineates procedures for updating the Stationary Source Audit Sample (SSAS) Table. The procedures described herein apply to all methods and analytes used in the TNI SSAS Program.

2.0 Summary

A request is made by a Participant to modify the SSAS Table. The SSAS Expert Committee reviews all requests, such as adding or removing methods or analytes, setting or changing concentration ranges and acceptance criteria, and correcting typographical and formatting errors. When any modification is approved, a new revision number and effective date are established according to defined timelines.

3.0 Definitions

Audit Sample Reporting Limit (ASRL): The lowest result that could be obtained from the lowest spike level for an analyte, provided in the SSAS Table as guidance to laboratories analyzing SSAS samples.

Participant: The Facility, Regulatory Agency, Stationary Source Tester, Laboratory, and Provider participating in a stationary source test.

Regulatory Agency: The federal, state, local, or tribal agency having responsibility and accountability for overseeing testing of atmospheric emissions from stationary sources.

SSAS Table: Table in which the analytes and acceptance limits for audit sample materials are defined.

Sponsor: A Regulatory Agency that agrees with the need to add a method, analyte, or group of analytes to the SSAS Table.

4.0 General Procedure for SSAS Table Modification

4.1 Requests to modify the SSAS Table may be made by a Participant in the TNI SSAS Program. Modifications requested may be one or more of the types listed below:

- 4.1.1 Addition or removal of a method
- 4.1.2 Addition or removal of an analyte
- 4.1.3 Changes to NELAC (TNI) Analyte Codes
- 4.1.4 Changes to concentration ranges, units, acceptance criteria, and ASRLs
- 4.1.5 Changes to footnotes
- 4.1.6 Changes to group headers
- 4.1.7 Changes to effective dates
- 4.1.8 Changes as a result of the biennial SSAS Table review per the TNI SSAS Standard
- 4.1.9 Corrections to typographical or formatting errors

4.1.9.1 Changes to numerical values or acceptance criteria are not considered typographical errors.

4.1.9.2 Corrections to typographical or formatting errors do not require a change in the SSAS Table's Effective Date.

4.2 Request for typographical or formatting corrections must be sent to the SSAS Expert Committee Chair, whose contact information is available on the TNI SSAS Expert Committee page on the TNI website.

4.3 Request for modifications other than typographical or formatting corrections must be initiated using the SSAS Table Change Request Application (CRA) and submitted electronically to the SSAS Expert Committee Chair.

4.3.1 A CRA must be filled out for each type of modification requested. See Attachment 1.

4.3.2 If the modification requested is for the addition or removal of a method, analyte, or group of analytes, a Sponsor is required (see Section 5.1.1). All other modifications do not require a Sponsor.

4.4 Depending on the type of modification requested, the SSAS Expert Committee may direct the SSAS Table Subcommittee to review requested modification and prepare formal recommendations for consideration by the SSAS Expert Committee voting members. The SSAS Expert Committee will work with the subcommittee to set acceptable timetable goals for completion of their review and proposal.

4.5 Modifications to the SSAS Table, when deemed necessary, must be first approved by the SSAS Expert Committee and then by EPA. Approved modifications will be effective 6 months thereafter, or on the date approved by the SSAS Expert Committee, whichever is sooner.

4.6 The newly modified SSAS Table will reflect a new effective date and a new revision number.

4.6.1 When the SSAS Table undergoes modifications not related to typographical or formatting corrections, the assigned revision number follows a progression of Rev.1.0, 2.0, 3.0, and so on.

4.6.2 When the SSAS Table undergoes modifications related to typographical or formatting corrections, the assigned revision number follows a progression of Rev.
1.1, 1.2, 1.3, and so on.

4.7 Public notice will be posted on the TNI website as notification that an updated SSAS Table has been approved.

4.8 In the event of initial acceptance criteria, derived from sources other than historical audit sample data, having been assigned to new analytes, ~~default acceptance criteria having been assigned to new analytes due to insufficient historical data,~~ the SSAS Expert Committee shall monitor the SSAS Central Database until 20 data points, or other number as determined appropriate by the SSAS Expert Committee, have been collected for the new analyte. At that
time, the SSAS Expert Committee shall evaluate the collected data to assign new acceptance criteria based upon the historical data.

5.0 Addition of Methods or Analytes

5.1 **Requirements for Requests**

5.1.1 Requests to add a method, analyte, or group of analytes to the SSAS Table must be sponsored by at least one Regulatory Agency. If the requestor is a Regulatory Agency, an additional sponsor is not required.

5.1.2 The CRA submittal shall include:

5.1.2.1 The method(s) and/or analyte(s) being requested

5.1.2.2 The requestor's reason(s) for adding the method(s) or analyte(s)

5.1.2.3 The proposed spiking concentration and initial acceptance criteria

5.1.2.4 The required supporting documentation noted on the CRA

5.2 **Review Procedure**

5.2.1 The SSAS Expert Committee completes a preliminary review of the request, within 21 days of receipt of the request, to determine whether to proceed with a formal review. Factors that may determine whether to proceed include, but are not limited to, regulatory need and impact to the TNI SSAS Program.

5.2.2 If the SSAS Expert Committee determines that the request merits a formal review, the SSAS Expert Committee notifies EPA to ascertain whether or not EPA would consider allowing the addition of the requested method, analyte, or group of analytes. If the SSAS Expert Committee determines that the request is without merit, the SSAS Expert Committee Chair shall notify the requestor of the SSAS Expert Committee's decision within 14 days thereafter.

5.2.3 If EPA deems the request appropriate, the SSAS Expert Committee will initiate a formal review of the request within 14 days of notification from EPA. When deemed necessary, the SSAS Table Subcommittee will be requested to review the request and submit a recommendation, within agreed upon timelines, to the SSAS Expert Committee. Whether the formal review is undertaken by the SSAS Expert Committee or by the SSAS Table Subcommittee, the formal review shall include, but not be limited to, the following elements:

5.2.3.1 The CRA and supplied documentation

5.2.3.2 Availability of SSAS Providers to provide an audit sample compatible with the proposed method(s) and spiked with the proposed analyte(s)

5.2.3.3 Historical data availability

5.2.3.4 Technical feasibility – This must include one or more method validation studies showing that the analyte(s) can be measured at the requested concentration range by the specified SSAS method. SSAS Providers may be requested to provide input, based on their experience with the requested new method, analyte, or group of analytes.

5.2.3.5 Concentration range – The requested concentration range will be evaluated for suitability based on input from Participants. A default concentration range appropriate to the method will be assigned, if necessary, based on a

reasonable expectation of method and analyte performance.

5.2.3.6 Initial acceptance criteria – When historical audit sample data do not exist, ~~default~~ acceptance criteria ~~of 10% to 200% Recovery~~ will be established using one or more of the following sources, unless more appropriate acceptance criteria can be derived from data supplied with the CRA:

5.2.3.6.1 Results of a pilot study, if available

5.2.3.6.2 Historical data from samples other than audit samples, using the same sample matrix and method as would be used with audit samples. Acceptable sources include, but are not limited to, data from the EPA Office of Water, the EPA Office of Solid Waste, or the EPA Office of Air and Radiation.

5.2.3.6.3 An expert evaluation of the capabilities of the method, based on calibration requirements and prior experience with other samples, recorded in CRA

5.2.3.7 Cost impact assessment to Providers, Laboratories, and Facilities

5.2.3.8 NELAC (TNI) Method or Analyte Code – Does one exist?

5.2.4 The SSAS Expert Committee Chair shall notify the requestor of the SSAS Expert Committee's decision within 14 days of completion of the formal review.

5.2.5 The entire review process shall be documented, including, but not limited to, minutes of relevant meetings, checklists, data pertaining to the request, calculations, graphs, and other information used in the decision-making process. Documentation shall be submitted to TNI for posting and archiving.

5.3 **Follow-up**

5.3.1 In the event of initial acceptance criteria, derived from sources other than historical audit sample data, having been assigned to new analytes, ~~default acceptance criteria having been assigned to new analytes due to insufficient historical data,~~ the SSAS Expert Committee shall monitor the SSAS Central Database until 20 data points, or other number as determined appropriate by the SSAS Expert Committee, have been collected for the new analyte. At that time, the SSAS Expert Committee shall evaluate the collected data to assign new acceptance criteria based upon the historical data.

6.0 Removal of Methods or Analytes

6.1 **Requirements for Requests**

6.1.1 Requests to remove a method, analyte, or group of analytes from the SSAS Table must be sponsored by at least one Regulatory Agency. If the requestor is a Regulatory Agency, an additional sponsor is not required.

6.1.2 The CRA submittal shall include:

6.1.2.1 The method(s) or analyte(s) to be removed

6.1.2.2 The requestor's reason(s) for removing the method(s) or analyte(s)

6.1.2.3 The required supporting documentation noted on the CRA.

6.2 **Review Procedure**

6.2.1 The SSAS Expert Committee completes a preliminary review of the request, within 21 days of receipt of the request, to determine whether to proceed with a formal review. Factors that may determine whether to proceed include, but are not limited to, regulatory need and impact to the TNI SSAS Program.

6.2.2 If the SSAS Expert Committee determines that the request merits a formal review, the SSAS Expert Committee notifies EPA to ascertain whether or not EPA would consider allowing the removal of the requested method, analyte, or group of analytes. If the SSAS Expert Committee determines that the request is without merit, the SSAS Expert Committee Chair will notify the requestor of the SSAS Expert Committee's decision within 14 days thereafter.

6.2.3 If EPA deems the request appropriate, the SSAS Expert Committee will initiate a formal review of the request within 14 days of notification from EPA. When deemed necessary, the SSAS Table Subcommittee will be requested to review the request and submit a recommendation, within agreed upon timelines, to the SSAS Expert Committee. Whether the formal review is undertaken by the SSAS Expert Committee or by the SSAS Table Subcommittee, the formal review shall include, but not be limited to, the following elements:

6.2.3.1 The CRA and supplied documentation

6.2.3.2 Impact on other SSAS Table – Does this change impact other methods or analytes?

6.2.4 The SSAS Expert Committee Chair shall notify the requestor of the SSAS Expert Committee's decision within 14 days of completion of the formal review.

6.2.5 The entire review process shall be documented, including, but not limited to, minutes of relevant meetings, checklists, data pertaining to the request, calculations, graphs, and other information used in the decision-making process. Documentation shall be submitted to TNI for posting and archiving.

7.0 References

7.1 TNI Standard, Stationary Source Audit Sample Program, Volume 1, Module 1: General Requirements for Stationary Source Audit Sample Providers, current revision

7.2 TNI Standard, Stationary Source Audit Sample Program, Volume 1, Module 2: General Requirements for an Accreditor of Stationary Source Audit Sample Providers, current revision

7.3 TNI Standard, Stationary Source Audit Sample Program, Volume 1, Module 3: Requirements for Participation in the TNI Stationary Source Audit Sample Program, current revision

8.0 SOP Approved Changes

Previous SOP No.	New SOP No.	Date of Change	Description of Change
	2-102	xx/xx/2012	New Document

9.0 Tables, Figures, Diagrams, Charts, Examples, Checklists, and Appendices *(will be added when SOP is approved)*

Attachment 2

6 - Sent by Maria Friedman on 8-7-2012:

To all:

Method 25 is not a new method being added to the SSAS Table. To that effect, the provisions under Section 5 (e.g., use of pilot study) in the SSAS Table Management SOP do not apply. The acceptance criteria for this method already existed, were reviewed when the audit program was privatized to ensure that the requirement (i.e., 90% of well qualified labs passing future audits) of the Final Rule was met, and revisions were already approved.

Method 25 acceptance criteria and concentration range will remain in effect until we have new audit sample data. This update will happen biennially or earlier if new data that will improve the acceptance criteria become available.

Thank you.

Maria Friedman
(949) 307-0949 - cell phone
(714) 656-4311 - office (direct line)

7 - Sent by Charles Simon on 8-8-2012:

Maria,
Why can't we (the M25 Improvement subcommittee) as participants in the SSAS program make a request under this SOP to change the acceptance criteria for M25. Yes, it's an existing method. How does that restrict applying for a modification of the SSAS Table?

"Purpose and Applicability
This Standard Operating Procedure (SOP) delineates procedures for updating the Stationary Source Audit Sample (SSAS) Table. **The procedures described herein apply to all methods and analytes used in the TNI SSAS Program**.

A request is made by a Participant to modify the SSAS Table. The SSAS Expert Committee reviews all requests, such as adding or removing methods or analytes, setting or **changing** concentration ranges and **acceptance criteria.** and correcting typographical and formatting errors. When any modification is approved, a new revision number and effective date are established according to defined timelines.

General Procedure for SSAS Table Modification
4.1 Requests to modify the SSAS Table may be made by a Participant in the TNI SSAS Program. Modifications requested may be one or more of the types listed below:
4.1.4 **Changes to** concentration ranges, units, **acceptance criteria**, and ASRLs"

We can still file an application to change the M25 audit acceptance criteria and rely on the pilot study data to produce the same regression-based acceptance criteria with different limits that meet the current CFR requirements for statical pass rates. If I'm reading the SOP correctly, it would be up to the TNI-SSAS committee to recommended to EPA acceptance or rejection of this application.

Do I have that wrong? We're looking for a way to define more reasonable starting acceptance criteria for M25 audits without having to modify the CFR now that we have an 'historical' pilot study to rely upon.

-Charles

8 - Sent by Maria Friedman on 8-8-2012:

Charles,

The concept of using pilot study data to determine acceptance criteria was proposed by EPA only with regard to setting initial acceptance criteria for new methods and analytes for which historical data do not exist. EPA did not propose, and the draft SSAS Table Management SOP does not suggest, that pilot study data may also be used to evaluate changes to existing methods and analytes. EPA's expectation, as written in the Final Rule and implemented in the TNI SSAS Program Standard Volume 1 Module 2, is that acceptance criteria for existing methods and analytes will be evaluated, at least biennially, using "historical audit sample data."

The draft SSAS Table Management SOP does not repeat this requirement, so I think we should insert a new section before section 4.4 (renumbering subsequent sections), stating "As required in the EPA Final Rule, historical audit sample data must be used when considering requests to change acceptance criteria for existing methods and analytes."

Additionally, the SSAS Table Subcommittee, under direction of the Expert Committee, was chartered with the responsibility for evaluating data and submitting proposals for changes to acceptance criteria, for consideration by the Expert Committee. In other words, we have a process we need to follow - one which we have followed in the past, and that we are formalizing in the SSAS Table Management SOP. For consistency and openness in how the TNI SSAS Program is managed, it is important that we adhere to that process.

Let us proceed forward with what we have and focus on method improvement, standardized sampling procedure, and soliciting providers for M25.

Thank you.

Maria Friedman
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Attachment 3

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Newberry, Florida 32669 Phone: (352) 472-2899

August 6, 2012

M25 Improvement Subcommittee report to TNI-SSAS full Committee

Charles Simon (chair)	VOC Reporting Inc., lab analyst & field tester	cgsimon@gowebway.com
Wayne Stollings	Triangle Environmental Services, lab analyst	Wstollings@aol.com
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Tom Mattei	Air Test Auditors, field testing consultant tmattei@airtestauditors.com	

Below are the thirteen recommendations for Method 25 improvements that our sub-committee approved along with a rationale for each topic. Several of these seek to formalize EPA guidance on Method 25 published in the "Manual for Coordination of VOC Emissions Testing Using EPA Methods 18, 21, 25 and 25A" (EPA340/1-91-008). Votes were submitted by representatives from two AETBs, three regulatory agencies, three vendors, and the two Method 25 laboratories.

The wording of each recommendation has been discussed by the members and their comments are incorporated. In some cases several alternative recommendations are proposed. This sub-committee has not been tasked with preparing final legal wording for the recommended improvements. We've tried to be as accurate as possible in the description of each proposed improvement, and the rationale

for proposing it. Any questions can be addressed to me and will be passed on to the full sub-committee. It would be beneficial if this Subcommittee could review the wording of each improvement as proposed by EPA.

There are ten remaining topics that were undecided in our first round of voting. We are currently addressing these issues.

We also note one error that remains in Method 25 at 12.1 under the definition of "N". The carbon number for decane is given as 12, whereas it is actually 10.

-Charles Simon

I. Sub-committee Approved Method 25 Audit Procedure Recommended Changes

1. Use historic M25 audit performance data with one standard deviation instead of two to generate regression based acceptance criteria for Method 25 audits under the SSAS program.

Rationale: Historic Method 25 audit performance data used by the TNI-SSAS committee with two standard deviations results in regression based acceptance criteria that blow–up at low concentrations. This effectively makes Method 25 audit acceptance criteria >±80% at low concentrations (see Figure 1). The committee followed the TNI SOP for determining acceptance criteria and the EPA final rule requirements to develop acceptance criteria that 90% of labs could pass 95% of the time based on historic performance. These large initial acceptance criteria are considered inappropriate by some regulators.

This recommendation will be incorporated by default in the SSAS program under the most recent proposed version of the SSAS Table Management SOP (8/1/12), which includes new language supported by EPA that allows the use of a pilot study of audit sample performance for setting initial acceptance criteria instead of historic data, either through a Change Request Application (CRA), or by direct initial action of the TNI committee and EPA. Either way, a review of the criteria will take place after 20 data points are reported from the actual audit program.

In September of 2011, on behalf of TNI, the only two laboratories providing Method 25 services outside of California undertook a pilot study of their ability to accurately analyze Method 25 audit samples collected in the lab and in the field over a concentration range of 60-3000 ppmC. A protocol gas supplier, Liquid Technology, produced 12 high-pressure Method 25 audit samples in 2L cylinders using the historic Method 25 non-methane-organic (NMO) audit gas components propane, ethane and methyl-ethyl-ketone (MEK) combined with ~5% CO2 (as the major interferent) and diluted in air. The study was completed in December, 2011.

Results from 46 field tests were grouped in 6 concentration ranges of approximately 60, 120, 250, 650, 1500 and 2900 ppmC. There were 8 samples in each range except the highest range, where there were 6 samples.
Two standard deviations were added to these averages and results showed:

- ❒ Method 25 audit samples with total NMO concentrations >150 ppmC passed under the historic acceptance criteria of ±20%

- ❒ Method 25 audit samples with total NMO concentrations <150 ppmC passed at ±50% without blank-correction, and at ±30% with blank-correction.

If the results of this pilot study are used along with the TNI-SSAS committee recommendation to have a lower limit of 150 ppmC for Method 25 audits, the historic acceptance criteria of ±20% will be an appropriate starting point based on the pilot study results. If Method 25 audit samples with <150 ppmC are included, the acceptance criteria should start at ±50% based on the pilot study results without blank correction, and at ±30% with an equipment-blank correction as defined in EPA340/1-91-008, page 6-12 (see Table 1. for pilot study results).

2. Designate methane as the "tracer gas" for the Method 25 audits.

Rationale: Inclusion of 100-1000 ppm methane in the Method 25 audit gases would provide an independent measure of the sample integrity. Since all of the methane would be collected in the sample tank, its concentration would provide an independent measure of the amount of audit gas that was pulled through the Method 25 sample train in the field. This has been a major reason for historic audit failures.

3. Allow, but don't require, either an equipment trip blank as defined in EPA340/1-91-008, page 6-12 with a maximum concentration of 10 mgC/m3, ***or*** a zero-audit blank as described in II.2 below with a maximum concentration of 15 mgC/m3, to be subtracted from all samples.

Rationale: Method 25 equipment or "trip" blank levels for a 5 liter sample average ~15 ±5 ppmC, or ~7.5 mgC/m^3, for both laboratories. This carbon background is ubiquitous with the method and the variance (sigma, σ) of the blank leads to the 50 ppmC limit of quantization of Method 25 (i.e., 10σ). The background (equipment blank) becomes significant at the 10% level, or at ~150 ppmC. Failure to subtract this background signal was a leading cause of both historic and recent (2011 pilot study) audit failures at low concentrations. When the results of the 2011 Method 25 audit study were corrected by each laboratory's blank level, all reported concentrations were within ±23% of the manufacturer's reported concentrations (see Table 1.). This is the strongest documented evidence to date of the importance of background subtraction for Method 25, particularly at low concentrations.

4. Allow EPA protocol gas vendors to produce the Method 25 audit gases according to established and approved procedures.

Rationale: Protocol gas vendors routinely supply the calibration and "audit" gases for the on-site instrumental versions of Reference Methods 3A, 6C, 7E, 10, 18, 25A and 25B. These products are high pressure multi-component gases blended in air or other balance-gas according to EPA protocols. Vendors that participate in the EPA Protocol Gas Vendor Program (PGVP) have additional oversight. One of these vendors, Liquid Technology, produced the Method 25 audit samples used in the 2011 pilot study.

Method 25 is the only method in the SSAS program that requires high-pressure gas cylinders for audit samples, the same type of product routinely supplied to calibrate and audit the on-site instrumental

Reference Test Methods. Since the production and certification protocols are already in place and practiced by protocol gas vendors, it should be acceptable to have the same protocols used to make and supply the Method 25 high-pressure audit gases.

5. When sampling audits, require the bypass to be connected to a low-flow rotameter to ensure that audit gas is going to the sample train and that no more audit gas is used than needed. This rotameter should be fully opened, with the audit gas flow controlled by the audit tank regulator and an excess flow of 50-100 ml/min.

Rationale: Historically the excess flow from the Method 25 audit gas cylinder was vented from a tee union at the junction of the Method 25 sample train probe tip. There is currently no designated procedure to insure that this excess flow is continual and not excessive. This has been a major reason of historic poor performance that would be corrected by this procedure designation.

6. The audit cylinder pre and post gas pressures should be recorded on the Field Data Sheet. An audit sample should not be used if the starting pressure is below 200 psig.

Rationale: A recommended updated Method 25 Field Data Sheet with time entry spaces for these events is shown in Figure 2. At a sample collection rate of 100 ml/min, and an excess flow rate of 100 ml/min, it takes 1 liter of audit gas to purge the Method 25 train for 10 minutes, and 12 liters of audit gas are expended during the collection of the sample over an hour, for a total of 13 liters of audit gas. The Method 25 audit cylinders have 2 liters of internal volume, so (13 liter/2 liters)(15 psi) ~ 100 psi of pressure are used when each sample is collected under ideal conditions. In the 2011 study the pressure drop averaged ~150 psi for each audit sample, indicating the 200 psi lower limit is appropriate to ensure an adequate sample for collection.

II. Sub-committee Approved Method 25 Field Procedure Changes

1. Designate the use of dual traps, one in ice water followed by one in dry ice, as recommended in EPA340/1-91-008, page 6-12, for sources with high moisture (>40%) to prevent trap plugging and the need to warm the trap to recover flow during sampling.

Rationale: This technique has been used routinely since 1989 to prevent plugging from frozen water when the Method 25 cryogenic trap design changed from an impinger-type unit to a u-tube. This change would formalize and/or reinforce the 1991 EPA guidelines.

2. Allow the generation of an equipment "trip blank" whereby a Method 25 tank and trap travel to and from a field site and are subsequently analyzed along with the samples, or, a "zero audit" blank whereby a certified hydrocarbon free gas is sampled in the field using a standard Method 25 sample train and procedures for collecting an audit gas, and then returned to the lab and analyzed with the samples.

Rationale: EPA340/1-91-008 describes the trip blank on page 6-12 as a Method 25 tank and trap pair that travel to and from the field and are subsequently analyzed with the samples. This change would formalize and/or reinforce the 1991 EPA guidelines and serve as the "blank" as described in the above section. A maximum value (cap) of 10 mg/m3, or 20 ppmC, is recommended for this blank.

Alternately, allow the generation of a zero-audit blank by the field team using a certified hydrocarbon free (<1 ppm THC) gas and the same procedures as those used to collect, recover and transport audit samples. A maximum value (cap) of 15 mg/m3, or 30 ppmC, is recommended for this blank.

The Subcommittee notes the issue of blanks applying to audits is an item that needs to be addressed with regard to consistency with NSPS and MACT Subpart A regulations.

3. Require the clock time (pre and post if applicable) of all significant/key steps of the sampling to be recorded on the Field Data Sheets to ensure they are properly performed, especially in cases of unobserved tests. These steps include: (a) leak checks (b) temperature/pressure readings (c) heat-up (d) purge (e) time dry ice is applied to traps.

Rationale: Regulators expressed concern for this change and field operators pointed out the importance of correlating sample events with production events after testing. A recommended updated Method 25 Field Data Sheet with time entry spaces for these events is presented in Figure 2.

4. Require crushed dry ice be added to the sample trap 10 min (not 30 minutes) before the start of sampling.

Rationale: Note: while "crushed dry ice" is mentioned in Section 7.1.1 of Method 25, it also needs to be mentioned in Section 8.1.3 to reinforce the point.

Adding crushed dry ice to the sample trap cooler 30 minutes prior to the start of sampling is physically unnecessary and overly restrictive for complex field operations. Data collected from thousands of cycles of cooling and heating traps between dry ice and ambient temperatures in the laboratory has shown conclusively that 5 minutes is more than enough time to affect cooling or warming. Frequently field tests are performed under tight time constraints and dry ice cannot be added safely to the Method 25 trap cooler prior to the sample train being in place on the stack. The 30-minute rule generates an additional delay in testing that is not justified by the physics of cryogenics.

5. When at all possible, trains should be dedicated as Inlet (high VOC loading) and Outlet (low VOC loading) trains across test programs.

Rationale: Method 25 does not prohibit this logical action, and most testers routinely dedicate their trains for high and low VOC sources during a test. This action minimizes contamination potential from carryover and should be explicitly required.

6. Require that the calculation for allowable train leak rate include the volume of all connecting tubing/fittings to the manometer.

Rationale: Method 25 does not state that this action should be taken, but it is implied in the leak rate calculation. This change would explicitly require the entire volume under vacuum to be used in the leak rate calculation.

III. Sub-committee Approved Method 25 Laboratory Procedure Recommended Changes

1. Report all carbonaceous compounds recovered from the analysis of the ICV.

Rationale: Method 25 currently requires CO2 and any non-methane organic compound found in the ICV to be added together to yield the condensable VOC fraction of the Method 25 sample. However, when an unexpectedly high-concentration sample overwhelms the Method 25 sample recovery system oxidation catalyst, carbon monoxide and even methane are generated along with partially combusted organic compounds. All of these compounds come from the condensed VOC and end up in the ICV. This change would explicitly require all of these carbonaceous gas concentrations to be summed to yield the best measurement of the high-VOC samples. In practice, once such a sample has been identified, subsequent samples are recovered under different conditions (e.g., higher than specified O2 flow rate, lower than specified sample flow rate, and larger or multiple ICVs).

There are ten remaining topics that were undecided in our first round of voting. We are currently addressing the following issues.

1. Designate the use of a heated probe/filter as optional.
2. Designate the use of a heated probe/filter as optional when any potential bias favors enforcement.
3. Allow modified filtration specifications for PCD inlet samples to prevent exclusion of organic aerosols that are bound for destruction/removal in the PCD.
4. Once the Method 25 sample train has been heated, keep it hot.
5. Take pre- and post-sampling tank temperatures at the tank surface simultaneous with tank absolute pressure measurements.
6. Require Method 7E type stratification check to justify single point sampling.
7. Require probe or probe extension with a gas residence time of <200 msec in the hot zone at a flow rate of 100 ml/min when the stack gas temperature is >500°F to prevent combustion of VOC in the probe.
8. Allow recovery of VOC from the Method 25 sample trap at temperatures up to 50°C less than the trap cleanout temperature.
9. Allow a calibrated low-range CO2-NDIR to determine the Method 25 back-flush and sample recovery endpoints.
10. Allow gravimetric measurements per Method 24 procedures of hexane & decane injections.