2009

[SECTOR]

[VOLUME 1, MODULE 1]

GENERAL REQUIREMENTS FOR STATIONARY SOURCE AUDIT SAMPLE PROVIDERS

TNI Voting Draft Standard

Drafted May 3, 2009

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PREFACE

This Standard is the result of many hours of effort by those volunteers on The NELAC Institute (TNI) Stationary Source Audit Sample Expert Committee. The TNI Board of Directors wishes to thank these committee members for their efforts in preparing this Standard as well as those TNI members who offered comments during the drafting process.

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GENERAL REQUIREMENTS FOR STATIONARY SOURCE AUDIT SAMPLE PROVIDERS

1.0 INTRODUCTION, SCOPE, AND APPLICABILITY

1.1 Introduction

This Standard (Volume 1, Module 1) specifies the requirements for providers of stationary source audit samples, hereafter referred to as "audit samples," used for the confirmation of stationary source testing results.

1.2 Scope

The TNI Stationary Source Audit Sample Program (SSAS Program) includes the following elements:

a) The production and supply of audit samples that challenge the critical components of each analytical procedure, from initial sample collection to final data analysis;

b) The production and supply of audit samples that are as similar to real-world samples as are reasonably possible and are representative of materials analyzed for environmental regulatory programs, regulatory agencies, and communities;

c) The yielding of audit sample data that are technically defensible on the basis of the type and quality of the audit samples provided;

d) The preparation of audit samples that pose equivalent difficulty and challenge, regardless of the manner in which they are designed and manufactured by the Stationary Source Audit Sample Providers, hereafter referred to as "Providers."

e) Establishment of requirements for Facilities, Regulatory Agencies, Stationary Source Testers, Laboratories, Providers, and Stationary Source Audit Sample Provider Accreditors, hereafter referred to as "Provider Accreditors," participating in the SSAS Program.

1.3 Applicability

This Standard (Volume 1, Module 1) does not address issues of laboratory accreditation.

2.0 REFERENCES

2.1 *ISO 9001 Quality Management Systems – Requirements.*

2.2 *ISO/IEC 17025 General requirements for the competence of testing and calibration laboratories.*

2.3 *ISO Guide 34 General requirements for the competence of reference material producers.*

2.4 *ILAC G-13 Guidelines for the Requirements for the Competence of Providers of Proficiency Testing Schemes.*

3.0 TERMS AND DEFINITIONS

For the purpose of this Standard (Volume 1, Module 1), the relevant terms and definitions conform to *ISO/IEC 17011:2004(E), Clause 3* and *ISO/IEC 17025:2005(E), Clause 3*. Additional relevant terms are defined below.

3.1 **Acceptance Limits:** The range of values that constitute acceptable performance for a Participant providing results for an audit sample material.

3.2 **Analyte of Interest:** Target analyte that is spiked into the audit sample, as requested by the Participants.

3.3 **Assigned Value:** Value attributed to a particular quantity and accepted, sometimes by convention, as having an uncertainty appropriate for a given purpose.

Note: See Section 6.4 of this Standard for further discussion of assigned values.

3.4 **Facility:** The responsible owner or operator for the stationary source test or their authorized representative.

3.5 **Laboratory:** The organization that analyzes the samples collected during the stationary source test to determine emissions. This organization may be (1) the Stationary Source Tester or analytical chemist contractor analyzing the samples at the facility being tested or in a mobile laboratory or (2) an analytical laboratory or Stationary Source Tester analyzing the samples in their laboratory facility.

3.6 **Participants:** The Facilities, Regulatory Agencies, Stationary Source Testers, Laboratories, and Providers participating in a stationary source test.

3.7 **Referee Laboratory:** An independent laboratory that analyzes samples to provide a second opinion.

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

3.9 **Stationary source:** Any building, structure, facility, or installation that emits or may emit any air pollutant.

3.10 **Stationary Source Audit Sample (audit sample):** A sample, the composition of which is unknown to the Stationary Source Testers and Laboratory, and that is provided to evaluate whether the Stationary Source Testers and/or Laboratory can produce measurement results within specified acceptance criteria. The audit sample is analyzed, or collected and analyzed, as part of the batch of field test samples using the same personnel, procedures, and materials.

3.11 **Stationary Source Audit Sample Central Database (SSAS 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.

3.12 **Stationary Source Audit Sample Number (SSAS Number):** A unique number assigned by the Provider to identify the shipment of audit samples to a particular Facility for a stationary source test.

3.13 **Stationary Source Audit Sample Program (SSAS Program):** The procedures and operations for providing rigorously controlled and standardized environmental samples, analyzing or collecting and analyzing them, reporting measured values, and reporting evaluations of the accuracy of the measured values.

3.14 **Stationary Source Audit Sample Provider (Provider):** A person or organization that offers an SSAS Program in accordance with Section 6.0 of the Provider Accreditor Standard (Volume 1, Module 2).

3.15 **Stationary Source Audit Sample Provider Accreditor (Provider Accreditor):** An organization that is evaluated and approved by TNI to accredit and monitor the performance of Providers.

3.16 **Stationary Source Audit Sample Table (SSAS Table):** Table in which the analytes and acceptance limits for audit sample materials are defined.

3.17 **Stationary Source Test:** The determination of the qualitative and/or quantitative composition of atmospheric emissions from a stationary source.

3.18 **Stationary Source Tester:** Person or persons testing a stationary source for atmospheric emissions.

3.19 **TNI Proficiency Testing Board (PT Board):** A board consisting of TNI members or affiliates, appointed by the TNI Board of Directors, which is responsible for the successful implementation and operation of the TNI SSAS Program. The duties of the PT Board are defined in the PT Board Charter.

4.0 PROVIDER ACCREDITATION

4.1 The Provider shall be accredited by a TNI-approved Provider Accreditor for every audit sample (as listed in the SSAS Table) they will offer in their SSAS Program.

4.2 In order to receive and maintain accreditation for any analyte per matrix, the Provider shall demonstrate compliance with all requirements of this Standard (Volume 1, Module 1) during onsite assessments and ongoing oversight conducted by the Provider Accreditor per Volume 1, Module 2.

4.3 Providers shall be subject to biennial onsite assessments conducted by their chosen TNI-approved Provider Accreditor. They may also be subject to unannounced assessments for cause.

4.4 Providers shall submit data from each of their audit sample manufactured lots to the Provider Accreditor for review to determine compliance with this Standard.

4.4.1 The information required in these submittals, including the format and frequency/timing, shall be determined by the Provider Accreditor.

4.4.2 The Provider shall not identify any Participant to the Provider Accreditor without the expressed written consent of the Participant.

4.5 Upon request by the Provider Accreditor, the Provider shall supply to the Provider Accreditor, at no charge, audit samples specified by the Provider Accreditor and which are included in the Provider's scope of accreditation, for submission to a referee laboratory.

4.6 If conflicts with the Provider Accreditor arise, Providers shall follow the Provider Accreditor's appeals process.

4.7 Unresolved conflicts with the Provider Accreditor shall be submitted to the PT Board.

5.0 MANAGEMENT REQUIREMENTS

5.1 Quality System Requirements

5.1.1 The Provider's quality management system shall meet the requirements of ISO 9001 for the design, production, testing, and distribution of audit samples, and the evaluation of audit sample analyses results or measurements.

5.1.2 The Provider's manufacturing system shall meet the requirements of ISO Guide 34.

5.1.3 The design and operation of the Provider's SSAS Program shall meet the relevant requirements of ILAC G-13 or replaced by ISO 17043, when approved.

5.1.4 The testing facilities used to support the verification, homogeneity, and stability testing required in this Standard (Volume 1, Module 1) shall meet the requirements of ISO 17025.

5.1.5 If the Provider holds specific accreditations related to any of the requirements in Sections 5.1.1 through 5.1.4, this shall not limit the Provider Accreditor's ability to assess and make determinations related to the Provider's conformance to these requirements.

5.1.6 Providers shall maintain all records related to each audit sample manufacturing lot for a minimum of five (5) years.

5.2 Provider Conflict of Interest and Confidentiality

Providers seeking to obtain or maintain accreditation shall:

a) Document and certify to the satisfaction of the Provider Accreditor that they do not have any conflict of interest with any Participant in their SSAS Program;

NOTE: Such a conflict of interest could take the form of a financial interest or sharing of personnel, facilities, or equipment with any Participant in the Provider's SSAS Program.

b) Inform all internal and contract personnel who perform work on the SSAS Program of the Provider's obligation to report personal and organizational conflicts of interest to the Provider Accreditor;

c) Have a continuing obligation to identify and report any actual or potential conflicts of interest arising during the performance of work in support of the SSAS Program;

d) Immediately make a full disclosure to the Provider Accreditor of any identified actual or potential organizational conflict of interest. The disclosure shall include a description of any action that the Provider has taken or proposes to take after consultation with the Provider Accreditor to avoid, mitigate, or neutralize the actual or potential conflict of interest;

e) Have written procedures to ensure that the confidentiality of data associated with audit samples and the SSAS Program is not compromised;

f) Not release the assigned values or acceptance limits of any audit sample prior to the reporting of the audit sample analyses results; and

g) Not disclose specific Facility, Laboratory, or Stationary Source Tester results or evaluations to any parties other than as specified in Section 11.1.2 without written release from the Facility, Laboratory, or Stationary Source Tester.

NOTE: Providers may release, without permission of participant Laboratories, summaries of participant Laboratory results that do not identify individual Laboratories.

5.3 Provider Facilities and Personnel

5.3.1 Providers shall have appropriate facilities, equipment, and analytical instrumentation in place to produce, analytically verify, distribute, and provide data evaluation and reporting functions for every audit sample for which they wish to obtain or maintain accreditation.

5.3.2 Providers shall employ sufficient technical and support staff to design, produce, analyze, distribute, and provide data evaluation and reporting functions for every audit sample for which they wish to achieve or maintain accreditation.

5.3.3 No portion of the design, production, testing, distribution, data collection, data evaluation, or data reporting functions may be outside the direct control of the Provider for any particular manufacturing lot. For the purposes of this Standard (Volume 1, Module 1), "direct control" means that these functions are performed in the Provider's facilities by the Provider's staff or are subcontracted by means of a written agreement with defined Provider supervision to ensure that all requirements of this Standard are met.

5.3.4 Any subcontracted function related to the design, production, testing, distribution, data collection, data evaluation, or data reporting shall be assessed by the Provider Accreditor and shall meet the applicable requirements of this Standard.

5.4 Complaints Handling

5.4.1 Providers shall have written procedures for handling both written and verbal complaints from Participants who receive audit sample reports.

5.4.2 Providers shall record all complaints received concerning their SSAS Program, including any remedial or corrective actions taken. This record shall be provided to the Provider Accreditor upon request.

5.4.3 Any complaint received by a Provider that remains unresolved after ninety (90) days shall be submitted to the Provider Accreditor.

5.5 Notification of Sample Integrity

If any audit sample or analyte used in the SSAS Program is found to not meet any of the requirements of this Standard (Volume 1, Module 1), the Provider shall notify all affected Participants and the Provider's Provider Accreditor within seven (7) calendar days of the discovery of the nonconformance.

6.0 AUDIT SAMPLE DESIGN AND MANUFACTURE

6.1 Design Review

Providers shall demonstrate to the satisfaction of the Provider Accreditor that their audit sample design and manufacturing processes:

a) Permit Participants, conforming to the calibration and quality control requirements of the analytical method(s) for which the audit sample was designed, to generate results that fall within the acceptance limits defined in the SSAS Table;

b) Provide equivalent challenge to all Participants; and

c) Result in Participant acceptable/not acceptable rates that are consistent with historical norms.

6.2 Audit Sample Matrices

The matrices of all audit samples shall, to the extent possible, resemble the matrices which Participants routinely analyze.

6.3 Audit Sample Analytes

6.3.1 Providers shall prepare audit samples that are compliant with the criteria defined by the SSAS Expert Committee and published in the SSAS Table on the TNI website. If requested by the Regulatory Agency and/or the Facility, analytes or ranges that are not listed in the SSAS Table may be included in an audit sample if the purpose and technical justification are documented, and if, where appropriate, the Regulatory Agency and/or Facility are notified in advance.

6.3.2 When the SSAS Expert Committee makes changes to the audit sample design criteria, Providers shall comply with the revised requirements per the SSAS Expert Committee's implementation schedule.

6.3.3 The Provider shall spike the analytes of interest into the audit sample according to the SSAS Table.

6.3.4 The Provider shall produce audit samples that conform to the method being tested.

6.3.5 The Provider shall be allowed to add interferences (not to be analyzed), normally present in the matrix being tested, to the audit sample.

6.4 Audit Sample Concentration Ranges

6.4.1 Providers shall supply audit samples that reflect the concentration ranges in the SSAS Table.

6.4.2 Assigned values for audit sample analytes that are measured (chemical concentrations, isotope activities, etc.) shall:

a) Be equal to the made-to values of the analytes based on gravimetric and volumetric measurements of a starting material of known concentration if possible, and if not possible, shall be set to the mean of the determined measured value; and

b) Be presented in three (3) significant figures.

7.0 AUDIT SAMPLE TESTING

7.1 Verification of Assigned Value

7.1.1 Providers shall analytically verify the assigned value of all analytes in all manufacturing lots of audit samples prior to use.

7.1.2 Providers shall verify the assigned value by direct analysis against a calibration standard made from, or traceable to, a primary reference material (e.g., National Institute of Standards and Technology), if available.

7.1.3 If a primary reference material is not available, then verification shall be performed against an independently prepared calibration material.

NOTE: An independently prepared calibration material is one prepared from a raw material source independent of the source used to prepare the audit sample or one prepared and documented by a source external to the Provider.

7.1.4 The assigned value verification analytical event shall also include the analysis of a second source reference material from a source independent of the calibration standard and the audit sample being verified.

7.1.5 The Provider shall have documented criteria for the acceptance of the results of the second source reference material.

7.1.6 The analytical method used by the Provider for assigned value verification shall have a repeatability relative standard deviation of not more than one-sixth of the acceptance limits for the participant Laboratories.

7.1.7 For test methods listed in the SSAS Table, where the method performance precludes the use of the one-sixth limit defined in Section 7.1.6, the Provider shall document the technical justification that the method used to verify the audit sample assigned value is adequate to ensure that it meets data user requirements. This shall be reviewed and approved by the Provider Accreditor.

7.1.8 The relative standard deviation of the Provider's verification method shall be established by a method validation study for each method and instrument.

7.1.9 For analytes in aqueous media, the assigned value of an analyte is verified if the mean of the Provider's verification analyses is within one-third of the Laboratory acceptance limits, to a maximum of 10%, as calculated per Section 10.2, of either:

a) The assigned value, if an unbiased verification method is used; or

b) The expected mean value for the analyte, if a biased method is used.

7.1.10 For analytes contained on or in sampling media, the assigned value of an analyte is verified if the mean of the Provider's verification analyses is within one-half of the Laboratory acceptance limits, as calculated per Section 10.2, of either:

a) The assigned value, if an unbiased verification method is used; or

b) The expected mean value for the analyte, if a biased method is used.

7.1.11 The standard deviation of the verification analyses shall be less than one standard deviation, as calculated for the participant Laboratories.

7.1.12 Any manufacturing lot that fails to meet the requirements of this Section shall not be used as an audit sample.

7.2 Homogeneity Testing

7.2.1 Providers shall analytically verify that all analytes in all manufacturing lots of audit samples within a packaging event are sufficiently homogenous prior to their use as an audit sample.

7.2.2 Homogeneity shall be verified using the procedure described in Appendix A or a procedure with an equivalent ability, as determined by the Provider Accreditor, to verify that differences between audit samples will not impact the evaluation of the stationary source test.

7.2.3 Homogeneity testing shall be performed on a representative selection of audit samples randomly selected from each final packaged audit sample batch prior to shipment to participant Laboratories.

7.2.4 Any manufacturing lot that fails to meet the requirements of this Section shall not be used as an audit sample.

7.3 Stability Testing

7.3.1 Providers shall verify the expiration date of the audit sample manufacturing lot and shall verify that all analytes in all audit samples remained stable.

7.3.1.1 Providers shall conduct stability testing of each lot of audit sample material or have data showing, to the satisfaction of the Provider Accreditor, that the sample was stable during the time period of use in the SSAS Program

7.3.2 Where appropriate, Providers shall retain samples of audit samples of the original audit sample manufacturing lot for use in confirmation of the lot assigned values and subsequent analytical verification.

7.3.3 The Provider shall use a stability verification procedure approved by the Provider Accreditor.

NOTE: Appendix A includes a suitable procedure for ensuring audit sample stability.

7.3.4 Audit samples or analytes that fail to meet the criteria of this Section shall be invalidated, and all sample recipients notified with a detailed discussion report.

7.4 Verification, Homogeneity, and Stability Testing Reporting

7.4.1 Upon request, and only after the Provider has released their evaluation of the audit sample analyses results, the Provider shall release, to a designated Participant, the results of the Provider's assigned value verification, homogeneity, and stability testing for any audit sample/analyte for which the Participant has reported or received data.

7.4.2 Upon request, and only after the Provider has released their evaluation of the audit sample analyses results, the Provider shall release to the PT Board the results of the Provider's assigned value verification, homogeneity, and stability testing for any audit sample/analyte.

7.4.3 To protect the blind nature of the audit sample, the Provider shall ensure the manufacturing lot number does not appear on any labels or documentation they provide to Participants and the PT Board, for assigned value verification, homogeneity, or stability testing.

7.5 Providers shall label each audit sample with a unique identifier.

8.0 Ordering and Reporting Instructions

8.1 The Provider shall receive an audit sample order from a Facility. The Provider shall contact the appropriate Regulatory Agency to request any specific requirements (e.g., changes to the audit sample concentration and/or shipment address) prior to shipment of the audit sample. The Provider may ship the audit sample if response is not received from the Regulatory Agency within fifteen (15) calendar days of the email of such request.

a) The Provider shall ensure that the audit sample is sealed such that opening or tampering will be apparent.

b) The Provider shall ship the audit sample to the Facility, unless the Regulatory Agency requests that it be shipped, instead, to the Regulatory Agency.

c) If the Facility cancels or modifies an audit sample order at any time, the Provider shall notify the Regulatory Agency of such cancellation or modification, within two (2) business days of the receipt of such notice.

8.2 The Provider shall provide instructions with each audit sample shipment, describing:

a) How to handle, store, dilute or otherwise prepare the audit sample;

b) How to report the data. The following attestation statement must be signed and submitted with the data;

"By affixing your signature below, you attest that the audit sample analyses results have met the following criteria:

1) You have no prior knowledge of the concentration of target analyte(s) in the audit sample. No additional information was solicited or received concerning the assigned values or acceptance ranges for the audit sample.

2) The audit sample(s) you are reporting was/were analyzed in the same laboratory under the same calibration, utilizing the same quality control standards, by the same analysts following audit sample instructions as the stationary source test samples."

3) The stationary source test results and the audit sample analyses results have been reported to the appropriate regulatory agency."

c) The expiration date or valid time frame of the audit sample being provided;

d) A warning that the TNI Standard requires audit samples to be analyzed at the same time as the stationary source test samples utilizing the same analysts, methods, and quality control procedures; and

e) A warning that the TNI Standard requires audit samples that test the field stack sampling process (e.g., for EPA Methods 18 and 25), to be collected before, during, or after the collection of the field samples, or as directed by the Regulatory Agency, utilizing the same methods and quality control procedures.

8.3 The Provider shall not:

a) Provide inappropriate assistance to the Participants, nor encourage the non-routine analysis of audit samples;

b) Suggest or direct that Laboratories use additional quality control samples or quality control samples designed specifically for a given audit sample, in conjunction with any audit sample;

c) Provide excessive volume of any audit sample that may encourage non-routine analyses;

NOTE: The Provider Accreditor, in consultation with the PT Board, will determine what constitutes excessive volume based on method requirements and common Provider practices within the industry.

d) Provide actual concentration ranges of audit sample to the Facility; and

e) Ship an audit sample past its established expiration date.

9.0 SYSTEM FOR REPORTING BY FACILITIES

The Provider shall:

a) Have procedures and systems in place to ensure the accurate, timely, and secure transmission of audit sample data from Facilities to the Provider;

b) Have a reporting mechanism that ensures that the results received by the Provider are consistent with those submitted by the Facilities;

c) Ensure that results reported by Facilities are not delayed or lost due to the Provider's reporting mechanism;

d) Ensure that Facility data are kept secure and that they are not subject to unauthorized dissemination either during or after data reporting to the Provider; and

e) Evaluate only the analytes of interest for each audit sample, as reported by the Facility.

10.0 AUDIT SAMPLE DATA ANALYSIS

10.1 Data Review

On a periodic basis to be determined by the Provider Accreditor, the Provider shall review the data reported by the Facilities for the following conditions:

10.1.1 Providers shall review all audit sample data for bimodal or multi-modal distributions and/or situations where results from a given method have disproportionately large failure rates or reporting anomalies.

10.1.2 If a multi-modal distribution is found related to an analytical method, this data shall be reported to the SSAS Expert Committee.

10.1.3 Providers shall review all audit sample data for disproportionately high or low failure rates compared to historical norms.

10.2 Acceptance Limit Determination

10.2.1 Providers shall calculate acceptance limits as defined in the SSAS Table.

10.3 Evaluation of Individual Participant Results

10.3.1 The Provider shall evaluate a result as "Acceptable" if it falls within the SSAS Table-defined acceptance limits as calculated in Section 10.2 above.

10.3.2 The Provider shall evaluate a result as "Not Acceptable" if it falls outside the acceptance limits as calculated in Section 10.2 above.

10.3.3 The Provider shall evaluate a result as "Not Acceptable" if it cannot be evaluated (e.g., alpha characters for a quantitative test or reported as a less than or greater than value).

10.3.4 If the Provider invalidates an analyte in the audit sample, all evaluations for data reported for that analyte shall be "No Evaluation" and a discussion of the situation leading to the invalidation shall be included in the final report to Participants.

11.0 GENERATION OF REPORTS

11.1 Schedule

11.1.1 The Provider shall submit the evaluation reports defined in Section 11.2 to the required parties no later than seven (7) calendar days after the reporting of the audit sample data.

11.1.2 The Provider shall submit evaluation reports to Facilities, Facility-requested Regulatory Agencies, other parties requested by the Facility, and to the SSAS Central Database within the same twenty-four (24) hour period.

NOTE: Evaluation reports may be submitted in hardcopy or electronic form.

11.2 Evaluation Report

11.2.1 The Provider shall include the following information in the evaluation report:

a) Provider name;

b) Provider accreditation number;

c) Participant Facility name;

d) Participant Facility physical address;

e) Name, title, and telephone number of Facility point of contact, as provided;

f) Laboratory's or Stationary Source Tester's name, address, and other contact information (e.g., telephone, email, and fax);

g) Date evaluation report was prepared;

h) Date evaluation report was amended, if applicable; and

i) Discussion including any pertinent information which addresses unusual details of the audit sample (e.g., need to change an assigned value or delete an analyte from evaluation).

11.2.2 The Provider shall include the following information for each audit sample/analyte in the final evaluation report:

a) SSAS Number;

b) Analyte name;

c) Analyte code defined in the SSAS Table;

d) Identification of any analytes not included in the Provider's accreditation;

e) Assigned value;

f) Acceptance limits;

g) Laboratory value, as reported;

h) Method name or description, as reported;

i) Matrix description

j) Analysis dates, as reported by the participating Laboratory; and

k) Evaluation, per Section 10.3 above;

11.2.3 Each page of the final evaluation report shall include an indication of the length of the report, presented by either “Page X of Y” or the total number of pages with each page consecutively numbered.

APPENDIX A

Guidance Procedure for Testing the Homogeneity and Stability of Stationary Source Audit Samples

A.1 PRETEST CONSIDERATIONS FOR VERIFICATION, HOMOGENEITY, AND STABILITY TESTING

Volume 1, Module 1 and ISO 17025 both have requirements for the repeatability of test methods used to validate stationary source audit samples, hereafter referred to as "audit samples." In order to satisfy these requirements, repeatability shall be determined at two or more levels. These recommendations provide a more comprehensive description of what is needed to describe repeatability if the Provider wishes to use an abbreviated method for testing homogeneity and stability.

a) Obtain reliable estimates of repeatability for the concentration levels of interest.

   i. To estimate repeatability, prepare samples at two or more levels across the audit sample analyte concentration range to be tested. Analyze at least seven (7) replicates at each level and calculate the repeatability as the standard deviation of the seven (7) replicates. The repeatability samples shall be treated exactly like normal audit samples, including taking sub-samples from a larger portion, if necessary.

   ii. Compare the repeatability estimates, both as standard deviations (SD) and as relative (percent) SDs. If either pair of these sets of estimates is very similar, then it might be safe to assume that the repeatability is constant across the concentration range. If the SDs (or RSDs) are not similar, then repeatability should be estimated at all levels where audit samples are produced. To be consistent with PT Board requirements, SDs shall be less than .167C at every level (C = size of the acceptance interval for audit samples; e.g., 2SD, 3SD, or fixed).

b) Update the repeatability estimates at least once within every accreditation cycle.

c) For every analyte in every matrix, determine whether homogeneity can be assumed on the basis of justifiable technical considerations. There should be considerations for manufacture of audit samples, including steps that can assure homogeneity, or procedures to address the reasons for heterogeneity. If heterogeneity is a concern, then there should be some expectation for how it will appear, such as a random problem (contamination), or follow a trend (filling operation).

When there is a concern about repeatability, such as having a repeatability standard deviation (S_r) at or above 0.167C, or when there is a possibility of sub-sampling errors, the recommended (general) protocol (5*2 or 10*2) should be followed.

A.1.1 Abbreviated Protocol

If repeatability is known at all areas of interest and if technical expertise assures a strong expectation of homogeneity, then an analyte can be assumed to be homogeneous and the Provider can follow an abbreviated protocol for homogeneity and stability testing. There should be general agreement among Providers that these assumptions are valid and there should be some data to support the assumption (not necessarily for every analyte). The abbreviated protocol eliminates testing of duplicate samples and thereby reduces the required testing in half.

In the following protocol, modifications are given for analytes that are assumed to be homogeneous. These modifications for the abbreviated protocol are for single tests rather than replicates, and are noted by appearing in a different font [in brackets and in italics].

A.2 GUIDANCE PROCEDURE FOR HOMOGENEITY AND VERIFICATION CHECK

a) Homogeneity checks shall be conducted on all analytes in the audit sample.

b) Use audit samples that have been packaged for distribution.

c) Determine a number g of the samples that will be tested, where g ≥ 5. For analytes where there is a concern about heterogeneity (most analytes in soil samples, and some analytes in water), let g ≥ 10. *[g = 5 is sufficient for the abbreviated protocol.]*

d) Select the samples in the following way (this is called a "systematic" sampling technique, and is considered to be a random process, where every sample has the same probability of selection).

e) Determine the selection interval, G = N/g, rounded to the closest number. N is the total number of prepared samples.

f) Using a random number table or a random number generator, select a number between 1 and G (or 01 to G if G ≥ 10); call this number T.

g) Select a sample produced in sequence order T and then select samples produced in sequence order numbers T + G, T + 2G, T + 3G, etc.

h) Prepare two (2) test portions from each sample using techniques appropriate to the test material to minimize between-test-portion differences (if the size of the sample portion is too small for duplicate samples for all the analytes to be tested, then repeat this protocol to select a second set of samples). *[Prepare a single test portion from each sample.]*

i) Taking the 2g test portions in a random order, use an appropriate method to obtain a measurement result on each, completing the whole series of measurements under repeatability conditions.

j) Calculate the average of each sample x_t…, the general average x..., the repeatability standard deviation s_r, and between-samples standard deviation s_s, as shown in Section A.2.2 of this procedure. *[Record the 5 test results and treat them as if they are "averages" in the following steps. Calculate the standard deviation of the 5 results.]*

k) List the sample averages x_t... in order of selection 1…g. Check for a trend by either visual assessment or with a plot the averages of the replicates on each level on a plot of the averages (vertical) vs. selection order 1…g (horizontal).

l) If the list of sample averages or the plot shows any consistent change in results, then assess the importance of the drift relative to the audit sample scheme. Calculate the difference between the last and the first sample averages. Compare this difference against the criteria used in Section A.2.2. If the drift is larger than the criteria, then the drift is large enough to affect laboratory evaluations.

A.2.1 Guidance Procedure for Assessment Criteria for Homogeneity Check

Determine the expected acceptance limits for the analyte; this varies for different analytes. Use C to denote the acceptance interval (as in ±C). For example, C could be 2SD, 3SD, or a fixed percentage. If the visual plot suggests a trend in concentration over the production run, calculate

the difference between the largest and smallest concentrations (not necessarily the first and last samples), call this d_s. If no visual trend is apparent, then this does not need to be done.

Compare the between-samples standard deviation s_s and the difference d_s with C, as follows:

The samples may be considered to be adequately homogeneous if:

s_s	≤	*0.25C*	*and if*	*(1)*
d_s	≤	*0.25C*		*(2)*

If these criteria are not met, the Provider shall consider the following options:

a) Examine the sample preparation procedure to see if improvements are possible.

b) If the analyte is evaluated using standard deviations of actual participant results (as with use of z scores that are based on consensus results), then the heterogeneity of samples is included in the inter-laboratory standard deviation, and will be accounted for in the calculation of the performance statistic.

A.2.2 Guidance Procedure for Formulae for Homogeneity Check

The data from a homogeneity check are represented by:

$x_{t,k}$ *where* *(3)*

g is the number of samples
t represents the sample (t = 1 , 2 , ... , g)
k represents the test portion (replicate) (k = 1 , 2)

Define the sample averages as:

$x_{t,}$ $=(x_{t,1} + x_{t,2}) / 2.0$ *[use the sample results as " averages"]* *(4)*

and the between-test portion ranges as:

w_t $=/x_{t,1} - x_{t,2}/$ *[skip this step]* *(5)*

Calculate the general average:

$x = \sum x_{t,.} / g$ *(6)*

the standard deviation of sample averages:

$$s_x = \sqrt{\sum \left(x_{t,.} - x_{.,.}\right)^2 / \left(g - 1\right)} \quad (7)$$

and the repeatability standard deviation:

$$s_r = \sqrt{\sum w_t^2 / \left(2g\right)} \quad \textit{[use a suitable repeatability estimate } s_r\textit{]} \quad (8)$$

where the summations are over all samples (t = 1, 2, ... , g).

Finally, calculate the between-samples standard deviation as:

$$s_s = \sqrt{s_x^{\,2} - \left(s_r^{\,2}/2\right)} \quad \textit{[do not divide by two, or skip and let } s_s = s_x\textit{]} \qquad (9)$$

It is possible that the difference inside the square root will be negative. This can occur when there are no detectable differences between samples (s_s=0). When this occurs, assume s_s=0; and if the estimate of repeatability (s_r) is to be used elsewhere, it should be recalculated as the standard deviation of all the homogeneity results.

A.2.3 Guidance Procedure for a Stability Check

a) Conduct the stability tests after the initial reporting for a manufacturing lot, and periodically thereafter as defined by the Provider Accreditor, but prior to formal evaluation of Participant results. Samples shall have been stored under conditions similar as those required of participant Laboratories.

b) Use the same measurement method for the stability check as for the homogeneity check, under conditions as similar as possible to those of the homogeneity check.

c) Select a number g of the samples at random, where g ≥ 3 (g = 3 should be sufficient for most situations).

d) Prepare two test portions from each sample using the same techniques as for the homogeneity check. *[Prepare single test portions.]*

e) Taking the 2g test portions in a random order, obtain a measurement result $y_{t,k}$ on each, completing the whole series of measurements under repeatability conditions.

f) Calculate the general average y of the measurements obtained in the stability test.

A.2.4 Guidance Procedure for Assessment Criteria for Stability Check

Compare the general average of the measurements obtained in the homogeneity check (x) with the general average of the results obtained in the stability check (y). The samples may be considered to be adequately stable if:

$$\left|x_{...} - y_{...}\right| \leq 0.2C \qquad (10)$$

If this criterion is not met, examine the sample preparation and storage procedures to see if improvements are possible.

Some measurands are inherently unstable, but still may be used in audit sample testing. The effect of instability may be predictable and therefore subject to mathematical correction, or Participants can be instructed to conduct the measurements at a specified time.