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GENERAL REQUIREMENTS FOR STATIONARY SOURCE AUDIT SAMPLE PROVIDERS

TNI Working Draft Standard

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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 Volume specifies the requirements for providers of stationary source audit samples (SSAS) used for the confirmation of stationary source testing results.

1.2 Scope

The TNI SSAS program includes the following elements:

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

b) The production and supply of SSASs 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 SSAS data that are technically defensible on the basis of the type and quality of the SSASs provided; and

d) The preparation of SSASs that pose equivalent difficulty and challenge, regardless of the manner in which the SSASs are designed and manufactured by the SSAS providers.

1.3 Applicability

This Volume does not address issues of laboratory accreditation.

2.0 REFERENCES

2.1 Kafadar, Karen. “A Biweight Approach to the One-Sample Problem,” *Journal of the American Statistical Association*, Vol. 77, No. 378, June, 1982, pp. 416-424.

2.2 *ISO 9001 Quality Management Systems – Requirements.*

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

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

2.5 *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, the relevant terms and definitions are conformant with *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 SSAS material.

3.2 **Assigned Value:** Value attributed to a particular quantity and accepted, sometimes by convention, as having an uncertainty appropriate for a given purpose. See Section 6.4 for further discussion of assigned values.

3.3 **Facility:** The responsible party for the stationary source test or their authorized representative.

3.4 **Field of Proficiency Testing (FoPT) Table:** Table in which the analytes and acceptance limits for SSAS materials are defined.

3.5 **Stationary Source Audit Sample (SSAS):** 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 SSAS is analyzed, or collected and analyzed, as part of the batch of field test samples using the same personnel, procedures, and materials.

3.6 **Stationary Source Audit Sample Program (SSAS Program):** The procedures 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. The program establishes requirements for facilities, stationary source testers, and laboratories for field collection (if applicable), analysis, reporting of measurement results, and reporting of SSAS analyses results.

3.7 **Stationary Source Audit Sample Provider (SSAS Provider):** A person or organization accredited by the TNI-approved Stationary Source Audit Sample Provider Accreditor (SSAS PA).

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

3.10 **TNI PT Board (PT Board):** A board consisting of TNI members or affiliates, appointed by the TNI Board of Directors (BOD), 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 SSAS PROVIDER ACCREDITATION

4.1 The SSAS provider shall be accredited by a TNI-approved SSAS PA for every SSAS (as listed in the TNI FoPT table) they will offer in their SSAS program.

4.2 In order to receive and maintain accreditation for any analyte per matrix in any FoPT, the SSAS provider shall demonstrate compliance with all requirements of this Standard during on-site assessments and ongoing oversight conducted by the SSAS PA per Volume 4 of this Standard.

4.3 SSAS providers shall be subject to biennial onsite assessments conducted by their chosen TNI-approved SSAS PA. They may also be subject to unannounced assessments for cause.

4.4 SSAS providers shall submit data from each of their SSAS manufactured lots to the SSAS PA 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 SSAS PA.

4.4.2 The provider shall not identify any participant to the SSAS PA without the expressed written consent of the participant.

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

4.6 In conflicts with the SSAS PA, SSAS providers shall follow the SSAS PA's appeals process.

4.7 Unresolved conflicts with the SSAS PA shall be submitted to the TNI PT Board.

5.0 MANAGEMENT REQUIREMENTS

5.1 Quality System Requirements

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

5.1.2 The SSAS provider's manufacturing system shall meet the requirements of ISO Guide 34 (Quality System Guidelines for the Production of Reference Materials).

5.1.3 The design and operation of the SSAS provider's SSAS program shall meet the relevant requirements of ILAC G-13 (Guidelines for the Requirements for the Competence of Providers of Proficiency Testing Schemes).

5.1.4 The testing facilities used to support the verification, homogeneity, and stability testing required in this Standard shall meet the requirements of ISO 17025 (General Requirements for the Competency of Testing and Calibration Laboratories).

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

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

5.2 Provider Conflict of Interest and Confidentiality

SSAS providers seeking to obtain or maintain accreditation shall:

a) Document and certify to the satisfaction of the SSAS PA 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 SSAS provider's obligation to report personal and organizational conflicts of interest to the SSAS PA;

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 SSAS PA 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 SSAS PA 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 SSAS and the SSAS program is not compromised;

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

g) Not disclose specific laboratory results or evaluations to any parties other than as specified in Section 11.1.2 without a written release from the laboratory.

NOTE: SSAS 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 SSAS 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 SSAS for which they wish to obtain or maintain accreditation.

5.3.2 SSAS providers shall employ sufficient technical and support staff to design, produce, analyze, distribute, and provide data evaluation and reporting functions for every SSAS 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 SSAS provider for any particular manufacturing lot. For the purposes of this Standard, “direct control” means that these functions are performed in the SSAS provider’s facilities by the SSAS provider’s staff or are subcontracted by means of a written agreement with defined SSAS provider supervision to ensure that all requirements of this Standard are met.

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

5.4 Complaints Handling

5.4.1 SSAS providers shall have written procedures for handling both written and verbal complaints from SSAS participants and regulatory agencies who receive SSAS reports.

5.4.2 SSAS providers shall record all complaints received concerning their SSAS program, including any remedial or corrective actions taken. This record shall be provided to the SSAS PA upon request.

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

5.5 Notification of Sample Integrity

If any SSAS or analyte used in the SSAS program is found to not meet any of the requirements of this Standard, the SSAS provider shall notify all affected participants, their designated regulatory agencies, and the provider’s SSAS PA within seven (7) calendar days of the discovery of the non-conformance.

6.0 SSA SAMPLE DESIGN AND MANUFACTURE

6.1 Design Review

SSAS providers shall demonstrate to the satisfaction of the SSAS PA that their SSAS design and manufacturing processes:

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

b) Provide equivalent challenge to all participants, and

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

6.2 SSAS Matrices

6.2.1 The matrices of all SSAS shall, to the extent possible, resemble the matrices which participants routinely analyze.

6.3 SSAS Analytes

6.3.1 SSAS providers shall prepare SSAS that are compliant with the criteria defined by the PT Board and published in the TNI FoPT Tables on the TNI website.

6.3.2 When the PT Board makes changes to the SSAS design criteria, SSAS providers shall comply with the revised requirements per the PT Board's implementation schedule.

6.3.3 The SSAS provider shall spike all analytes of interest into the SSAS.

6.3.4 The SSAS provider shall produce SSAS that are conformant with the method being tested.

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

6.4 SSAS Concentration Ranges

6.4.1 SSAS providers shall supply SSAS that reflect the concentration ranges needed by the facilities.

6.4.1.1 SSAS providers may make modifications to selected assigned values based on technical (i.e., solubility, compatibility, interference) issues.

6.4.1.2 Any modifications to selected assigned values shall be documented.

6.4.2 Assigned values for non-microbiological, aqueous, solid, and chemical matrix 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, and

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

6.4.3 Assigned values for gas analytes shall:

a) Be equal to the made-to values of the analytes based on gravimetric or volumetric measurements of a starting material of known concentration, or shall be set to the mean of

the analytically determined measured value, and

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

7.0 SSAS TESTING

7.1 Verification of Assigned Value

7.1.1 SSAS providers shall analytically verify the assigned value of all analytes in all manufacturing lots of SSAS prior to use.

7.1.2 SSAS 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 (NIST), United States Pharmacopeia (USP), etc.) 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 SSAS 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 SSAS being verified.

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

7.1.8 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.

7.1.9 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.

7.1.10 For analytes contained in cylinders or canisters, 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.

7.1.13 Any SSAS that fails to meet the requirements of this Section shall not be used as an SSAS.

7.2 Homogeneity Testing

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

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 SSAS PA, to verify that differences between SSAS will not impact the evaluation of the stationary source test.

7.2.3 Homogeneity testing shall be performed on a representative selection of SSAS randomly selected from each final packaged SSAS batch prior to shipment to participant laboratories.

7.2.4 SSAS that fail to meet the requirements of this Section shall not be used as an SSAS.

7.3 Stability Testing

7.3.1 SSAS providers shall verify the expiration date of the SSAS manufacturing lot and shall verify that all analytes in all SSASs remained stable. The SSAS provider may extend the expiration date by demonstrating the ongoing stability of the SSAS manufacturing lot.

7.3.2 SSAS providers shall retain SSAS of the original SSAS manufacturing lot for use in confirmation of the lot assigned values and subsequent analytical verification.

7.3.3 SSAS stability assessments shall be based on analytical data comparing the mean of a series of random SSAS analytically tested before release of a manufacturing lot to the mean of a series of random SSAS analytically tested after the expiration date. If the difference between the two means cannot be shown to affect an evaluation, then the analyte can be considered stable for the period of SSAS availability.

7.3.4 The SSAS provider shall use a stability verification procedure approved by the SSAS PA.

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

7.3.6 SSASs 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 SSAS provider has released their evaluation of the SSAS 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 SSAS/analyte for which the participant has reported data.

7.4.2 Upon request, and only after the provider has released their evaluation of the SSAS analyses results, the provider shall release, to the regulatory agency, the results of the provider's assigned value verification, homogeneity, and stability testing for any SSAS/analyte for which results were submitted to that regulatory agency.

7.4.3 Upon request, and only after the provider has released their evaluation of the SSAS 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 SSAS/analyte.

To protect the blind nature of the SSAS, the SSAS provider shall ensure the manufacturing lot number does not appear on any labels or documentation they provide to participants, regulatory agencies, and the PT Board, for assigned value verification, homogeneity, or stability testing.

7.4.4 SSAS providers shall supply to their SSAS PA the results of the SSAS provider's assigned value verification, homogeneity, and stability testing for all SSAS/analytes.

7.4.5 The SSAS provider shall follow the format and schedule for submittal of these data as provided by the SSAS PA.

8.0 PROVISION OF SSAS

8.1 Ordering and Reporting Instructions

8.1.1 Ordering of SSAS for a stationary source test shall be performed by the facility in conjunction with the regulatory agency that is overseeing the stationary source test. After placing the order with an SSAS provider, the facility shall notify their regulatory agency which SSAS providers they are utilizing. The SSAS provider must receive contact information for the regulatory agency from the ordering party before SSAS shipment. The SSAS provider shall contact the regulatory agency within two (2) business days after the order, to request any specific requirements prior to shipment of the SSAS. The SSAS provider may ship the SSAS if the regulatory agency does not notify the SSAS provider of any specific requirements within fifteen (15) calendar days of the regulatory agency's receipt of the request. If the SSAS order is cancelled or modified by the facility, the SSAS provider shall notify the regulatory agency within two (2) business days.

a) The SSAS provider shall ensure that the SSAS is sealed such that opening or tampering will be apparent.

b) The SSAS provider shall ship the SSAS to the facility, unless the regulatory agency requests that it be shipped, instead, to the regulatory agency.

8.1.2 The SSAS provider shall provide instructions with each SSAS shipment, describing:

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

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 Stationary Source Audit Sample (SSAS) analyses results have met the following criteria:

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

2) The SSAS(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 SSAS instructions as the stationary source test samples."

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

c) The initial expiration date of the SSAS being provided;

d) A warning that the TNI Standard requires SSAS 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 stationary source test samples, which test the stack sampling process, to be analyzed between test runs or as directed by the regulatory agency, utilizing the same methods and quality control procedures.

8.1.3 The SSAS provider shall not:

a) Provide inappropriate assistance to the participant laboratories, nor encourage the non-routine analysis of SSAS;

b) Suggest or direct laboratories to use additional quality control samples or quality control samples designed specifically for a given SSAS, in conjunction with any SSAS;

c) Provide excessive volume of any SSAS that may encourage multiple, non-routine analyses.

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

d) Provide concentration ranges of SSAS to the facility.

9.0 SYSTEM FOR REPORTING BY FACILITIES

The SSAS provider shall:

a) Have procedures and systems in place to ensure the accurate, timely, and secure transmission of SSAS data from facilities to the SSAS provider;

b) Have a reporting mechanism that ensures that the results received by the SSAS provider are consistent with those submitted by the facilities;

c) Ensure that results reported by facilities are not delayed or lost due to the SSAS 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 the data have been reported to the SSAS provider;

e) Evaluate only the analytes of interest for each SSAS, as reported by the facility.

10.0 SSAS DATA ANALYSIS

10.1 Data Review

On a quarterly basis, the SSAS provider shall review the data reported by the facilities for the following conditions:

10.1.1 SSAS providers shall review all SSAS 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 analytical method and acceptance criteria are calculated using robust statistical analysis of participant data, results shall be evaluated on a method-specific basis.

10.1.3 SSAS providers shall review all SSAS data for disproportionately high or low failure rates compared to historical norms.

10.2 Acceptance Limit Determination

10.2.1 SSAS providers shall calculate acceptance limits per the requirements defined in the TNI FoPT tables. For acceptance limits not defined in the TNI FoPT tables, SSAS providers shall use the procedures in Sections 10.2.5, 10.2.6, or 10.2.7 to calculate acceptance limits.

10.2.2 Analyte- or data-specific evaluation criteria defined in the TNI FoPT tables shall supersede the criteria in this Section.

10.2.3 Acceptance limits shall be represented following the same significant figure rules as defined for assigned values in Section 6.4.

10.2.4 For acceptance limits calculated using only the SSAS provider's assigned value (i.e., a fixed percentage limit around the assigned value, regression equation using the assigned value to determine an estimated mean and estimated standard deviation, etc.), the SSAS provider shall use their assigned value and calculate the acceptance limits defined in the TNI FoPT tables.

10.2.5 For acceptance limits calculated using the actual data mean, the SSAS provider shall use the mean as calculated by the following procedures:

a) For sample sizes of twenty (20) or more values: the biweight mean (per Section 2.1) using fifteen (15) iterations with c=4 and c_o=6;

b) For sample sizes of seven (7) to twenty (20) values: the arithmetic mean after outlier testing using the T test (see ASTM E178) or other SSAS PA-accepted outlier testing procedure. No more than 20% of the values in any set shall be treated as outliers;

c) Sample sizes of less than seven (7) values shall only be evaluated using a statistical procedure approved by the SSAS PA.

10.2.6 For acceptance limits calculated using the actual data standard deviation, the SSAS provider shall use the standard deviation as calculated by the following procedures:

a) For sample sizes of twenty (20) or more values: the biweight standard deviation (per Section 2.1) using fifteen (15) iterations with c=4 and c_o=6;

b) For sample sizes of seven (7) to twenty (20) values: the standard deviation after outlier testing with the T test (see ASTM E178) or other SSAS PA-accepted outlier testing procedure. No more than 20% of the values in any set may be treated as outliers;

c) Sample sizes of less than seven (7) values shall only be evaluated using a statistical procedure approved by the SSAS PA.

10.2.7 For acceptance limits calculated using the actual data median, the SSAS provider shall use the median calculated from all properly reported data points, as defined by the FoPT tables, in the data set.

10.3 Evaluation of Individual Participant Results

10.3.1 The SSAS provider shall evaluate a result as "Acceptable" if it falls within in the TNI FoPT table - defined acceptance limits as calculated in Section 10.2.

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

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

10.3.4 If the SSAS provider invalidates an analyte in the SSAS, 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 and regulatory agencies.

11.0 GENERATION OF REPORTS

11.1 Schedule

11.1.1 The SSAS provider shall submit the reports defined in Sections 11.2 and 11.3 to the required parties no later than fifteen (15) calendar days after the reporting of the SSAS data.

11.1.2 The SSAS provider shall submit reports to facilities, facility-requested regulatory agencies, and upon request, other relevant parties within the same twenty-four (24) hour period.

NOTE: Evaluation reports may be submitted in hardcopy or electronic form. Rapid response of SSAS reports may be pre-arranged with SSAS providers.

11.2 Evaluation Report

11.2.1 The SSAS provider shall include the following information in the evaluation report:

a) SSAS provider name;

b) SSAS provider SSAS PA 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) Participant laboratory's primary accreditation body ID, as provided;

g) Date evaluation report was prepared;

h) Date evaluation report was amended, if applicable;

i) Discussion including any pertinent information which addresses unusual details of the SSAS (for example, description of "not acceptable" test results, need to change an assigned value or delete an analyte from evaluation).

11.2.2 The SSAS provider shall include the following information for each SSAS/analyte in the final evaluation report:

a) SSAS number;

b) Analyte name;

c) Analyte code defined in the TNI FoPT Tables;

d) Identification of any analytes not included in the SSAS provider's SSAS PA accreditation;

e) Assigned value;

f) Acceptance limits;

g) Laboratory value, as reported;

h) Method name or description, as reported;

i) Matrix description, as reported;

i) Analysis dates, as reported by the participating laboratory;

j) Evaluation, per Section 10.3;

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.

11.3 Failure Rate Report

11.3.1 Upon request by either a participant laboratory or a regulatory agency, the SSAS provider shall make available a report listing the total number of participating laboratories and the number of laboratories scoring "Not Acceptable" for those analytes reported by the laboratory.

11.3.2 The SSAS provider shall not disclose specific laboratory results or evaluations to any parties other than as specified in Section 11.1.2, without a written release from the laboratory.

APPENDIX A

Guidance Procedure for Testing the Homogeneity and Stability of SSAS

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

Volume 3 and ISO 17025 both have requirements for the repeatability of test methods used to validate SSAS. 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 SSAS 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 SSAS 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 SSAS, 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 SSAS are produced. To be consistent with TNI PT Board requirements, SDs shall be less than .167C at every level (C = size of the acceptance interval for SSAS; 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 SSAS, 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 SSAS provider can follow an abbreviated protocol for homogeneity and stability testing. There should be general agreement among SSAS 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 SSAS.

b) Use SSAS 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 SSAS 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 SSAS 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 SSAS PA, 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 SSAS 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.