

**SUMMARY OF THE
TNI LABORATORY PROFICIENCY TESTING EXPERT COMMITTEE MEETING
SEPTEMBER 6, 2013**

The Committee met by teleconference on Friday, September 6, 2013, at 11:00 am EST. Chair Shawn Kassner led the meeting.

1 – Roll call

Fred Anderson, Advanced Analytical Solutions (Other)	Present
Stephen Arpie, Absolute Standards (Other)	Present
Kareen Baker, Veolia Water N. American (Other)	Absent
Yumi Creason, PA DEP (AB)	Absent
Rachel Ellis, NJ DEP (AB)	Present
Scott Hoatson, Oregon DEQ (AB)	Present
Shawn Kassner, Phenova (Chair; Other)	Present
Roger Kenton, Eastman Chemical Co. (Lab)	Present
Stacie Metzler, Hampton Roads San. Distr. (Lab)	Present
Mitzi Miller, Dade Moeller Assocs. (Other)	Absent
Judy Morgan, Env. Science Corp. (Lab)	Present
Virgene Mulligan, Amrad (Lab)	Present
Joe Pardue, P2S (Other)	Present
Jim Todaro, Alpha Analytical (Lab)	Absent
Lisa Touet, MA DEP (AB)	Absent
Ken Jackson, Program Administrator	Present

Associate Committee Members present: Nicole Cairns, NYSDOH; Audrey Cornell, ERA; Brian Stringer, ERA.

2 – Previous Minutes

Steve had recommended some changes to the August 23 draft minutes. Shawn said he would make the changes and re-distribute the minutes for consideration.

3 – Preparation Method Data

Shawn had not yet contacted EPA people about gathering preparation method data. He suggested, however, the data would be more important to TNI accreditation than to EPA. On the previous call, it had been suggested contacting the NY accreditation program, who may have preparation method data available. Since Nicole was on the call, she was able to address this. She confirmed such data were available for solid waste, where big differences in performance are seen by preparation method. Nicole named Dan Dickinson as the contact person.

4 – Subcommittees

Virgene was already working with the Radiochemistry Expert Committee on formation of a radiochemistry subcommittee. Joe had contacted David Sill of the DOE RESL laboratory, who had agreed to serve on the subcommittee. Virgene said she would call him.

Judy confirmed she had staff who could work on a microbiology/protozoan subcommittee. Brian reported he had spoken to Mike Blades who had agreed to help. Shawn had found an AB from LA prepared to work on a protozoan subcommittee, and Rachel said Deb Waller from NJ was prepared to do it. A laboratory person was also needed, and Judy said her person was both a microbiology and protozoan person. Fred said he would serve as the Expert Committee member of the subcommittee. Shawn would look for another PT person for microbiology.

There was no progress reported to date on SIR or WET subcommittees.

5 – Volume 3

Shawn had previously circulated the attached draft version of V3, in which he had highlighted those sections he considered to be TNI-specific requirements. The committee began working through this document, to decide what should be in the TNI standard and what can be omitted because it is already in ISO 17043 or Guide 34.

1.0 INTRODUCTION, SCOPE AND APPLICABILITY

Shawn suggested this section needed no changes.

2.0 REFERENCES

It was decided to leave this section alone until the committee had worked through the whole volume.

3.0 TERMS AND DEFINITIONS

It was similarly decided to leave this section until later.

4.0 PT PROVIDER ACCREDITATION

5.0 MANAGEMENT REQUIREMENTS

These sections were considered together, because it might be necessary to move some items between the sections.

Section 4.0 are all TNI requirements, basically unchanged for many years. It was agreed no changes should be made. Scott asked if this is where it needs to be added that they have to be accredited to ISO 17043. Shawn said Section 5 Quality System Requirements was also a possibility for this.

Section 5.1.1 currently relates to ISO 9001, and since this does not have any direct relevance to PT, it was suggested to remove it. Any PT provider accredited to ISO 17043 would already meet the ISO 9001 requirements. Sections 5.1.2 and 5.1.3 needed re-wording to say PT providers shall be accredited to ISO 17043 and Guide 34. Scott suggested saying the provider shall meet the requirements of and be accredited to ISO 17043. Nicole said it should go in 4.2.

Shawn drafted a new 4.2, saying that PT Providers shall be accredited to ISO 17043. In 4.3, he drafted wording saying in order to receive and maintain accreditation for an analyte and any FoPT, the PT provider shall demonstrate compliance with all requirements of ISO 17043 and this standard during on-site audits overseen by the PTPA per V4 of the standard.

Referring to the current Section 5.1.2, Shawn said he would review all providers' accreditation to Guide 34 and then decide if it should be added that they have to be so accredited. Meanwhile, he suggested putting it in Section 4, as a placeholder, to say they have to meet the requirements of ISO Guide 34. Nicole thought NY was not accredited to Guide 34, since they are not a reference material provider, though they are assessed to it

Section 5.1.4 requires the providers' testing facilities to be accredited to ISO 17025. Scott suggested leaving that where it is. This has to remain because ISO 17043 does not require the providers' testing facilities to be accredited to ISO 17025.

Sections 5.1.5 and 5.1.6 are not in ISO 17043, so they should remain. Shawn questioned if they should be listed under management requirements or accreditation requirements, and after discussion it was decided to leave them where they are. It was clarified that Section 5.1.5 was inserted at the request of the PTPAs to allow them to accredit to ISO 17025 for designated FoPTs, even if another PTPA has already accredited the provider to ISO 17025 for other FoPTs. It was decided to move 5.1.5 into Section 4. A note was added that this also needs to be in V4.

Shawn said most of **Section 5.2, Provider Conflict of Interest and Confidentiality**, is in ISO 17043. An exception is the reporting requirements to the PTPA, and he highlighted those requirements in 5.2 b, d, and f. Scott added that 5.2 g (with the note) is also not covered in ISO 17043 and needs to be in the TNI standard. Shawn said he would consolidate these parts into a single standard clause.

Shawn reported all of **Section 5.3, Provider Facilities and Personnel**, were in ISO 17043. Scott recommended a statement should go in its place to say "as required in ISO 17043".

In **Section 5.4, Complaints Handling**, 5.4.2 and 5.4.3 are not covered in ISO 17043. Shawn said he would consolidate the two sections. Nicole suggested, instead of stating "as required in ISO 17043" in 5.4.1 (and elsewhere), put a blanket statement in the scope that ISO 17043 is followed throughout. Then it would only be necessary to have the TNI requirements throughout the standard. The committee agreed to follow this recommendation. Steve suggested the additional TNI statement in 5.4.2 ("This record shall be provided to the PTPA upon request") was not needed, since PTPAs examine this during their audits.

6.0 PT SAMPLE DESIGN AND MANUFACTURE

In Section 6.1, Steve questioned the statement "PT providers shall demonstrate to the satisfaction of the PTPA that their PT sample designs and manufacturing processes:" This is a subjective term. It is required to satisfy the requirements of G34 and ISO 17043 that auditors can then run a check list against. Shawn said he would re-word this.

At this point, Shawn suggested stopping, and volunteered to redraft just the TNI-specific requirements in the same format as ISO 17043, and distribute it before the next meeting. The committee agreed with this approach.

Adjournment

The meeting was adjourned at 12:20 pm EDT.

VOLUME 3

GENERAL REQUIREMENTS FOR ENVIRONMENTAL
PROFICIENCY TEST PROVIDERS

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VOLUME 3

GENERAL REQUIREMENTS FOR ENVIRONMENTAL PROFICIENCY TEST PROVIDERS

1.0 INTRODUCTION, SCOPE AND APPLICABILITY

1.1 Introduction

This Volume specifies the requirements for proficiency testing (PT) providers conducting PT studies for the evaluation of environmental testing laboratories.

Note from Kirstin: I think the introduction needs to be expanded to include provide PT samples or

1.2 Scope

The PT program includes the following elements:

- a) The production and supply of PT samples that challenge the critical components of each analytical procedure, from initial sample preparation to final data analysis;
- ~~b) The production and supply of PT samples that are as similar to real-world samples as are reasonably possible and representative of materials analyzed for environmental regulatory programs, agencies and communities^[71];~~
- ~~b~~e) The yielding of PT data that are technically defensible on the basis of the type and quality of the PT samples provided; and
- ~~d~~c) The preparation of PT samples which pose equivalent difficulty and challenge regardless of the manner in which the PT samples are designed and manufactured by the PT providers.

The scope has been changed in V1, V2 — we should drop bullet B-x

1.3 Applicability

This Volume does not purport to address issues of laboratory accreditation. The laboratory accreditation process is defined in Volumes 1 and 2 of this Standard.

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.*

2.6 *ASTME178 Standard Practice ~~for Dealing~~for Dealing With Outlying Observations*

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 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.
- 3.2 Acceptance Limits:** The range of values that constitute acceptable performance for a laboratory participating in PT study.
- 3.3 Field of Proficiency Testing (FoPT):** Matrix, technology/method, analyte combinations for which the composition, spike concentration ranges and acceptance criteria have been established by the Proficiency Testing Program Executive Committee.
- 3.4 Primary Accreditation Body (Primary AB):** The accreditation body responsible for assessing a laboratory's total quality system, on-site assessment, and PT performance tracking for fields of accreditation.
- 3.5 Proficiency Testing (PT):** A means to evaluate a laboratory's performance, under controlled conditions, relative to a given set of criteria, through analysis of unknown samples provided by an external source.
- 3.6 Proficiency Testing Program (PT Program):** The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of results and the collective demographics and results summary of all participating laboratories.
- 3.7 Proficiency Testing Provider (PT Provider):** A person or organization accredited by the TNI-approved Proficiency Testing Provider Accreditor to operate a TNI-compliant PT program.
- 3.8 Proficiency Testing Provider Accreditor (PTPA):** An organization that is approved by TNI to accredit and monitor the performance of proficiency testing providers.
- 3.9 Proficiency Testing Reporting Limit (PTRL):** The value that corresponds to the lowest acceptable result that could be obtained from the lowest spike level for each analyte in a PT sample. PTRLs are established and published by the TNI PT Board.
- 3.10 Proficiency Testing Sample (PT Sample):** A sample, the composition of which is unknown to the laboratory and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.
- 3.11 Proficiency Testing Study (PT Study):** A single complete sequence of circulation of proficiency testing samples to all participants in a proficiency test program.
- 3.12 PT Study Closing Date:** The latest calendar date for which analytical results for a PT sample shall be received by the PT provider from the laboratory.
- 3.13 PT Study Opening Date:** The calendar date that a PT sample is first made available to any laboratory by a PT provider.
- 3.14 Study:** This term refers to a PT Study or Supplemental PT Study.

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- 3.15 Supplemental Proficiency Testing Study (Supplemental PT Study):** A PT sample that may be from a lot previously released by a PT Provider that meets the requirements for supplemental PT samples given in this Volume, but that does not have a pre-determined opening date and closing date.
- 3.16 Supplemental PT Study Closing Date:** The calendar date for which analytical results of a PT sample are received by the PT provider.
- 3.17 Supplemental PT Study Opening Date:** The calendar date that a PT sample is shipped from the PT provider to a laboratory.

4.0 PT PROVIDER ACCREDITATION

- 4.1** The PT provider shall be accredited by a TNI-approved PTPA for every TNI FoPT which they will offer in their PT programs.
- 4.2** In order to receive and maintain accreditation for any analyte in any FoPT, the PT provider shall demonstrate compliance with all requirements of this Standard during onsite audits and ongoing oversight conducted by the PTPA per Volume 4 of this Standard.
- 4.3** PT providers shall be subject to biennial onsite audits conducted by their chosen TNI-approved PTPA. They may also be subject to unannounced audits for cause.
- 4.4** PT providers shall submit data from each of their PTPA-accredited PT studies to the PTPA 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 PTPA.
- 4.4.2** The provider shall not identify any participant laboratory to the PTPA without the expressed written consent of the laboratory.
- 4.5** Upon request by the PTPA, the PT Provider shall supply, at no charge, PT samples as specified by the PTPA, which are included in the PT Provider's scope of accreditation, to the PTPA for submission to a referee laboratory.
- 4.6** In conflicts with the PTPA, PT providers shall follow the PTPA's appeals process.
- 4.7** Unresolved conflicts with the PTPA shall be submitted to the PTP Executive Committee.

5.0 MANAGEMENT REQUIREMENTS

5.1 Quality System Requirements

- 5.1.1** The PT provider's quality management system shall meet the requirements of ISO 9001 for the design, production, testing, and distribution of PT samples and the evaluation of PT results.
- 5.1.2** The PT 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 PT provider's proficiency testing program shall meet requirements of ISO 17043 (General Requirements for Proficiency Testing).

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

5.1.6 Providers shall maintain all records related to each PT study for a minimum of five (5) years after the close of the PT study.

5.2 Provider Conflict of Interest and Confidentiality

PT providers seeking to obtain or maintain accreditation shall:

a) document and certify to the satisfaction of the PTPA that they do not have any conflict of interest with any laboratory that may participate in their PT programs;

NOTE: Such a conflict of interest could take the form of a financial interest or sharing of personnel, facilities or equipment with any laboratory that may participate in the provider's PT studies.

b) inform all internal and contract personnel who perform work on PT studies of the PT provider's obligation to report personal and organizational conflicts of interest to the PTPA;

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 PT Programs;

d) immediately make a full disclosure to the PTPA 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 PTPA 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 PT samples and programs is not compromised;

f) not release the assigned values or acceptance limits of any PT sample prior to the conclusion of the study, except to the PTPA upon request;

g) only release participant laboratories' PT study results and/or evaluations to a designated contact at the laboratory and to laboratory accreditation bodies and/or other entities as specifically designated by the laboratory.

NOTE: PT providers may release, at the conclusion of a PT study, 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 PT 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 PT sample for which they wish to obtain or maintain accreditation.

- 5.3.2 PT providers shall employ sufficient technical and support staff to design, produce, analyze, distribute, and provide data evaluation and reporting functions for every PT 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 PT provider for any particular study. For the purposes of this Standard, "direct control" means that these functions are performed in the PT provider's facilities by the PT provider's staff or are sub-contracted by means of a written agreement with defined PT 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 PTPA and shall meet the applicable requirements of this Standard.

5.4 Complaints Handling

- 5.4.1 PT providers shall have written procedures for handling both written and verbal complaints from PT study participants and laboratory accreditation bodies who receive PT study reports.
- 5.4.2 PT providers shall record all complaints received concerning their PT studies including any remedial or corrective actions taken. This record shall be provided to the PTPA upon request.
- 5.4.3 Any complaint received by a PT provider that remains unresolved after ninety (90) days shall be submitted to the PTPA.

5.5 Notification of Sample Integrity

If any sample or analyte used in a PT study is found to not meet any of the requirements of this Standard, the PT provider shall notify all affected laboratories and their designated accreditation bodies and the provider's PTPA within seven (7) calendar days of the discovery of the non-compliance.

6.0 PT SAMPLE DESIGN AND MANUFACTURE

6.1 Design Review

PT providers shall demonstrate to the satisfaction of the PTPA that their PT sample designs and manufacturing processes:

- a) permit proficient laboratories, conforming to the calibration and quality control requirements of the analytical method(s) for which the sample was designed, to generate results that fall within the PT sample acceptance limits defined in the TNI Fields of Proficiency Testing Tables;
- b) provide equivalent challenge to all participant laboratories; and
- c) result in laboratory pass/fail rates that are consistent with historical norms.

6.2 Sample Matrices

6.2.1 The matrices of all PT samples shall, to the extent possible, resemble the matrices which participant laboratories routinely analyze.

6.2.2 The matrix for soil PT samples shall be well-characterized natural soil and shall not contain greater than 90% sand by mass.

6.3 Sample Analytes

- 6.3.1 PT providers shall prepare samples that are compliant with the criteria defined by the PTP Executive Committee and published in the TNI FoPT Tables on the TNI website.
- 6.3.2 When the PTP Executive Committee makes changes to the PT sample design criteria, PT providers shall comply with the revised requirements per the PT Board's implementation schedule.
- 6.3.3 For those multi-analyte categories designated in the TNI FoPT tables as not requiring all analytes to be spiked, the PT Provider shall determine the number of analytes based on the following.
- a) PT samples that are to be scored for one (1) to ten (10) analytes shall include all of the analytes;
 - b) PT samples that are to be scored for ten (10) to twenty (20) analytes shall include at least ten (10) analytes or 80% of the total, whichever number is greater;
 - c) PT samples that are to be scored for more than twenty (20) analytes shall include at least sixteen (16) analytes, or 60% of the total analytes, whichever number is greater;
 - d) If following 6.3.3.b) or 6.3.3.c) above and the calculated percentage of the total number of analytes in the PT sample is a fraction, the fraction shall be rounded up to the next whole number.
- 6.3.4 PT providers shall use a random-selection process to determine which analytes will be spiked and unspiked within any given PT sample.
- 6.3.4.1 PT providers may make modifications to randomly-selected analyte lists based on technical (i.e. compatibility, interference) issues.
- 6.3.4.2 Modifications to randomly-determined analyte lists shall be documented.
- 6.3.5 If the provider spikes analytes not on the TNI FoPT Tables in their PT samples, the PT provider shall ensure, to the satisfaction of the PTPA, that these additional analytes do not interfere with laboratory performance for the required analytes. Such additional analytes do not count toward the minimum analyte requirements of Section 6.3.3.

6.4 Assigned Values

- 6.4.1 PT providers shall use a random process to determine the target assigned values for their PT samples.
- 6.4.1.1 PT providers may make modifications to randomly-selected assigned values based on technical (i.e., solubility, compatibility, interference) issues.
- 6.4.1.2 Any modifications to randomly-selected assigned values shall be documented.
- 6.4.2 Assigned values for aqueous, non-microbiological analytes that are measured (chemical concentrations, isotope activities, etc.):

a) shall 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) shall be presented as three (3) significant figures.

6.4.3 Assigned values for quantitative microbiology analytes:

a) shall be equal to the study calculated study mean as specified in sections 10.2.5 and 10.2.6 of the assigned value verification and/or homogeneity testing conducted per Sections 7.1 and 7.2, and

b) shall be presented as a whole number with no more than three (3) significant figures for quantitative methods utilizing microbial colony counting techniques; for example membrane filtration methods (MF) and pour plate methods,

c) shall be set to three (3) significant figures for quantitative methods utilizing statistical probability techniques; for example most probable number (MPN) methods

6.4.4 Assigned values for solid and chemical matrix analytes:

a) shall be equal to the natural (background) concentration as analytically determined by the PT provider, plus the made-to concentrations of any spiked analytes based on gravimetric and volumetric measurements of a starting material of known concentration, and

b) shall be presented as three (3) significant figures.

6.4.5 Assigned values for qualitative analytes shall be represented as "Present" or "Absent".

6.4.6 All unspiked analytes shall have their assigned values set to "<" PTRL, as the analytes' PTRL's are listed in the FOPT tables.

7.0 PT SAMPLE TESTING

7.1 Verification of Assigned Value

- 7.1.1 PT providers shall analytically verify the assigned value of all analytes in all PT samples prior to use in a PT study.
- 7.1.2 PT 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 PT 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 PT sample being verified.

7.1.5 The PT 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 PT provider for assigned value verification shall have a repeatability relative standard deviation of not more than one-sixth of the acceptance limits (C) for the participant laboratories, as calculated per Section 10.2. For analytes that are based on the study mean and study standard deviation, the PT provider shall establish criteria approved by the PTPA that demonstrate verification of the assigned value.

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 aqueous chemistry analytes, 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 (C), 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.

For analytes that are based on the study mean and study standard deviation, the PT provider shall establish criteria approved by the PTPA that demonstrate verification of the assigned value.

7.1.9 For solid matrix and microbiology analytes, 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 (C), 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.

For analytes that are based on the study mean and study standard deviation, the PT provider shall establish criteria approved by the PTPA that demonstrate verification of the assigned value.

~~7.1.10 The standard deviation of the verification analyses shall be less than one standard deviation as calculated for the participant laboratories.~~

7.1.10+ All unspiked analytes shall be analytically verified to ensure that they are not present at or above one-half the PTRL.

7.1.11~~2~~ Any PT sample that fails to meet the requirements of this Section shall not be used in a PT study.

7.2 Homogeneity Testing

7.2.1 PT providers shall analytically verify that all analytes in all PT samples within a packaging event are sufficiently homogenous prior to their use in a PT study.

7.2.2 The homogeneity of an analyte is verified if the between-samples standard deviation of the provider's verification analyses is within one-fourth of the laboratory acceptance limits (C) as calculated per Section 10.2. For analytes that are based on the study mean and study standard deviation, the PT provider shall establish criteria approved by the PTPA that demonstrate sufficient homogeneity. **ADDING MAX of 10% BACK INTO THE STANDARD.**

~~Homogeneity shall be verified using the procedure described in Appendix A or a procedure with an equivalent ability, as determined by the PTPA, to verify that differences between samples will not impact the laboratory evaluations.~~

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

7.2.4 PT samples which fail to meet the requirements of this Section shall not be used in a PT study.

7.3 Stability Testing

7.3.1 PT providers shall verify, ~~after the closing date of the PT study but prior to the issuance of final reports,~~ that all analytes in all PT samples remained stable during the course of the study.

7.3.2 PT providers shall retain samples of the original PT study material until the close of the study for use in post-study analytical verification.

7.3.3 PT sample stability assessments shall be based on analytical data comparing the mean of a series of random samples analytically tested before the start of a study to the mean of a series of random samples analytically tested after the study close date. If the difference between the two means cannot be shown to affect an evaluation, then the analyte can be considered stable for the study period.

7.3.4 The stability of an analyte is verified if either:

a) ~~the difference between the mean of the provider's verification analyses and the mean of the provider's stability analyses is within one-fifth of the laboratory acceptance limits as calculated per Section 10.2; or~~

~~b) the provider's stability analyses meet the requirements for verification as defined in Section 7.1.~~

~~the provider's stability analyses meet the requirements for verification as defined in Section 7.1. The PT provider shall use a stability verification procedure approved of by the PTPA.~~

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

7.3.5 Post-study stability verification shall include ensuring that unspiked analytes are still below one-half the PTRL.

7.3.6 PT samples or analytes which fail to meet the criteria of this Section shall be invalidated in the PT study and described in the study discussion report.

7.4 Verification, Homogeneity and Stability Testing Reporting

7.4.1 Upon request, and only after the issuance of final evaluation reports, the PT provider shall release to a designated participant-laboratory representative the results of the provider's assigned value verification, homogeneity, and stability testing for any PT sample that the laboratory has reported data for.

7.4.2 Upon request, and only after the issuance of final evaluation reports, the PT provider shall release to laboratory accreditation bodies the results of the provider's assigned value verification,

homogeneity, and stability testing for any PT sample/analyte for which results were submitted to that laboratory accreditation body.

7.4.3 Upon request, and only after the issuance of final evaluation reports, the PT provider shall release to the PT ~~Board~~PTA the results of the provider's assigned value verification, homogeneity, and stability testing for any PT study sample/analyte.

7.4.4 PT providers shall supply to their PTPA the results of the PT provider's assigned value verification, homogeneity and stability testing for all PT samples/analytes included in each PT study.

7.4.5 The PT provider shall follow the format and schedule for submittal of this data as provided by the PTPA.

8.0 PROVISION OF PT SAMPLES

8.1 Study Duration

The study closing date shall be no more than forty-five (45) calendar days after the opening date of the study or as specified by the PTP ~~Board~~Executive Committee.

8.2 Study Instructions

8.2.1 The PT provider shall provide instructions to each participant describing:

- a) how to dilute or otherwise prepare the PT samples;
- b) how to report their data to the PT provider;
- c) the close date of the PT study; and
- ~~d) a warning that the TNI standard requires PT samples to be analyzed like "real" samples utilizing the same analysts, methods, and quality control procedures.~~

Need to remove d

8.2.2 The PT provider shall not:

- ~~a) _____~~ a) provide inappropriate assistance to the participant laboratories ~~not~~
- ~~b) _____~~ b) encourage the non-routine analysis of PT samples;

clause a is poorly worded.

- ~~c) _____~~ b) suggest or direct laboratories to use additional quality control samples or quality control samples designed specifically for a given PT sample or PT Study; ~~in conjunction with any PT study;~~

clause b is poorly worded

- ~~d) _____~~ e) provide excessive volume of any PT sample that may encourage multiple, non-routine analyses.

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

~~PT Board need to be replaced — where is this determination published? Labs are permitted to use the same PT sample for multiple methods — poor requirement.~~

8.3 Regularly Scheduled PT Studies

8.3.1 Regularly scheduled PT studies shall consist of PT sample lots (or batches) that have not been provided, in any form or by any entity, to actual or potential participant laboratories prior to the opening date of the study.

8.3.2 The assigned values for regularly scheduled PT samples shall not be released to any entity outside of the PT provider, other than the provider's PTPA, prior to the closing date of the PT study.

8.4 Supplemental PT Studies

8.4.1 For supplemental PT samples, the PT provider shall:

a) select a batch of PT samples that has been shown to meet all of the requirements of Sections 6 and 7 of this Volume;

~~should follow same format as regularly scheduled PT studies.~~

NOTE: Supplemental PT samples may be from lots that have been previously used in a PT study.

b) conduct stability testing at the close of the supplemental PT study or have data showing, to the satisfaction of the PTPA, that the sample was stable during the time period of the supplemental study;

c) have documented procedures and systems in place to track all lots and assigned values of samples received by laboratories that may be used as supplemental PT samples;

d) not supply a supplemental PT sample to a laboratory that has received that sample in a previous PT study, or in any other form, or has had access to the assigned values for that sample;

e) remove the original lot number, study number, and/or tracking ID number of each supplemental PT sample and assign a unique identifier.

~~8.4.2 If the laboratory informs the PT provider that a supplemental PT sample is being used for corrective action purposes for a specific quantitative analyte or analytes, the PT provider shall supply a supplemental PT sample that contains the specified analyte(s) spiked into the sample [i.e. the sample does not have an assigned value of zero or <PTRL for the laboratory specified analyte(s)].~~

~~I thought this clause was supposed to have been dropped and the next clause applies to all.~~

8.4.23 If the laboratory informs the PT provider that a supplemental PT sample is being used for corrective action purposes for a specific qualitative (presence/absence) test, whether the analyte of interest is spiked into the sample shall be randomly determined by the PT provider so that the laboratory will not automatically know that it is present or not.

8.4.34 The closing date of a supplemental PT study shall be the date that the participant(s) has reported study data for the required analytes^[72].

8.4.54 The closing date of supplemental PT studies shall be no more than forty-five (45) days after the opening date of the study.

9.0 SYSTEM FOR REPORTING BY PARTICIPANTS

The PT provider shall:

- a) have procedures and systems in place to ensure the accurate, timely and secure transmission of PT data from participant laboratories to the PT provider;
- b) have a reporting mechanism that ensures that the results received by the PT provider are consistent with those submitted by the participant laboratory;
- c) ensure that results reported by participant laboratories are not delayed or lost due to the provider's reporting mechanism;
- d) ensure that participant laboratory data are kept secure and that they are not subject to unauthorized dissemination either during or after the data have been reported to the PT provider.

10.0 PT STUDY DATA ANALYSIS

10.1 Data Review

- 10.1.1 PT providers shall review all PT study data sets 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 PT providers shall review all PT study data sets for disproportionately high or low failure rates compared to historical norms.

10.2 Acceptance Limit Determination

10.2.1 PT providers shall calculate acceptance limits per the requirements defined in the TNI Fields of Proficiency Testing (FoPT) Tables. Use C to denote the acceptance interval (as in $\pm C$).
~~PT providers shall calculate acceptance limits per the requirements defined in the TNI Fields of Proficiency Testing Tables. Use C to denote the acceptance interval (as in $\pm C$).~~

[KB3] 10.2.2 Analyte- or study-specific evaluation criteria defined in the TNI Fields of Proficiency Testing 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 PT 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 PT provider shall use their assigned value and calculate the acceptance limits defined in the TNI Fields of Proficiency Testing Tables.

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

- a) for samples sizes of twenty (20) or more values: the biweight mean (per Section 2.1) using fifteen (15) iterations with $c=4$ and $c_0=6$;

- b) for samples sizes of seven (7) to twenty (20) values: the arithmetic mean after outlier testing using the T test (see ASTM E178) or other PTPA-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 PTPA^[74].

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

- a) for samples sizes of twenty (20) or more values: the biweight standard deviation (per Section 2.1) using fifteen (15) iterations with $c=4$ and $c_0=6$;
- b) for samples sizes of seven (7) to twenty (20) values: the standard deviation after outlier testing with the T test (see ASTM E178) or other PTPA-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 PTPA^[75].

10.2.7 For acceptance limits calculated using the actual study median, the PT provider shall use the median calculated from all properly reported data points, as defined by the PTP ~~Board~~Executive Committee, in the data set.

10.2.8 PT Providers shall not use results reported with greater than (>) and less than (<) signs in statistical calculations.

~~PTRL~~10.3 Evaluation of Individual Participant Results^[76]

~~PTRL~~10.3.1 Assigned Value Greater than Zero ("0")the PTRL.

10.3.1.1 The result shall be scored "Acceptable" if:

- a) The numeric value reported is within the established acceptance limits.
- b) The numeric value is reported with a greater than (>) sign and the numeric value reported with the greater than sign is less than the upper acceptance limit. This clause applies to test methods for which results are reported with a greater than sign. Examples of test methods for which greater than reporting is expected include Microbiology MPN and ignitability

10.3.1.2 The result shall be evaluated "Not Acceptable" if:

- a) The numeric value is reported with a less than (<) sign.
- b) The numeric value reported is outside the established acceptance limits.
- c) The numeric value is reported with a greater than (>) sign and the numeric value reported with the greater than sign is greater than or equal to the upper acceptance limit. This clause applies to test methods for which results are reported with a greater than sign. Examples of test methods for which greater than reporting is expected include Microbiology MPN and ignitability.

~~PTRL~~10.3.2 Assigned Value "Less Than" PTRL

10.3.2.1 The result shall be scored "Acceptable" if:

a) The numeric value reported is less than the PTRL or the numeric value reported is reported with a less than sign.

10.3.2.2 The result shall be scored “Not Acceptable” if:

a) The numeric value reported is greater than or equal to the PTRL. Note that the PTP verifies the analytes that are less than the PTRL, at half the PTRL.

b) The numeric value is reported with a greater than (>) sign.

PTRL 10.3.3 No Evaluation Scoring

A reported value shall be scored “No Evaluation” if it cannot be evaluated (e.g., alpha characters for a quantitative test).

If an analyte in the PT sample is invalidated the reported value shall be scored “No Evaluation” and the PTP shall provide an explanation of the cause for invalidation in the performance evaluation report submitted to participant laboratories and the ABs for which the laboratories designated submission of the report.

PTRL 10.3.4 Not Reported Scoring

Analytes included in a PT sample but not reported by the laboratory shall be scored as “Not Reported”.

~~10.3.1~~ If the assigned value is greater than “0” the numerical value reported shall be evaluated “Acceptable” if it is within the established acceptance limits and evaluated “Not Acceptable” if the numerical value reported is outside the established acceptance limits or the numerical value is reported with a less than (<) sign and the numerical value is less than the lower acceptance limit.

~~Examples are as follows:~~

~~If the Assigned Value is “10.0”, the lower acceptance limit is “5.00” and the upper acceptance limit is “15.0”.~~

~~a) Any reported numeric value between 5.00 and 15.0 shall be evaluated “Acceptable”~~

~~b) Any reported numeric value greater than 15.0 shall be evaluated “Not Acceptable”.~~

~~c) Any reported numeric value less than 5.00 shall be evaluated “Not Acceptable”.~~

~~d) Any numeric value reported with a less than sign (<) shall be evaluated “Acceptable” if the reported numeric value associated with the less than sign is equal to or greater than the lower acceptance limit. In this example, a reported value of ‘< 5.00’ shall be evaluated as “Acceptable” because 5.00 is equal to the lower acceptance limit.~~

~~e) Any numeric value reported with a less than sign (<) shall be evaluated “Not Acceptable” if the reported numeric values associated with the less than sign is less than the lower acceptance limit. In this example, a reported value of ‘< 4.99’ shall be evaluated as “Not Acceptable” because 4.99 is less than the lower acceptance limit.~~

~~10.3.2~~ If the Assigned Value is set to the PTRL with a less than sign (<) or set to “0”, any numeric value reported with a less than sign (<), a reported value of “0” or a reported numeric value less than the PTRL shall be scored “Acceptable”.

|

For example, if the assigned value is set to "< 2.50" and 2.50 is the PTRL associated with a less than sign (<):

a) Any reported numeric value reported with a less than (<) sign shall be evaluated "Acceptable".

b) A reported value of zero "0" shall be evaluated "Acceptable".

c) A reported numeric value between "0" and 2.50 shall be evaluated "Acceptable".

A reported numeric value greater than 2.50 shall be evaluated "Not Acceptable". If the Assigned Value is set to the PTRL with a less than sign (<), any numeric value reported with a less than sign (<), or a reported numeric value less than the PTRL shall be scored "Acceptable".

For example, if the assigned value is set to "< 2.50" and 2.50 is the PTRL associated with a less than sign (<):

a) Any reported numeric value reported with a less than (<) sign shall be evaluated "Acceptable".

b) A reported value of zero "0" shall be evaluated "Not Acceptable".

c) A reported numeric value greater than 0 and less than 2.50 shall be evaluated "Acceptable".

d) A reported numeric value greater than or equal to 2.50 shall be evaluated "Not Acceptable".

WHERE IN THIS MODULE DOES IT ALLOW FOR SETTING OF THE ASSIGNED VALUE TO THE PTRL?

10.3.3 A reported value shall be evaluated as "No Evaluation" if it cannot be evaluated (e.g., alpha characters for a quantitative test).

10.3.4 Analytes included in a PT sample but not reported by the laboratory shall be evaluated as "Not Reported".

10.3.5 If the PT Provider invalidates an analyte in a PT study, 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 participant labs and ABs.

11.0 GENERATION OF STUDY REPORTS

11.1 Schedule

11.1.1 The reports defined in Sections 11.2 and 11.3 shall be submitted to the required parties no later than twenty-one days after the close of the study.

11.1.2 Reports shall be submitted to participant laboratories and laboratory-requested accreditation bodies within the same twenty-four (24) hour period.

11.2 Final Evaluation Report

11.2.1 PT providers shall submit final evaluation reports to all participant laboratories.

11.2.2 PT providers shall submit final evaluation reports in hardcopy or electronic form to all laboratory accreditation bodies that have been requested by the laboratories to receive reports.

~~NOTE: Final evaluation reports may be submitted in hardcopy or electronic form. ROLL THIS CLAUSE INTO 11.2.2~~

11.2.3 The following information shall be included in the final evaluation report:

- a) PT provider name;
- b) PT provider PTPA accreditation number;**
- c) participant laboratory name;
- d) participant laboratory physical address;
- e) name, title and telephone number of laboratory point of contact, as provided;
- f) participant laboratory's primary accreditation body ID, as provided;
- ~~g) **EPA laboratory identification number**~~

~~g)h)~~ study type and study number; WHAT IS A STUDY TYPE?

~~h)i)~~ opening and closing dates of the study;

~~i)j)~~ date report was prepared;

~~j)k)~~ date report was amended, if applicable;

~~k)l)~~ study discussion including any pertinent information which addresses unusual details of the study (e.g., need to change an assigned value or delete an analyte from evaluation).

11.2.4 The following information shall be included for each PT sample/analyte in the final evaluation report:

a) lot or study number;

b) analyte name;

c) analyte code defined in the TNI FoPT Tables;

d) identification of those analytes included and not included in the PT provider's PTPA accreditation;

e) assigned value;

f) acceptance limits;

g) laboratory value, as reported;

h) method description, as reported;

~~i) analysis dates as reported by the participating laboratory;~~

~~ji)~~ evaluation per Section 10.3;

~~kj)~~ mean calculated from all study participant data; and

~~lk)~~ standard deviation calculated from all study participant data.

11.2.5 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 Study Failure Rate Report

11.3.1 Upon request by either a participant laboratory or a laboratory accreditation body, the PT 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 PT provider shall not disclose specific laboratory results or evaluations to any parties without a written release from the laboratory.

APPENDIX A

Guidance Procedure for Testing the Homogeneity and Stability of PT Samples

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 PT 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 PT 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 PT 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 PT 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 PT 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 PT; 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 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 PT 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 sample.
- b) Use samples that have been packaged for distribution for a round of the proficiency testing scheme.
- c) Determine a number g of the samples that will be tested, where $g \geq 5$. For analytes where there is a concern about heterogeneity (most analytes in soil samples, and some analytes in water), let $g \geq 10$. [*$g = 5$ 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 $G \geq 10$); 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 2 g 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_i ..., the general average \bar{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_i ... 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 PT 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 $\pm C$). 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_5 . 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:

$$(1) \quad s_s \leq 0.25C \quad \text{and if}$$

$$(2) \quad d_s \leq 0.25C$$

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:

$$(3) \quad x_{t,k} \text{ where}$$

g is the number of samples

t represents the sample ($t = 1, 2, \dots, g$)

k represents the test portion (replicate) ($k = 1, 2$)

Define the sample averages as:

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

and the between-test portion ranges as:

$$(5) \quad w_t = |x_{t,1} - x_{t,2}| \quad \text{[skip this step]}$$

Calculate the general average:

$$(6) \quad x_{..} = \sum x_{t.} / g$$

the standard deviation of sample averages:

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

and the repeatability standard deviation:

$$s_i = \sqrt{\sum w_i^2 / (2g)} \quad \text{[use a suitable repeatability estimate } s_i \text{]} \quad (8)$$

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

Finally, calculate the between-samples standard deviation as:

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

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 closing date for the round, but prior to formal evaluation of participant results. Samples shall have been stored under conditions similar as these 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 $g \geq 3$ ($g = 3$ 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:

$$|x_{..} - y_{..}| \leq 0.2C \quad (10)$$

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 proficiency 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.

A.3 GUIDANCE EXAMPLE: MERCURY IN WATER ($\mu\text{g/L}$)

In a proficiency testing scheme for water the standard deviation for proficiency assessment for Mercury at 10 µg/L has been set at $\sigma = 1.1$, and the evaluation interval is 2 times this, or 2.20. According to the criterion given in Section A.2.1, the between-samples standard deviation should be no more than: $0.25 \times 2.2 = 0.550$.

For a heterogeneity check, five (5) of the water samples prepared for the proficiency-testing scheme were selected at random, and the mercury contents of two test portions from each sample were determined. The data are shown in Table 1, together with sample averages and between-test portion ranges. There is no apparent trend in the sample averages.

The formulae given in Section A.2.2 are used to calculate the following values:

general average	X_{\dots}	=	9.94
standard deviation of sample averages	s_x	=	0.361
repeatability standard deviation	s_r	=	0.228
between-samples standard deviation	s_s	=	0.323

The between sample SD (s_s) is less than 0.550, so it may be concluded that the samples are adequately homogeneous for use in the proficiency testing scheme.

At the conclusion of the study time period, three retained samples are randomly selected for the stability check. These samples are noted as samples 6-8 in Table 1. The criterion for the stability check is for the difference between means to be less than: $0.2 \cdot 2.2 = 0.44$.

The average of the three samples is 10.07 and the difference between the average of the homogeneity check samples and stability samples is $(10.07 - 9.94 = 0.13)$, which is less than 0.44, so the samples are sufficiently stable.

Table 1. Results for Homogeneity and Stability Checks, Mercury in Water

SAMPLE NUMBER i	TEST PORTION 1	TEST PORTION 2	SAMPLE AVERAGE x_i	BETWEEN-TEST- PORTION RANGE w_i
1	10.5	10.4	10.45	0.1
2	9.6	9.5	9.55	0.1
3	10.4	9.9	10.15	0.5
4	9.5	9.9	9.70	0.4
5	10.0	9.7	9.85	0.3
STABILITY SAMPLES				
6	10.3	10.5	10.35	
7	9.9	9.5	9.7	
8	10.3	10.0	10.15	