PT15-01 – CALA PT Program - Scheme
Revision 1.8 – April 17 2014

CALA
Proficiency Testing

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CALA PT PROGRAM - SCHEME

1.0 INTRODUCTION

The Canadian Association for Laboratory Accreditation Inc. (CALA) is a not-for-profit organization and operates the Proficiency Testing (PT) Program on a cost-recovery basis. The CALA PT Program is accredited by A2LA for most of its PT samples and conforms to ISO/IEC 17043:2010 *Conformity Assessment-General requirements for proficiency testing.* The scope of accreditation can be found at a2la.org/dirsearchnew/ptproviders.cfm

This document describes the general overview of the CALA PT scheme. Detailed procedures for the undertaking of a PT study are found in PT15-03 – *CALA PT Program - Procedures*.

Other CALA documents related to the CALA PT Program include:

- PT15-02 – *CALA PT Program – Policies*;
- PT15-03 – *CALA PT Program – Procedures*;
- PT15-04 – *CALA PT Program – Collaborator Laboratories*;
- PT15-05 – *CALA PT Program – Regression Equations*;
- P02-04 – *CALA Program Description – PT Catalogue*;
- P02-05 – *CALA Program Description – List of Approved PT Providers*; and
- PT33 – *List of PT Collaborators*.

These documents can all be found in the CALA on-line library (www.cala.ca/library.html).

2.0 REFERENCES

- ISO/IEC 17043:2010 – *Conformity assessment – General requirements for proficiency testing*;
- ISO/IEC 17025:2005 – *General requirements for the competence of testing and calibration laboratories*;
- IUPAC/ISO/AOAC –2006. *The international harmonized protocol for the proficiency testing of analytical Chemistry laboratories*; and,
- Grubbs, F. E., 1969. *Procedures for Detecting Outlying Observations in Samples*. Technometrics, Vol. II, No. 1, pp. 1-21.
- ISO 13528 – *Statistical methods for use in proficiency testing by interlaboratory comparisons*.

3.0 CALA PT SCHEME

3.1 Objectives Of The PT Scheme

The objective of the CALA PT program is to provide cost-effective, internationally recognized, proficiency testing services to its participants. The purpose of the scheme is to provide laboratories with an educational tool that allows them to assess their performance relative that of their peers, using the 95% confidence interval as the general acceptance criteria.

3.2 PT Plan

The following table provides the general scheme information that is followed for all CALA PT offerings:

Requirement from clause 4.4.1.3 of ISO/IEC 17043	CALA's Fulfilment of requirement
a) Provider	The Canadian Association for Laboratory Accreditation Inc. Suite 310, 1565 Carling Avenue, Ottawa, Ontario K1Z 8R1 (613)233-5300/(613)233-5501.
b) Coordinator	The CALA Proficiency Testing Manager is responsible for all aspects of the Proficiency Testing Trogram. The CALA Data Manager is responsible for maintaining the CALA web-data-entry system and database, and is responsible for all data processing and issuing of reports.
c) Subcontracted Activities	CALA uses subcontractors for the production, characterization and shipping of PT samples. CALA only contracts with organizations competent for the production, characterizing and shipping of PT samples. Competence is determined as either accreditation to ISO/IEC 17043-ISO/IEC 17025 or through periodic audits. The current list of subcontractors is found in PT33-*List of PT Collaborators* and PT31-*List of Testing Labs used for Environment Canada PT Sample Verification*.
d) Participation	Participation in the CALA PT Program is open to all testing laboratories, regardless of location. Where limitations are placed on methodology, these limitations are indicated in P02-04-*CALA Program Description – PT Catalogue*.
e) Participation Level	The typical participation levels range between 20 and 250 participants per analyte. Participation levels lower than this will be accepted if there is an acceptable history but the minimum participation level is 11.
f) Selection of analyte	Only analytes that are commonly performed by environmental and food testing laboratories are included in the PT Program. They must be sufficiently homogeneous and stable, and matrix interference will not contribute significantly to overall uncertainty of the study.
g) Concentration ranges	Concentration ranges are based on typical analytical capabilities, concentrations typically found in customer samples, and specific regulatory limits. In this way, the PT samples are fit-for-purpose. Refer to P02-04-*CALA Program Description – PT Catalogue*.

Requirement from clause 4.4.1.3 of ISO/IEC 17043	CALA's Fulfilment of requirement
h) Potential major sources of errors	The potential major sources of error in the CALA PT Program include: sample homogeneity; sample stability; sample packaging; sample handling and shipping; differences between methods used by participants; and inter-participant variation.
i) Sample production, characterization and distribution.	All PT samples are produced, characterized and distributed under contract to CALA. Details on these requirements are detailed in PT12A-*Request for Proposal* (revised as necessary). Contractors are required to document their CALA specific sample production, characterization, storage and shipping procedures.
j) Procedures for preventing collusion and falsification	All participants must agree to P04-01 - *Terms and Conditions of Accreditation and/or Proficiency Testing* before they can receive samples. In addition to CALA specific requirements, the terms and conditions require participants to comply with the policies listed in PT15-02 *CALA PT Program - Policies*. As well, participants are not permitted to report PT results on the CALA Web-Data-Entry system without indicating that they have read and accept the CALA terms and conditions.
k) Information provided to participants	Details of the information provided to participants is summarized in section 3.7 Below and in PT15-03-*CALA PT Program – Procedures.*
l) Dates for shipments and reporting deadlines	Deadlines for changes to PT registration, dates for sample shipment and deadlines for result reporting are posted on the CALA web-site (http://www.cala.ca/pt_ship_schedule.html). As well, the deadline for reporting is included with each sample shipment. If there are any changes to the published schedule, all registered participants are notified by email.
m) Instructions to participants on methods to use	Each PT test group has a specific instruction sheet associated with it. Each instruction sheet provides instructions on special handling requirements and, where necessary, limitations on methods that can be used. Analytical limitations are also provided in P02-04-*CALA Program Description – PT Catalogue*. The instruction sheets also contain information on how to report problems with shipping.
n) PT sample homogeneity and stability	The uncertainty associated with PT sample homogeneity and stability shall not contribute significantly to the overall uncertainty of the PT study. This is assessed through an examination of participant reported data. Refer to PT15-03-*CALA PT Program – Procedures* Sections 2.11.

Requirement from clause 4.4.1.3 of ISO/IEC 17043	CALA's Fulfilment of requirement
o) Participant reporting	Participants report their PT results to CALA using the CALA Web-Data-Entry System (www.cala.ca/profwebdata.html). Instructions for use of the Web-Data-Entry System are available on the CALA web-site. Access to this system is restricted to participants through the use of user names and confidential passwords. The Web-Data-Entry System is available to all participants from the date of sample shipping to midnight of the reporting deadline. Participants may verify entered results and make corrections until the reporting deadline. With every web-entry page, there is the opportunity for participants to enter comments. These comments are reviewed and, where necessary, acted on before final PT reports are issued. CALA controls the number of significant figures that can be reported by restricting the number of decimal places that can be reported.
p) Statistical analysis	The CALA PT Program is essentially a consensus-based program. The assigned value is the Robust mean of participant results after obvious errors have been removed. The standard deviation of proficiency is established from Robust inter-laboratory standard deviation, either from historic studies (regression equations) or specific to the actual study. Laboratory performance is determined by use of a z-score. Refer to section 3.6 below and PT15-03-*CALA PT Program – Procedures* (Appendix I) for details.
q) Metrological traceability and uncertainty of the assigned value	The CALA PT scheme is an evaluation of participant performance as it compares to other participants. As such, metrological traceability is not applicable. Standard uncertainty of the assigned value is detailed in PT15-03-*CALA PT Program – Procedures (*Appendix I.8*)*.
r) Evaluation of participant performance	Laboratory performance is determined by use of a z-score. Refer to section 3.6 below and PT15-03-*CALA PT Program – Procedures* (Appendix I) for details.
s) Preliminary reports, confidential reports and generic reports	Reports provided to participants are detailed in section 3.7 below.
t) Confidentiality	Unless otherwise agreed or requested by the participant, all communication between CALA and the participant, and all participant specific PT reports, are maintained in confidence.
u) Lost or damaged samples	When notified by participants of lost or damaged samples, replacement samples will be provided as per section 2.6 of PT15-02-*CALA PT Program – Policies*. Further instructions are provided with each instruction sheet.

Requirement from clause 4.4.1.3 of ISO/IEC 17043	CALA's Fulfilment of requirement
Program Committee	The CALA Program Committee is a panel of experts that provide technical advice to CALA. This committee is comprised of representatives from member laboratories, technical experts and regulators. Input to this committee is also obtained through surveys, workshops, feedback from presentations, outcome of complaint and non-conformance investigations, etc. The committee meets at least four times per year and is used to provide recommendations to CALA on all CALA programs. These recommendations are reviewed by the appropriate Manger and, where appropriate, approved for implementation by the President & CEO (hereinafter referred to in this document as the CEO). The list of Program Committee members and its terms of reference are documented in Q13-*Program Committee Terms of Reference and Operating Procedures.*

3.3 Addition Of New Analytes To The CALA PT Scheme

Recommendations for new PT are received from the Program Committee, as the result of a survey of CALA members, or recommendations from other stakeholders.

Approval of new PT is the responsibility of the Proficiency Testing Manager. The Proficiency Testing Manager ensures that the selected Collaborator laboratory is advised in writing of the requirements and that a documented discussion on capability, resources and the decisions made takes place. Information generally required before CALA authorises delivery of new PT samples or existing PT samples from a new Collaborator laboratory is included in PT15-04 *CALA PT Program - Collaborator Laboratories*.

Unless otherwise specified, new PT follows the same scheme as used for existing PT.

At least the first two studies for newly added PT are designated as *pilot* studies. The participant receives a copy of their own data from CALA at the conclusion of the study along with the normal summary data, including z-scores. However, final PT scores are not assigned.

Participation in a pilot study does not adversely affect a participant's existing CALA accreditation status, nor does it affect any current applications for CALA accreditation.

For a participant accredited by CALA for a newly offered PT analyte, failure in the first live round after a minimum of two pilot rounds:

- results in a *Possible Suspension* if the laboratory participated in the last two pilot rounds; and,
- results in a *Suspension* if the laboratory did not participate in the last two pilot rounds.

3.4 Study Frequency And Composition

In general, each Test Group is shipped twice per year. They are split into two groups, one group that is shipped in January and June, and a second group that is shipped in March and

October. The water microbiology samples are shipped in March and October to avoid the hottest and coldest times of the year.

Important dates for each study (i.e., shipping date, reporting deadline and deadline for changes to PT registration) are posted on the CALA web site (www.cala.ca/pt_ship_schedule.html).

Most test groups consist of four separate samples, usually of different analyte concentration. Approximate analyte concentration ranges are detailed in P02-04 – *CALA Program Description - PT Catalogue* and are referenced in the PT Instruction Sheets. These are approximate concentrations intended to provide guidance to laboratories about the appropriateness of the CALA PT samples; actual sample concentrations may be marginally outside these ranges.

Each test group may contain one or more analyte.

3.5 Sample Characteristics

PT samples used in the CALA program are generally whole samples, not ampoules, concentrates or extracts. Whenever possible, the samples are designed to mimic typical matrices experienced by participant laboratories. The concentration range for each analyte are established based on analytical capabilities, typical sample concentrations, regulatory limits (where available) and the ability to produce homogeneous and stable samples.

When a new formulation of an existing PT is considered (e.g., different preservative, simulated wastewater, etc.), the new formulation is tested on one of the four samples during a normally scheduled study. When this occurs, participants are notified in advance and the final performance evaluation is limited to the three samples using the existing formulation.

The quantity of sample provided for each sample is sufficient for analysis and generally consistent with typical sample volumes collected by laboratories.

Each individual PT sample in a production lot is individually numbered in the order it is packaged, and tracked to a participating laboratory to facilitate investigation of homogeneity.

Each production lot of samples is characterized for homogeneity and stability as per procedures detailed in PT15-03 *CALA PT Program - Procedures*.

3.5.1 *Challenge* Samples

On occasion, CALA may introduce a *challenge* sample into the scheme. A *challenge* sample is presented as one of the samples in a test group, containing the same analytes. However, it may be presented in a matrix that is known to be more challenging or contain a known interferent. When a *challenge* sample is used, the participants are not made aware of it until after the study is closed. The *challenge* sample is not used to determine the performance of

the participant but a separate summary report is produced and provided to all participants for their review.

3.6 Scoring System

Participant performance is evaluated for each analyte in the PT study by a quantitative method that is consistent with ISO/IEC 17043:2010, ISO 13528 and the *International Harmonized Protocol for Proficiency Testing of (Chemical) Analytical Laboratories* (2006).

Unless otherwise specified, the CALA PT program uses three significant digits when accepting and reporting analytical data. Computer routines and other calculated values such as reference value reports and summary statistics use more than three significant digits to avoid rounding error.

Numbers ending in a five (5) are always rounded up in the CALA Database.

The CALA scoring system is a comparison against peers. Both the target values and acceptance criteria are based on consensus of participants. This approach is used because it has been used since the CALA PT Program was started (1991) and has been demonstrated to work well for the standard environmental tests. It is also used because it is flexible enough to change with improvements in technologies. This scheme was developed, and continues to be modified, through the input of participants, accreditation bodies and regulators. The scoring system is based on the following assumptions:

- The distribution of reported data approximates a normal distribution with no significant and recurring skewing or bi-modality;
- For any analyte, average results are similar, regardless of method used. When this is observed not to be the case, biased methods are excluded from participation;

Although performance evaluations are not made on a method specific basis, a report is produced for each analyte in each study that provides a statistical summary by method as well as a graphical representation of the same. PT15-03 – *CALA PT Program – Procedures*, Section 2.11 describes how this report is reviewed by CALA and the actions that may be taken as a result of this review.

The general procedure for evaluating participant performance for most test groups is as follows:

i) non-detect values are temporarily removed from the set of reported data. This is done because it has been observed that including the non-detect levels in the determination of the assigned value would result in a positive bias;

ii) obvious reporting errors (e.g., wrong units) are removed temporarily;

iii) Robust consensus mean, $\overline{X}$, and standard deviation, *stdev*, are calculated from the remaining data;

iv) regression equation standard deviation, s!, is estimated from the regression between consensus mean and consensus standard deviation of historic studies (PT15-05 – *CALA PT Program –Regression Equations*);

v) z-scores are calculated for each reported result as follows:

if stdev > s! then, $$z\ score = \frac{(x_i - \overline{X})}{stdev}$$

or (if RDL is reported) $$z\ score = \frac{(x_i - \overline{X})}{\sqrt{stdev^2 + (RDL/3)^2}}$$

if stdev < s! then, $$z\ score = \frac{(x_i - \overline{X})}{s!}$$

or (if RDL is reported) $$z\ score = \frac{(x_i - \overline{X})}{\sqrt{s!^2 + (RDL/3)^2}}$$

where x_i = the reported result,
$\overline{X}$ = consensus mean,
stdev = inter-laboratory standard deviation,
s! = regression equation standard deviation,
RDL = the participant detection level

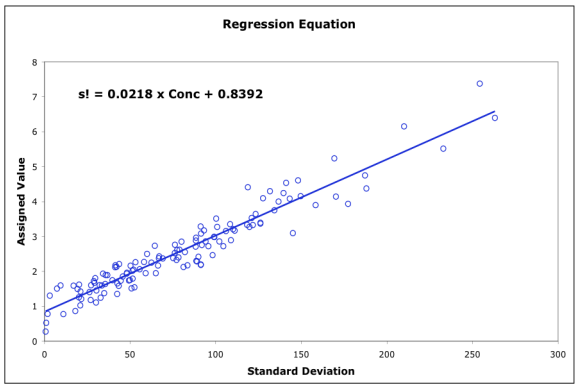
vi) the average absolute z-score is calculated from the four samples for each analyte,
vii) an average absolute z-score of 2.0 or lower is considered an acceptable score.

The regression equations are in the following format,

$$s! = m \times Conc + b$$

where m = the slope of conc vs inter-lab stdev of historic studies,
Conc = the consensus mean from historic studies,
b = the intercept of conc vs inter-lab stdev of historic studies.

The procedures for establishing the regression equations as well as current regression equation values are found in PT15-05 *CALA PT Program - Regression Equations*. The plot to the right displays a typical regression equation plot.



3.7 Proficiency Testing Reports

Several reports are provided to the participant during the course of a PT study.

3.7.1 Preliminary Report

An electronic report that contains all of the evaluation data that are found in the final report is emailed to participants within one week of the close of the study and is intended to provide participants with an indication of their performance so that investigations may commence without unnecessary delay. These reports use the arithmetic mean (outliers removed by Grubbs) rather than the robust procedures and are just intended to provide rapid feedback on possible problems. These reports are not an official evaluation and final scores will change throughout the course of data examination by CALA.

3.7.2 Final Proficiency Testing Report

Within 3 weeks of the deadline for submission of results, CALA issues a Final Proficiency Testing Report that contains both the confidential results of the individual participant's performance (pdf), an excel file containing the same information, and the generic summary report described below.

3.7.3 Generic Summary Report

A general summary report (PT35 – *Proficiency Testing Summary Report*) is produced for each quantitative test group. Each report contains:

- Summary of evaluation procedure;
- Comparison between CALA assigned value, robust mean and median;

- Plots used to assess homogeneity and stability, and indication when the standard deviation has been modified;
- Overall frequency distribution with the normal curve based on mean and standard deviation overlaid;
- Z-score plots;
- Graphical representation of the mean and distribution of results for the most commonly used methods; and
- Statistical summary of the most commonly used methods.

3.8 Notification Of PT Recognition

CALA grants proficiency testing recognition for analytes following a successful PT study. After each PT study, participants are notified in writing of any new tests for which CALA PT recognition has been granted and for any status changes resulting from PT performance.

A password-protected directory is available on the web site that contains all PT registration and current status for each participant.

3.9 Suspension And Withdrawal

After PT recognition has been granted, the following actions are taken upon an unacceptable PT score:

- First Failure: a Notice of Possible Suspension is issued;
- Second Consecutive Failure: a Notice of Suspension is issued; and,
- Third Consecutive Failure: a Notice of Withdrawal is issued.

Withdrawal of proficiency testing recognition does not stop the shipment of the affected PT samples. These samples will continue to be shipped until CALA is notified otherwise.

3.10 Remedial Actions

With each notice of *Possible Suspension* and *Suspension* (as well as with notices of Withdrawal for accredited laboratories) a blank Corrective Action Report (CAR) form is issued to the participant (www.cala.ca/carform.html). Participants must complete the form, detailing the investigation conducted, the root cause identified and corrective action selected to prevent recurrence, and submit it to CALA for review.

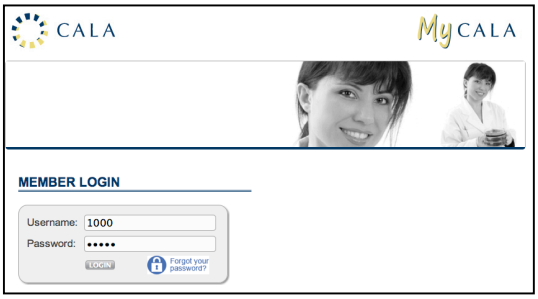
Each corrective action report is reviewed for completeness by the Proficiency Testing Manager. This review does not attempt to judge the accurateness of the root cause or the appropriateness of the corrective action; the primary objective of these evaluations is to ensure that laboratories are thoroughly investigating unacceptable PT performance.

This policy is further detailed in PT15-02 – *CALA PT Program – Policies*.

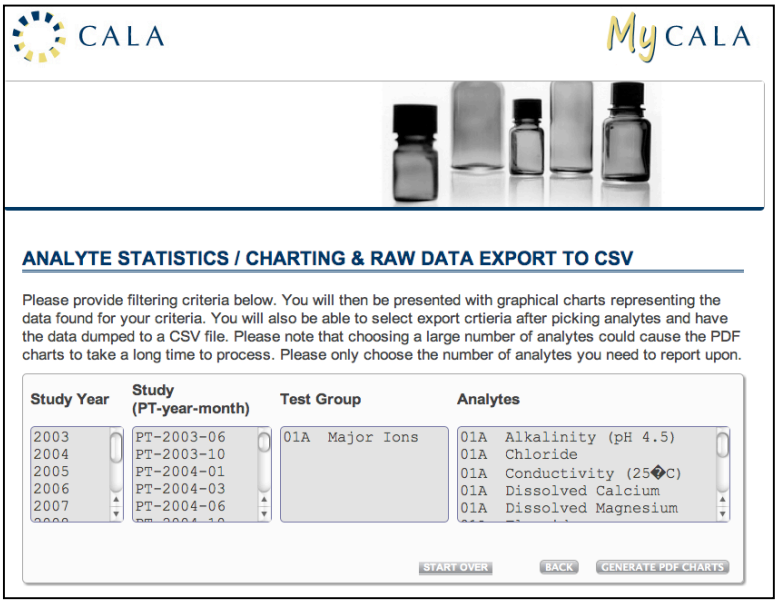
4.0 ON-LINE TRENDING TOOLS

At any time, CALA PT participants can use the CALA on-line trending tools to download data from historic PT studies and produce various trend plots based on participant selected criteria.

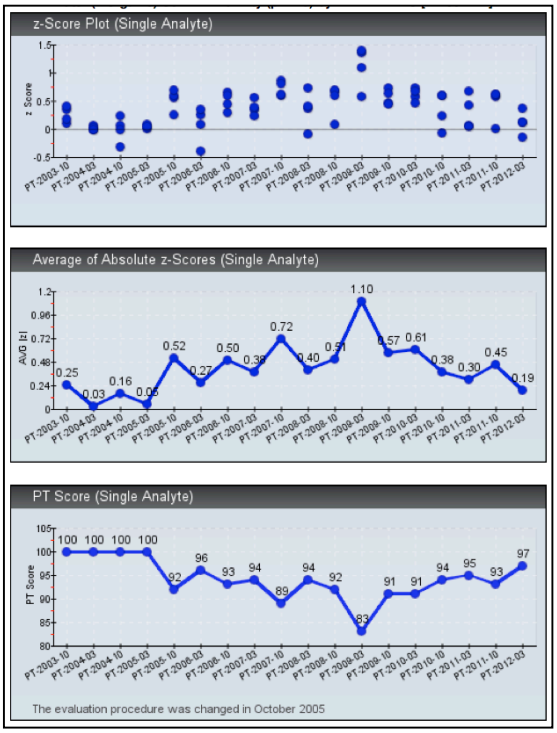
These tools are accessed through www.my.cala.ca by entering the CALA laboratory number and the laboratory specific PT password.



The laboratory then selects a year(s), test group(s) and analyte(s) to produce trend plots for the selected data.



The tool then produces a multi-page report in .pdf format. The same tool can also be used to download the raw data to allow the laboratory to produce their own trend plots.



5.0 DEFINITIONS

Analyte: The component of the sample that is quantified and reported. Often referred to as a parameter (e.g., Phosphorus).

Assigned Standard Deviation s: The measure of dispersion used to determine the allowable deviation for reported results. This value is determined according to the PT scheme.

Assigned Value X: The value attributed to a particular property of a proficiency test item. Participants will often refer to this as the target value or the expected value.

Bias: A systematic, non-random, deviation from the true value.

Collaborator: organization or individual contracted by the proficiency testing provider to perform activities specified in ISO/IEC 17043 and that affects the quality of a proficiency testing scheme. Analogous to a subcontractor in ISO/IEC 17025.

Coordinator: one or more individuals with responsibility for organizing and managing all of the activities involved in the operation of a proficiency testing scheme.

Design Value: This is the sample concentration that the collaborator laboratory is aiming for in production of the PT sample. Due to the large volumes involved, losses during production, and the use of natural materials, these values are not used as the assigned value.

Participant: A laboratory, organization or individual, that receives proficiency test items and submits results for review by the proficiency testing provider.

PT Provider: The organization that takes responsibility for all tasks in the development and operation of a proficiency testing scheme.

PT Scheme: Proficiency testing designed and operated in one or more rounds for a specified area of testing, measurement, calibration or inspection.

PT Sample: A sample, product, artifact, reference material, piece of equipment, measurement standard, data set or other information used for proficiency testing.

PT Study: A single complete sequence of distribution of proficiency test items, and the evaluation and reporting of results to the participants.

Reference Laboratory: A laboratory that is subcontracted to provide analytical characterization (i.e., homogeneity and stability testing) of PT samples. Analogous to a subcontractor in ISO/IEC 17025.

Test Group: A specific PT offering with a unique matrix and analyte composition (e.g., C01A Major Ions).

z-score: The normalized score upon which the final PT score is determined.