




Standards Council of Canada
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Requirements for the Accreditation of Mineral Analysis Testing Laboratories

CAN-P-1579
September 2008

Program Specialty Area – Mineral Analysis (PSA-MA)

REQUIREMENTS FOR THE ACCREDITATION OF MINERAL ANALYSIS TESTING LABORATORIES

**PROGRAM SPECIALTY AREA - MINERAL ANALYSIS
(PSA-MA)**

EXIGENCES RELATIVES À L'ACCRÉDITATION DES LABORATOIRES D'ESSAIS D'ANALYSE MINÉRALE

DOMAINES DE SPÉCIALITÉ DE PROGRAMME – ANALYSE MINÉRALE

CAN-P-1579

September 2008

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The following, issued separately from this document, are an integral part of these requirements:

CAN-P-1579 APPENDIX A - PSA-MA Criteria for Assessment of Proficiency Testing Performance

CAN-P-1579 APPENDIX B - Definitions for the PSA-MA Program

About the Standards Council of Canada

The Standards Council of Canada ("SCC" or "the Council") is a crown corporation established by an Act of Parliament in 1970, amended in 1996, to foster and promote efficient and effective voluntary standardization in Canada. It is independent of government in its policies and operations, although it is financed partially by Parliamentary appropriation. The SCC Governing Council consists of members from government and the private sectors.

The mandate of the Council is to promote the participation of Canadians in voluntary standards activities, promote public-private sector cooperation in relation to voluntary standardization in Canada, coordinate and oversee the efforts of the persons and organizations involved in the National Standards System, foster quality, performance and technological innovation in Canadian goods and services through standards-related activities, and develop standards-related strategies and long-term objectives.

In essence, the Council promotes efficient and effective voluntary standardization in Canada in order to advance the national economy, support sustainable development, benefit the health, safety and welfare of workers and the public, assist and protect consumers, facilitate domestic and international trade and further international cooperation in relation to standardization.

The Council serves as the government's focal point for voluntary standardization and represents Canada in international standardization activities, sets out policies and procedures for the development of National Standards of Canada, and for the accreditation of standards development organizations, of product certification bodies, of testing and calibration laboratories, of quality and environmental management systems registration bodies and of quality management systems and environmental auditor certifiers and training course providers, and promotes and supports the principle of recognition of accreditation or equivalent systems as a means of decreasing the number of multiple assessments and audits, both in Canada and with Canada's trading partners.

This document is one of several issued by the Standards Council of Canada to define the policies, plans, and procedures established by the Council to help achieve its mandate.

Requests for clarification and recommendations for amendment of this document, or requests for additional copies should be addressed to the publisher directly.

PREFACE

The Program Specialty Area - Mineral Analysis (PSA-MA) program is operated and managed by the SCC through its Program for Accreditation of Laboratories - Canada (PALCAN). The assurance that a mineral analysis laboratory adheres to recognized practices and standards can be achieved through accreditation in this program. Accreditation under the PSA-MA program is the formal recognition by the Standards Council of Canada of the competence of a mineral analysis laboratory to perform this type of activity. It is not a guarantee that test results will conform to standards or agreements between a testing laboratory and its customer's business transactions between an accredited testing laboratory and its customers are legal matters between the two parties.

Mineral analysis testing includes all media used in mining exploration and processing including, but not limited to, sediments, rocks, ores, metal products, tailings, other mineral samples, water and vegetation.

The Task Group Laboratories (TG Labs) is constituted by and reports to the Advisory Committee on Conformity Assessment (ACCA). The TG Labs is responsible for applications for accreditation from laboratories, assessments of applicant laboratories and reassessments of accredited laboratories and making recommendations, as required, to the ACCA and the Council.

The specific requirements for mineral analysis testing in these requirements were developed through the Mineral Analysis Working Group (MAWG) that is constituted by and reports to the TG Labs. The technical basis is drawn from published principles, practices and procedures used or promoted by national/international organizations.

This document was designed to meet International Organization for Standardization (ISO)/International Electrotechnical Commission (IEC) Standard 17025:2005 requirements. Rather than serving as a "stand alone" document, it was designed to harmonize with and complement the SCC document CAN-P-4E (ISO/IEC 17025:2005), "*General Requirements for the Accreditation of Calibration and Testing Laboratories*", which is ISO/IEC 17025:2005 verbatim, and to follow the standard SCC assessment protocol. These requirements are also based on the ISO/IEC Guide 43-1:1997(E), "*Proficiency Testing by Interlaboratory Comparisons - Part 1: Development and Operation of Proficiency Testing Schemes*" and the other SCC, International Laboratory Accreditation Cooperation (ILAC), Asia Pacific Laboratory Accreditation Cooperation (APLAC) and ISO documents and references.

Accreditation by SCC requires an on-site assessment of the laboratory to demonstrate competence and conformance with the requirements of CAN-P-4E as well as prior and continued participation and satisfactory performance in the proficiency testing scheme for each test accredited as outlined in this document.

The scope of these requirements will be evaluated periodically to respond to customer, laboratory and accreditation requirements as well as improvements in the available science and technology or regulatory changes.

This Preface is not an integral part of this document.

INTRODUCTION

The general requirements for the competence of testing and calibration laboratories are described in CAN-P-4E (ISO/IEC 17025:2005). These requirements are designed to apply to all types of calibration and objective testing and therefore need to be interpreted with respect to the type of calibration and testing concerned and the techniques involved. CAN-P-1510E (Assessment Rating Guide) is the tool used to assess conformance to requirements in CAN-P-4E. The SCC policy documents (CAN-P-1630, CAN-P-1570, etc.) also apply.

This PSA-MA document provides an elaboration, interpretation and additional requirements to those requirements in CAN-P-4E that are required for laboratories involved in performing mineral analysis testing. It is expected that where no elaborations, interpretations or additional requirements are stipulated in this document for the elements of the standard, that the SCC PALCAN Policy documents (CAN-P-1630, CAN-P-1570, etc.) and best scientific practices in the area of mineral analysis testing will guide the assessment process.

The program is designed to ensure mineral analysis testing laboratories meet minimum quality and reliability standards and to ensure a demonstrated uniform level of proficiency among these mineral analysis testing laboratories. This document identifies the minimum requirements for accreditation of laboratories supplying mineral analysis testing services. This includes, but is not limited to, the measurement of all media used in mining exploration and processing including sediments, rocks, ores, metal products, tailings, other mineral samples, water and vegetation.

This document does not re-state all the provisions of CAN-P-4E and laboratories are reminded of the need to comply with all of the relevant criteria detailed in CAN-P-4E and the current edition of the CAN-P-1570 *"PALCAN Handbook"*. The main clause numbers in this document generally follow those of CAN-P-4E, but since not all clauses require interpretation, the numbering of clauses may not be continuous. Clause 6, the specific requirements for the evaluation of laboratory performance by proficiency testing, is unique to this document.

To obtain initial accreditation by SCC under the PSA-MA program, a laboratory shall successfully complete both a proficiency testing regimen and an on-site assessment by technical specialists. The assessments will be conducted using standard SCC assessment protocols such that:

- a comprehensive on-site assessment of the program will occur every two years; and
- surveillance questionnaires, including evaluation of the laboratory's quality manual and proficiency testing results, will be conducted in the intervening years.

For the initial assessment, the applicant shall complete and return the CAN-P-1570 Appendix A *"Application for Accreditation"* document as outlined in CAN-P-1570 *"PALCAN Handbook"* as well as placing in the right hand column of the *Assessment Rating Guide* (CAN-P-1510E) the appropriate references to their Quality System (QS) Quality Manual, any other Quality System documents and Standard Operating Procedures (SOP). Any requested copies of their specific SOPs or other documents shall be supplied at least two weeks prior to the on-site assessment visit. For every scope extension and/or re-assessment visit, the applicant shall follow the processes as outlined in the CAN-P-1570 *"PALCAN Handbook"* including providing a completed *Assessment Rating Guide*, placing in the right hand column all the appropriate references to their QS Manual, any other Quality System documents and SOP's as well as supplying any requested SOP or other document at least two weeks prior to the on-site re-assessment visit.

This PSA employs designated technical assessors, including those from provincial/federal regulatory agencies, for the assessment of the participating laboratories. These technical assessors have committed to adhere to the standard SCC assessment protocols and rules of confidentiality; however, they may be required by law to report to their own regulatory agency any contravention of the acts and regulations they are duty-bound to enforce.

Accreditation under the PSA-MA specific requirements is the formal recognition by SCC of the competence of a mineral analysis testing laboratory to manage and perform this type of activity. It is not a guarantee that test results will conform to standards or agreements between a testing laboratory and its customers. Business transactions between an accredited testing laboratory and its customers are legal matters between the two parties.

Laboratories are also reminded of the need to comply with any and all relevant statutory or legislative requirements applicable to the jurisdiction in which they operate. With respect to health and safety legislation, this normally requires the establishment of a health and safety committee, or if the laboratory is small, an employee with responsibility for overall safety, as per Section 1.5 of CAN-P-4E.

This document has been approved by the Mineral Analysis Working Group (MAWG), by the Task Group Laboratories (TG Labs) and the Advisory Committee on Conformity Assessment (ACCA) of SCC.

GENERAL AND ADDITIONAL REQUIREMENTS

Laboratories accredited through PALCAN shall meet all requirements in the international standard CAN-P-4E (ISO/IEC 17025:2005) *"General Requirements for the Competence of Testing and Calibration Laboratories"*, these PSA-MA requirements, and Appendices to these requirements to qualify for the SCC Program Specialty Area - Mineral Analysis accreditation. Mineral analysis testing laboratories that do not meet these CAN-P-1579 requirements for their mineral analysis tests will not be accredited by SCC for those mineral analysis tests. The checklist that is used to assess the management and technical requirements of CAN-P-4E and these PSA requirements is the latest version of CAN-P-1510E, *"Assessment Rating Guide"*. The requirements of CAN-P-15 (*"Accreditation Programs: Requirements and Procedures for Suspension and Withdrawal, Complaints, Appeals and Hearings"*) also apply to all SCC accredited laboratories. For information on application and terms and conditions of accreditation, refer to the current edition of the SCC CAN-P-1570 *"PALCAN HANDBOOK Program Requirements for Applicant and Accredited Laboratories"*. If an accredited testing laboratory cannot maintain these requirements, it shall cease any publicity referring to the accredited status for the analysis of sediment, rocks, ores, metal products, tailings, other mineral samples, water and vegetation, and inform SCC in writing within five (5) days. See the termination and withdrawal procedure in the current version of CAN-P-15.

All laboratories shall also meet all the pertinent provisions of the most recent editions of the SCC PALCAN Policy documents (CAN-P-16xx) defined in the normative references.

1 SCOPE

The PSA-MA program for mineral analysis testing laboratories applies to tests associated with the measurement of all media used in mining exploration and processing. This includes, but is not limited to, sediments, rocks, ores, metal products, tailings, other mineral samples, water and vegetation. However, it cannot cover all aspects of mineral analysis testing and shall be regarded as being representative of this area of activity. The specific scope described below was selected because of the market demand. This scope may be modified, depending on market and regulatory requirements.

Proficiency testing (including inter laboratory comparisons (ILCs)) is a demonstration of the ability of the laboratory to produce credible results. It is one of the important tools used by laboratories and accreditation bodies for monitoring test results and for verifying the accreditation process itself. The SCC Policy for participation in proficiency testing schemes is to comply with the general minimum proficiency testing requirements outlined in the ILAC P9:2005 *"ILAC Policy for Participation in National and International Proficiency Testing Activities"* as well as the more specific proficiency testing benchmark participation frequency guidelines for all its accredited mineral analysis laboratories of two (2) proficiency testing rounds/year for the mineral analysis sub-discipline as outlined in the APLAC PT 006:2008 *"Proficiency Testing Frequency Guidelines"*.

The SCC also complies with the responsibilities of an accreditation body for APLAC testing interlaboratory comparisons outlined in the APLAC PT 002:2008 *"Testing Interlaboratory Comparisons"*.

Accreditation in this PSA-MA program requires ongoing continued participation and demonstrated satisfactory performance for each test accredited in the proficiency testing scheme outlined in this document, as well as other proficiency testing programs where appropriate and "fit for purpose".

The SCC will maintain a CAN-P-1579 Appendix A (“*PSA-MA Criteria for Assessment of Proficiency Testing Performance*”) containing a list of the specific proficiency testing measurands in the designated proficiency testing scheme for this PSA-MA program. Applicant and accredited laboratories shall employ this designated proficiency testing scheme for their accredited mineral analysis tests and associated specific measurands in this PSA-MA program.

Multi-measurand methods can be included in the scope even if not all of the measurands are included in the proficiency testing scheme. It is expected that a test method will be validated for all measurands and this validation be documented.

There is also recognition that some testing is not conducive to a formalized proficiency testing scheme and, therefore, other mechanisms, such as in-house proficiency programs, blind splits, ILCs etc., shall be used to evaluate the laboratory performance.

2 NORMATIVE REFERENCES

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

CAN-P-4E (ISO/IEC 17025:2005), General Requirements for the Competence of Testing and Calibration Laboratories. Standards Council of Canada, Ottawa, Ontario, Canada.

CAN-P-15, March 2000, Accreditation Programs: Requirements and Procedures for Suspension and Withdrawal, Complaints, Appeals and Hearings. Standards Council of Canada, Ottawa, Ontario, Canada.

CAN-P- 43 (ISO/IEC Guide 43:1997), November 2001, Proficiency Testing by Interlaboratory Comparisons. Standards Council of Canada, Ottawa, Ontario, Canada.

CAN-P-1510E, Assessment Rating Guide. Standards Council of Canada, Ottawa, Ontario, Canada.

CAN-P-1570 PALCAN Handbook, Program Requirements for Applicant and Accredited Laboratories. Standards Council of Canada, Ottawa, Ontario, Canada.

CAN-P-1623, PALCAN Interpretation and Guidance on the Estimation of Uncertainty of Measurement in Testing (APLAC T005). Standards Council of Canada, Ottawa, Ontario, Canada.

CAN-P-1624, PALCAN Policy on the Use of Proficiency Testing as a Tool for Accreditation in Testing (ILAC G22:2004). Standards Council of Canada, Ottawa, Ontario, Canada.

CAN-P-1625, Policy on Guidelines and Procedures for Laboratories with Serious and Critical Non-Conformities. Standards Council of Canada, Ottawa, Ontario, Canada.

CAN-P-1626, PALCAN Policy on Traceability Requirements for Calibration Sources Used by Accredited Testing Laboratories. Standards Council of Canada, Ottawa, Ontario, Canada.

CAN-P-1627, PALCAN Policy on the Selection of Physical Measurement Calibration Sources for Testing Laboratories. Standards Council of Canada, Ottawa, Ontario, Canada.

CAN-P-1628, PALCAN Policy on the Use of Information Technology in Accredited Laboratories. Standards Council of Canada, Ottawa, Ontario, Canada.

CAN-P-1629, PALCAN Guidance for the Validation of Test Methods. Standards Council of Canada, Ottawa, Ontario, Canada.

CAN-P-1630, PALCAN Interpretations for Conducting Assessments of Testing and Calibration Laboratories. Standards Council of Canada, Ottawa, Ontario, Canada.

CAN-P-1631, PALCAN Guidelines for the Use of Accreditation Body Logos and for Claims of Accreditation Status (ILAC G14:2000). Standards Council of Canada, Ottawa, Ontario, Canada.

PALCAN Policy on samples prepared at Mineral Analysis Laboratory's off-site sample preparation facilities. SCC TG Labs (18.7, 2006) approved and recommended as the PALCAN Policy, effective 1 May 2007.

ILAC P9:2005, ILAC Policy for Participation in National and International Proficiency Testing Activities.

APLAC PT 002:2008, Testing Interlaboratory Comparisons.

APLAC PT 006:2008, Proficiency Testing Frequency Guidelines.

(VIM) 3rd ed.:2007, International Vocabulary of Metrology - Basic and General Concepts and Associated Terms (VIM) (ISO/IEC Guide 99, First edition 2007).

3 TERMS and DEFINITIONS

All definitions in CAN-P-4E and VIM 3rd ed. [e.g. laboratory, testing laboratory, calibration laboratory, calibration, test, calibration method, test method, verification, quality system, quality manual, reference standard, reference material, certified reference material, traceability, proficiency testing, (accreditation) requirements] and those applicable from Guide 43-1, ISO 9000 [e.g. quality assurance, quality control] apply. Some of these definitions are reproduced for convenience in the document CAN-P-1579 Appendix B (*Definitions for the PSA-MA Program*).

To ensure clarity and consistency, for the purposes of this PSA-MA program the definitions in CAN-P-1579 Appendix B (*Definitions for the PSA-MA Program*) shall apply and shall be employed by all mineral analysis testing laboratories accredited under this PSA:

NOTE: there are new or revised definitions for many terms in VIM 3rd Ed. 2007. Laboratories shall update all their Quality System documents to reflect these revised definitions as defined in CAN-P-1579 Appendix B.

4 MANAGEMENT REQUIREMENTS

All the requirements in section 4 of CAN-P-4E and all other relevant CAN-P series documents apply to all accredited laboratories. This section of these requirements is to be used in conjunction with the CAN-P-4E document. The intent of this section is to provide elaboration, interpretation and

additional requirements to some of the clauses of CAN-P-4E for which procedures are specifically applicable to mineral analysis testing will be used. **Some sub clause numbering will be unique to this section.** The following section numbers correspond directly to the clauses in CAN-P-4E.

4.2 Quality system

4.2.5 Documentation shall be maintained and include or make reference to the following:

- All test methods and standard operating procedures
- Protocols for method development and validation
- Chain of custody
- Quality assurance, audit records (internal and external) and proficiency testing as applied to each scope of testing

4.3 Document control

4.3.1 The laboratory shall establish and maintain policies and procedures to document the responsibility for all procedures performed (internal and external), how these procedures are monitored and when corrective actions are taken.

NOTE: see also CAN-P-1630 interpretative note.

4.4 Review of Requests, Tenders and Contracts

4.4.6 Laboratories that, as part of their accredited test(s), report test result(s) with respect to the parent sample from “representative” prepared samples or sub-samples received from an off-site physical sample preparation facility shall in contracts with their customers specify the crushed top particle size and pulverized pass criteria as required by section 5 a) & b) of the “*PALCAN Policy on samples prepared at Mineral Analysis Laboratory’s off-site sample preparation facilities*”.

4.6 Purchasing Services and Supplies

4.6.2.1 New reagents and standards shall be verified against old ones or verified by other means (ex. CRM, analytical QC etc), and records maintained.

4.6.4.1 List and records of investigation of all approved suppliers shall include subcontractors.

4.13 Control of Records

4.13.1 Technical records shall include reagent preparation logs. Reagent preparation logs shall include, as appropriate: supplier, grade, batch number; dates of preparation or verification; analyst preparing the reagent, measurement of weights, volumes, time intervals, temperatures and related calculations; relevant processes (e.g. pH adjustment, etc); verification results; and, discard or expiry date.

4.13.2.1

a) The laboratory shall have documented procedures to ensure that it maintains a coordinated record keeping system for its technical records. The information that is to be included shall be documented and may include items such as records of telephone conversations, evidence receipts, descriptions of evidence packaging and seals, subpoenas, records of observations and

test/examination results, reference to procedures used, diagrams, print-outs, photographs, etc. In general, the records required to support the technical data shall be such that in the absence of the analyst, another competent analyst could evaluate what had been performed and interpret the data.

b) Where instrumental analyses are conducted, operating parameters shall be appropriately recorded.

c) Where appropriate, all observations or test results shall be preserved. Electronic records, photocopies, tracings or hand-drawn facsimiles shall also be preserved (e.g. tape or CD/DVD backup of electronic files).

d) When a test result or observation is rejected, the reason(s) shall be recorded.

e) Test results, calculations and manual data transfers or electronic transfers, excluding those that form part of a validated electronic process, shall be checked by at least a second person. The record shall include an indication when the results and any corrective actions needed were performed and when such checks have been carried out and by whom.

f) Each document in the record shall be traceable to the analyst and where appropriate, to a uniquely identified laboratory number. It shall be clear from the record who has performed all stages of the analysis/examination and when each stage of the analysis/examination was performed (e.g. relevant date(s)).

g) Laboratory generated examination records and reports shall be paginated using a page numbering system which indicates the total number of pages.

h) The laboratory shall have documented policies and procedures for the review of records, including test reports.

i) Where independent checks on non-conformances are carried out by other authorized personnel, the records shall indicate when each non-conformance has been checked and agreed and by whom the checks were performed. This may be indicated in a number of ways including entries against each finding, entry on a summary of findings etc.

4.14 Internal Audits

4.14.1 The SCC policy requires that internal audits shall be conducted at least on an annual basis. Every part of their quality system shall be audited annually (including a representative sampling of test methods); however, it is not necessary to audit each person or each testing /measurement procedure, or to audit every aspect at one time.

4.15 Management Reviews

4.15.1 The SCC policy requires that management reviews shall be conducted at least annually even though the wording of the standard might appear to allow for a longer periodicity. Management reviews are often a series of events/meetings that percolate upwards.

5 TECHNICAL REQUIREMENTS

All the requirements in section 5 of CAN-P-4E and all other relevant CAN-P-series documents apply to all accredited laboratories. This section of these requirements is to be used in conjunction with the CAN-P-4E document. The intent of this section is to provide elaboration, interpretation and additional requirements to some clauses of CAN-P-4E for which procedures are specifically applicable to mineral analysis testing. The following section numbers correspond directly to the clauses in CAN-P-4E. **Some sub clause numbering will be unique to this section.**

5.2 Personnel

5.2.1 The laboratory shall have a defined policy that ensures that all staff working in the laboratory are competent to perform the work required. The term 'competent' implies demonstrating the requisite knowledge, skills and abilities to perform the job. The laboratory's policy shall also include procedures for documenting training, retraining and maintenance of skills, expertise and demonstrated on-going competence.

Where test or technique specific training is given, acceptance criteria shall be assigned. Whenever possible, satisfactory performance in the analysis of quality control/quality assurance samples or correlation of results with those obtained by other trained staff shall be used. The observation of the relevant tests or analyses by an experienced officer is acceptable where a quantitative assessment is not possible. The appropriate sign off shall be recorded.

Qualifications generally required in a mineral analysis testing laboratory are as follows:

- key supervisors: appropriate degree, diploma, or equivalent and at least 3 years laboratory experience
- analysts: appropriate technical diploma or equivalent and variable years laboratory experience depending on technical complexity of duties, which is relevant to the test(s) being accredited

5.2.2 The laboratory shall maintain personnel training and qualification records and certificates.

In addition to the above criterion, some provinces may have additional legislated requirements. The Quality Manual or other QS documents shall reflect these requirements.

5.2.5 A laboratory shall have clear statements of the competencies required for all jobs and records shall be maintained to demonstrate that all staff is competent for the jobs they are asked to perform.

Records of demonstrated competence are analogous to documented evidence of analyst proficiency.

Laboratories shall have a formal policy of cross training staff so that each task can be assigned to a second staff member should the primary staff to which the task has been assigned be unable to perform their duties.

Each laboratory or section shall maintain an up-to-date record of the training that each member of staff has received. These records shall include academic and professional qualifications, external or internal courses attended and relevant training (and retraining, where necessary) received whilst working in the laboratory.

Records shall be sufficiently detailed to provide evidence that staff performing particular tasks have been properly trained and that their subsequent ability to perform these tests has been formally assessed.

5.3 Accommodation and environmental conditions

5.3.1 Accommodations and environmental conditions will depend on the type and volume of work being performed, and may include (as required):

- adequate lighting at work areas
- adequate power
- sufficient appropriately grounded outlets which are free of surges and have voltage regulators in use
- back-up emergency power supply available
- sufficient sinks with hot and cold running water
- suitable reagent water supply
- air supply free of dust, fumes and oil and suitable for sample aeration and/or purging
- vacuum source is able to maintain sufficient vacuum
- bench tops are adequate
- adequate bench space
- adequate floor area
- temperature is controlled in specific laboratory areas as required
- humidity control in specific laboratory areas as required
- appropriate air quality in specific laboratory areas as required; otherwise ensure the laboratory is well ventilated (once through ventilation, where appropriate) and have controls in place to limit exposure to dust and/or fumes
- sufficient fume hoods, able to maintain appropriate face velocity
- appropriate refrigerated storage, including freezer storage, available for samples and other materials
- measures to avoid cross contamination in areas in which trace levels of contaminants in the work environment are evaluated and analyzed

5.3.2 The laboratory shall have procedures for monitoring, controlling and recording environmental conditions where applicable, such as:

- acceptable lighting
- cleanliness of the sample preparation work area
- replenishment of consumables used in reagent water and/or dilution water treatment
- water quality characteristics as required, especially conductivity on a daily or as used basis and corrective actions taken for non-conformance
- temperature
- humidity
- storage temperatures and corrective actions taken for non-conformance

5.3.3 Special care is needed in mineral analysis testing laboratories involved in the analysis or determination of trace levels of minerals. Physical separation of high-level and low-level work is required. Where special areas are set aside for this type of work, access to these areas shall be restricted and the work undertaken carefully controlled. Appropriate records shall be kept to demonstrate this control. It may also be necessary to carry out 'environmental monitoring' of equipment, work areas, clothing and consumables.

5.3.4 Access to the operational area of the laboratory (including the office and areas where records are stored) shall be controllable and limited. Visitors shall not have unrestricted access to the operational areas of the laboratory. A record shall be retained of all visitors to the operational areas of the laboratory.

5.3.5 Procedures shall be in place to ensure that the use of materials used in cleaning and/or pest control do not cause interference with testing.

5.4 Test and calibration methods and method validation

5.4.1 All methods shall be fully documented including procedures for quality control (which includes the use of reference materials).

5.4.2

a) All laboratory developed methods or methods adopted by the mineral analysis laboratory shall be fully validated or verified by the laboratory for “fitness of purpose” before being used on customer samples. This validation or verification shall be fully documented by the appropriate procedures as defined in CAN-P-1629.

b) Where a laboratory introduces a new validated method, it shall first demonstrate the ability to adequately perform the method against any documented performance characteristics of that procedure. All method validation and verification records shall be maintained for future reference.

c) Laboratories shall institute a procedure to identify infrequently performed tests or analyses (i.e. where the test is not performed for > 6 months). For these tests or analyses, there are methods of demonstrating competence. These include but are not limited to the following:

- i. regular analysis of control samples and use of control charts even when ‘real’ samples are not being analyzed
- ii. before the test or analysis in question is performed on a real sample re-verification involving at least the use of an appropriate reference material, followed by replicate testing or analysis of the real sample
- iii. continued demonstrated satisfactory performance in the proficiency testing scheme(s)

d) The quality of standard materials and reagents shall be adequate for the procedure used. Lot/batch numbers of standard materials and critical reagents shall be recorded. All critical reagents shall be tested for their reliability. The reagent preparation logbook shall record the identity of the preparer. Standard materials and reagents shall be labelled with:

- name
- concentration, where appropriate
- received date as well as preparation date and expiry date (if necessary)
- identity of preparer
- storage conditions, if relevant
- hazard warning, where necessary

NOTE: see also CAN-P-1630 interpretative note.

5.4.5 Validation of methods

NOTE: The Eurachem Guide “*Fitness for Purpose of Analytical Methods - A Laboratory Guide to Method Validation and Related Topics*” document is a very valuable resource for method validations. In addition to using that guide, the following are additional requirements to ensure all elements are considered in validating methods.

5.4.5.2 All technical procedures and laboratory methods used by a mineral analysis laboratory for measurand determinations, whether modified even slightly or applied outside its intended scope (i.e. applied to another matrix) from national/ international methods (5.4.2) or in-house methods (5.4.3, 5.4.4) shall be validated, or verified by the laboratory, for “fitness of purpose” prior to implementation. This validation or verification shall be fully documented by the appropriate procedures and at a minimum meet the requirements in CAN-P-1629.

Methods may be validated or verified by comparison with other established methods using certified reference materials (where available) or materials of known characteristics.

In validating quantitative test methods, the following issues (among others) shall be documented, as appropriate:

- matrix effects
- sample homogeneity
- specificity
- sensitivity
- detection limit
- limit of quantitation
- accuracy
- repeatability
- robustness
- bias
- intra laboratory variations
- analysis of reference materials
- interferences
- concentration ranges
- long term stability of measured compounds
- cross sensitivity
- reporting limit
- linearity range
- precision (including intermediate precision)
- reproducibility
- trueness
- measurement of uncertainty
- inter laboratory variations
- recovery studies

a) Evidence of measurand determinations, its separation from interfering substances and the applicability of the method for measuring the measurand in the particular matrix, consistent with the required reporting limits, shall be demonstrated. Methodology shall also provide documented acceptable accuracy and precision.

b) The validation of a method is only applicable to the methodology as written and any variation in procedure, analyst, instrumentation (ex addition of a new instrument) or application is subject to revalidation. A number of techniques may be used for determining trueness, a part of method validation, including comparison with a recognized method, the analysis of a certified reference material (CRM), comparison of results with a second laboratory or another reliable demonstration of method validity as might be available.

NOTE: Changes in conditions that have been shown through ruggedness testing to not have a significant effect on the results of a method (e.g. different operator or instrument) can be made without having to revalidate a method. Those specific conditions shall be clearly identified in the method.

c) The extent of validation data required prior to using a method routinely will depend on the type and the purpose of the method and the performance related documentation already available for the particular method. All requirements will be documented in the Quality Manual, individual method or covered in a specific laboratory wide SOP and at a minimum meet the requirements in CAN-P-1629.

d) For most methods used in the mineral analysis laboratory, the absence of significant interferences (cross sensitivity) shall be demonstrated by running matrix and reagent blanks during the validation process, if applicable.

e) If CRM analyses suggest a bias then the laboratory shall check all steps to isolate the problem and take corrective action.

f) During the validation or verification of a method the analytical range is assessed by using calibration standards covering at least the minimum range of expected sample results but preferably encompassing the orders of magnitude characteristic of the instrument. The sensitivity of the method, defined as the detector response per unit measurand concentration, is given by the slope of the calibration curve. Examination of this calibration curve will demonstrate the number and concentrations of the standards acceptable for routine analysis. Acceptability will depend upon linearity and intercept values as well as the overall shape of the calibration curve.

All technical procedures and laboratory methods used by a mineral analysis laboratory for measurand determinations, once validated or verified, shall have the following prior to implementation:

- i. A formal, complete, approved “**Method Validation Summary Report**” document (however named). This Method Validation Summary Report shall include a clear definition of the method/purpose. It shall also contain a clear list of the method validation definitions and, at a minimum, all the summary data for the appropriate or pertinent items in 5.4.5.2. It shall also contain a statement signed by the appropriate technical staff that this laboratory method is “fit for purpose” prior to implementation.
- ii. All resulting values obtained from the method validation or verification data shall be clearly reflected (listed) in the specific analytical method documentation.
- iii. The Quality Control for a particular analysis shall be based on the method validation data. (Eurachem guide Section 8.)

All technical procedures and laboratory methods used by a mineral analysis laboratory for measure and determinations, once validated or verified, shall be periodically reviewed (at least once every two years) to confirm the ongoing “fitness for purpose” of that procedure or method.

5.4.5.3

a) The detection limit (LOD) is the lowest concentration of measurand in a real sample matrix that can be reliably detected using a specific analytical procedure (test method) which is statistically different from the response obtained from a reagent blank carried through the complete method. When repeated analyses of reagent blanks show a positive response for the measurand, the LOD is defined as: $LOD = S_b + 3 S.D.$ where S_b is the average signal for the reagent blanks and S.D. is the standard deviation of the blanks. If the reagent blanks do not show a positive response for the measure and, the S.D. is obtained from replicate analysis ($n=7$ or more) of a typical sample spiked at a level within 2 to 3 times the estimated LOD.

b) The continuing improvement in technology has made available methodology that can measure smaller and smaller element concentrations in different samples. However, the detection of very low levels may not always be essential and it may be acceptable in some circumstances to define a "practical" reporting limit based on customer requirements and the proposed use of the analytical data. This would have the advantage of reducing the technical difficulty of obtaining data and of reducing costs.

5.4.6 Estimation of uncertainty of measurement

5.4.6 Laboratories shall demonstrate implemented use of adequate procedures consistent with GUM (and its' supplemental document ISO/IEC Guide 98-1) as well as the CITAC/Eurachem Guide CG4 (QUAM:2000) for estimation of the uncertainty of measurement associated with all accredited tests.

Irrespective of the type of testing, the laboratory shall identify the significant components of measurement uncertainty.

For quantitative tests, numerical estimates are expected for those tests which produce numerical results. At a minimum, this shall include the calculations for standard uncertainty, combined standard uncertainty and expanded uncertainty (normally at a coverage factor of $k=2$).

NOTE: The definitions for standard uncertainty, combined standard uncertainty and expanded uncertainty shall be those defined in VIM 3rd Ed: 2007 (which are the definitions employed by GUM and QUAM). Refer to CAN-P-1579 Appendix B.

5.6 Measurement Traceability

All the requirements for traditional measuring and testing equipment, where applicable, shall be met. The equipment shall receive adequate calibration and have valid measurement traceability on critical equipment as defined in CAN-P-1626.

NOTE: see also CAN-P-1626 note 4.2 for the definition for critical equipment.

5.6.1 Individual calibration programs shall be documented and controlled for the specific requirements of the testing or analytical work being carried out. It will normally be necessary to check instrument calibration after any shut down, whether deliberate or otherwise and following service or other substantial maintenance. In general, calibration intervals should not be less stringent than manufacturers' recommendations.

5.6.2 Items that laboratories need to consider ensuring conformance to measurement traceability shall include the following:

- the availability of Class S or Class 1 weights used for balance calibrations; laboratories shall have traceability for the item in its possession
- the accuracy of volumetric measurements by using Class A glassware, where appropriate
- the availability of a thermometer, traceable as per CAN-P-1626 used for calibrations; laboratories shall have traceability for the item in its possession
- certificates for certified weights and thermometers maintained on file
- certificates for reference materials, standards or reagents used in preparing reference materials or standards (e.g., certified reference materials and calibration standards) maintained on file

5.6.2.2.1 Method calibration procedures shall include, as appropriate: use of a reagent blank to establish a calibration baseline; use of equivalent standard/sample reagent background; use of an adequate number of standards; establishment of linearity and calculation of slope and/or RRF; use of a control standard to monitor calibration stability/accuracy; use of control charting; and, identification of calibration non-conformance criteria.

5.6.3 Reference and calibration materials or standards of stated purity will be obtained from a reliable source as outlined in CAN-P-1626 and 1627. These materials or standards shall be traceable to national or international sources. If such materials or standards are not certified, the laboratory will develop procedures for verifying their purity and identity as outlined in CAN-P-1627.

Reference and calibration materials or standards and their documentation shall be stored in such a way as to maintain their integrity and be labelled as to content, date received, date prepared or opened, analyst's initials and expiration date. The reagent preparation logbook shall include the analyst's initials or name. These materials or standards shall be replaced at appropriate intervals depending upon stability and storage conditions. To maintain their traceability, they shall not be used after the expiry date specified by the supplier and they shall meet the conditions specified in CAN-P-1627. If these materials or standards have expired they may be used as QC material or in-house reference materials.

In-house reference materials can be made traceable to Certified Reference Materials by running them along side of each other and documenting the results providing the procedures in CAN-P-1626 and 1627 are followed. This shall be repeated with a frequency that will be determined by the stability of the reference materials or standards. Acceptable uncertainty will be documented in the Quality Manual, method documentation or SOP.

Documentation allowing all dilutions to be traced to the primary reference material or standards shall be maintained.

5.7 Sampling

The laboratory shall monitor the reliability of its sampling of submitted samples to ensure any sub-sample taken (e.g. from a crushed rock split) is reliably and demonstrably representative of the original sample submitted. This shall be documented in the quality documentation and acceptable limits defined, controlled and maintained.

5.7.4 Laboratories that, as part of their accredited test(s), report test result(s) with respect to the parent sample from "representative" prepared samples or sub-samples received from an off-site physical sample preparation facility shall have policies and procedures to ensure adherence to the "*PALCAN Policy on samples prepared at Mineral Analysis Laboratory's off-site sample preparation facilities*".

5.8 Handling of Test and Calibration Items

5.8.3 The laboratory shall ensure any abnormalities and deficiencies are recorded upon receipt of the sample. Abnormalities and deficiencies may include:

- damaged sample
- insufficient sample for analysis
- deficiencies related to field filtration, chemical preservation, sample container, temperature on arrival, exclusion of air, elapsed time subsequent to sampling

5.8.4 The laboratory shall have appropriate facilities and environmental conditions to protect the integrity of the sample once the sample is received at the laboratory.

The laboratory shall follow any customer or regulatory directives to ensure sample integrity is maintained.

5.9 Assuring the quality of tests and calibration results

Accreditation by SCC in this PSA requires the laboratory to demonstrate competence with these requirements by continued participation and satisfactory performance for each test accredited in the proficiency testing scheme outlined in clause 6 of this document, as well as other proficiency testing programs as appropriate.

Unsatisfactory results shall be followed up with an investigation and if necessary corrective action(s). See also CAN-P-1630.

5.9.1 Records of instrument calibration and performance parameters shall be maintained. The records shall clearly indicate the calibration data that is associated with the specific samples analyzed. Appropriate quality control procedures shall include, but not be limited to:

- appropriate level of quality control effort (i.e., duplicate samples, replicate samples, replicate tests reference materials, measurand spikes, method blanks and control samples)
- use of control charting and the analysis of these charts such that short and long term trends are detected
- identification of non-conformance in method performance
- participation in proficiency testing
- and/or analysis of independently prepared check samples

The laboratory shall conduct regular reviews of their control chart data for all measurands to evaluate the appropriateness of the established mean and SD. The laboratory shall update those mean and SD values as appropriate.

5.9.2 Whenever possible in the quality control system, compliance with statistical control shall be monitored through techniques such as control charting such that long term trends are detectable. The results of quality control analyses (e.g. Certified Reference Materials, duplicate samples, replicate samples, replicate tests) are indicators of the performance of the analytical system and their interpretation depends partly on the concept of statistical control. Statistical control corresponds to stability of operation. Specifically, it implies that quality control results can be interpreted as arising from a normal population with mean μ and variance σ^2 .

The range of quality control activities expected to be incorporated into mineral analysis testing protocols on a routine basis includes the use of:

- reference collections of previously analyzed samples or reference materials
- certified reference materials and internally generated reference materials
- positive and negative controls
- control charting of reference material results
- duplicate sampling and analysis
- replicate sampling and analysis
- replicate tests
- range control charting of duplicate sample and replicate sample analysis results

- independent checks (verification) by other authorized personnel
- independent checks on commercial calibration solutions
- participation in proficiency testing schemes

5.9.3 Every analytical batch shall be accompanied by quality control measures that demonstrate the analytical system control status (e.g. determinations on quality control samples, water quality, balance tolerances, furnace temperatures). The ISO 7870 and ISO 7873 documents include information on the design and implementation of control charting.

5.9.4 Reagent blanks will be run with each set of samples and will represent at least 5% of the samples analyzed. However, in instances where a large number of samples of a given commodity require analysis, the frequency of matrix blanks could be significantly reduced if, after analysing a number of samples, most (greater than 90%) are negative. Under these circumstances, the samples serve as adequate blanks.

5.9.5 Reagent quality shall be monitored. Reagents are to be purchased, where possible, from ISO 9000 certified suppliers that provide reagents of required quality. Laboratories shall test each lot or batch and compare the results to the previous analysis. Records are to be kept as to which samples are run using each batch.

5.9.6 Laboratory reagent grade water shall be tested, monitored, controlled and these results shall be documented. It is required that each data set collected shall be related to the appropriate water quality data for the period of time that the test certificates are required to meet traceability requirements.

5.10 Reporting the results

5.10.2 Results shall be reported, usually in a test report, and shall include all the information requested by the customer and necessary for the interpretation of the test result and all information required by the test method used.

The requirements for reporting test results to customers shall apply not only to hard copy reports but also electronic reporting of results by such methods as Excel spreadsheets, e-reporting, database files, web-based reporting etc., as appropriate.

Certificates of Analysis and/or test reports shall be signed by the authorized personnel as described in the Quality Manual and other quality documents, where appropriate. Certificates of Analysis are often used for legal purposes. As such, information contained in these reports is directed by the appropriate laws of the land.

(b) The laboratory shall be able to track the location at which the test was carried out, if tests were carried out at different locations. The laboratory shall put this information on the test report. The location and identity of subcontractors does not need to be identified on the test report.

(e) The laboratory shall have the capability to provide the identification of the test method and shall place this information on the test report.

(g) The laboratory shall be able to trace the date of analysis and shall include it on a test report.

(i) Test reports shall contain the test result, with units. Appropriate significant digits shall be used in reported results.

(j) The test report shall include at least the name of the person authorizing the report. The actual signature of the person authorizing the report need not be on the report, but shall be maintained on file. An electronic signature is sufficient, provided that the laboratory has procedures in place to guard against improper use of the electronic signature.

5.10.2.1 Incorrect quantifications or identifications on any customer sample(s) are unacceptable. The laboratory is obliged to notify the customer and implement corrective action(s) as outlined in CAN-P-4E (ISO/IEC 17025:2005).

5.10.3 Test reports shall include information necessary for the interpretation of results, such as:

- flags when data is reported below the detection limit (or other specified limit)
- flags when a result is qualified due to a non-conformance related to test method variance, sample history, method performance, interference or data validation
- flags when there is no result due to damaged or insufficient sample
- maximum allowable concentrations or standards

5.10.6 Test reports shall identify tests that were subcontracted but laboratories are not required to identify the subcontractor on the test report.

6 EVALUATION OF LABORATORY PERFORMANCE BY PROFICIENCY TESTING

All the requirements in CAN-P-4E and all other relevant CAN-P series documents apply to all accredited laboratories. This section contains the specific requirements for the evaluation of mineral analysis laboratory performance by proficiency testing. It is unique to this document and it provides the elaboration, interpretation and additional requirements for some of the clauses of CAN-P-4E for which proficiency testing procedures specifically applicable to mineral analysis testing shall be applied.

The SCC Policy for participation in proficiency testing schemes is to comply with the general minimum proficiency testing requirements outlined in the ILAC P9:2005 "*ILAC Policy for Participation in National and International Proficiency Testing Activities*" as well as the more specific proficiency testing benchmark participation frequency guidelines for all its' accredited mineral analysis laboratories of two (2) proficiency testing rounds/year for the mineral analysis sub-discipline as outlined in the APLAC PT 006:2008 "*Proficiency Testing Frequency Guidelines*".

The SCC also complies with the responsibilities of an accreditation body for APLAC testing interlaboratory comparisons outlined in the APLAC PT 002:2008 "*Testing Interlaboratory Comparisons*".

6.1 General Criteria for Proficiency Testing

6.1.1 Accreditation in this PSA-MA program requires ongoing continued participation and demonstrated satisfactory performance in the designated proficiency testing scheme for all mineral analysis tests appearing in the laboratory's Scope of Accreditation.

It is recognized that for some specialized tests the designated proficiency testing scheme does not provide measurands/samples and, therefore, other mechanisms, such as in-house proficiency programs, blind splits, ILCs etc., shall be used to evaluate the laboratory performance.

6.1.2 The SCC MAWG, in consultation with SCC, has designated CANMET-MMSL to manage the CAN-P-1579 Proficiency Testing Scheme in accordance with SCC CAN-P-1593 requirements, ISO/IEC Guide 43, ILAC Guide 13 and these requirements. That SCC accredited Proficiency Testing Provider will recommend a proficiency testing coordinator to the SCC MAWG. The SCC MAWG will recommend the proficiency testing coordinator to the TG Labs.

6.1.3 All procedures associated with the handling and testing of proficiency testing samples (items) by the laboratory shall be carried out to the greatest extent possible in a manner identical to routine method(s) of testing that applied to customer samples.

6.1.4 Laboratories shall analyze the proficiency testing samples using the test method listed in their Scope of Accreditation. If their scope contains more than one (1) accredited test method or analytical technique for the same measurand (e.g. zinc in sediment by AD2/FAA and also by AD3/ICPE and/or by AD3/ICP-MS) then each accredited test shall have its own proficiency testing result.

6.1.5 If the proficiency testing sample concentration for any measurand falls below the test method detection limit the laboratory shall clearly indicate that to the proficiency testing coordinator by reporting either a "<" or "< DL" for that specific measurand/test method in that specific proficiency testing sample (refer to section 6.3.2.4).

6.2 Proficiency Testing Requirements

6.2.1 Prior to becoming accredited

6.2.1 Prior to becoming accredited, a laboratory shall successfully complete three (3) proficiency testing cycles for each test which accreditation is requested. Analysis shall be completed and results reported within the time period specified by the proficiency testing coordinator after receipt of the proficiency testing cycle of samples by the laboratory. A laboratory that fails the first set may be provided with a replacement set [having different concentrations for the previous sets], after corrective action has been taken. If the replacement set is not analyzed satisfactorily, further corrective action shall be taken, but a third set of samples [having different concentrations for all previous sets], will not be sent for at least 6 months.

6.2.2 The "Proficiency Testing Scheme"

Once accredited, in order to remain accredited, the laboratory shall maintain demonstrated satisfactory performance in the designated proficiency testing scheme, for each accredited test on their scope.

6.2.2.1 This requires:

- a) participation in a minimum of two (2) proficiency testing rounds annually for each accredited test
- b) each proficiency testing round contains sample set(s) generally consisting of four (4) samples. These sample sets will generally have four (4) [except for Pt and Pd which should generally have two (2)] different concentrations spanning the normal target concentration range outlined in the current version of CAN-P-1579 Appendix A ("*PSA-MA Criteria for Assessment of Proficiency Testing Performance*")

c) there will generally be eight (8) proficiency testing results per year required per accredited measurand/test.

NOTE: Laboratories are encouraged to actively participate in other acceptable proficiency testing schemes from other Proficiency Testing Providers where they are appropriate and “fit for purpose”. In general these proficiency testing schemes should adhere to the principles of ISO Guide 43 and ILAC Guide 13.

6.2.2.2 To pass a set of quantitative proficiency testing samples, laboratories shall correctly report, for each accredited test, the measurand result along with the specific test method employed within the timelines outlined by the proficiency testing coordinator.

6.2.2.3 Proficiency testing results are due within the time period specified by the proficiency testing coordinator after receipt of proficiency testing samples by the laboratory. Laboratories not reporting results on time will be subject to the suspension procedure described in section 6.4 (Procedures for unsatisfactory laboratory performance).

6.3 Proficiency Testing Program Responsibilities

6.3.1 Laboratory’s responsibilities

6.3.1.1 The laboratory’s general responsibilities shall include the following:

- i) identifying the methodology for each measurand
- ii) reporting one result on the measurand per each proficiency testing sample for each test method for requested/ongoing accreditation (refer to 6.1.4)
- iii) reporting results to at least 2 significant figures

6.3.1.2 Laboratories shall declare their test method detection limit for each measurand of every accredited test method to the proficiency testing coordinator (refer to section 6.1.5).

6.3.1.3 The laboratory may lose one sample only once in a 3 year period. If this occurs more than once then the test method(s) shall be suspended until the corrective action is investigated and corrected

6.3.1.4 The laboratory shall, under normal circumstances, submit 8 results on each measurand (a < or < DL is a valid result) for each test method/relevant proficiency testing sample combination over the 2 proficiency testing cycles. However, if unforeseen circumstances occur, they shall submit a minimum of 4 results for 2 proficiency testing cycles - i.e. 4 results on each measure and for each test method over 2 cycles/year.

6.3.1.5 Laboratories shall provide the SCC MAWG Secretary (with a copy to the proficiency testing coordinator), within 10 working days from the first date of receiving the preliminary “Performance Report” (in what ever format first reported to the laboratory) from the proficiency testing provider, an initial corrective action report (CAR) for any unsatisfactory measurand result. This shall be done by electronically and the laboratory shall immediately initiate corrective action(s) (see 6.4.5)

Laboratories failing to accurately report their performance in any proficiency testing round within this timeline shall be immediately subject to suspension outlined in clause 6.4.6

6.3.1.6 Laboratories shall keep all proficiency testing provider final “Comprehensive Report” documents (however named) relating to their scope under this PSA-MA for a minimum of 3 years. They shall provide copies of any preliminary “Performance Report” or final “Comprehensive Report” to the SCC when requested by the SCC.

6.3.2 Proficiency Testing Provider’s responsibilities

6.3.2.1 The proficiency testing provider shall make available on request from participants or applicant laboratories a document that describes the proficiency testing scheme, its operations and requirements, including the time lines for submission of results by participants, the issuing of reports on proficiency, the measurands and concentration ranges.

6.3.2.2 The proficiency testing provider shall employ the “z-score” method (ISO/IEC Guide 43 and ISO 13528) for the evaluation of laboratory performance for all quantitative tests.

a) The performance on each single measurand result calculation of the z-score shall be as follows:

$$z = \frac{x - X}{\sigma_{pt}}$$

where:

- the quantity $(x - X)$ is called the “estimate of laboratory bias” in ISO 13528
- “x” is the participant’s result
- “X” is the “assigned value” (i.e. the consensus value which is the consensus proficiency testing mean)
- σ_{pt} is the “standard deviation for proficiency assessment”, an appropriate measure of variability which is selected to meet the requirements of the proficiency testing scheme. For this PSA-MA the σ_{pt} shall be the traditional standard deviation after removal of all outliers.

b) In order to avoid undue emphasis on an isolated poor result, z-scores greater than +3 or less than -3 will be assigned values of +3 and -3 respectively.

6.3.2.3 The proficiency testing coordinator shall establish the “assigned value” as follows:

a) Providing there is a minimum of 10 laboratories after the removal of outliers as described by the proficiency testing coordinator, the mean value of the results from all remaining laboratories in a particular round will be used as the assigned value. Participating laboratories shall report results to at least 2 significant figures.

NOTE: The procedure for the removal of outliers used by the proficiency testing coordinator shall be approved by SCC MAWG. The method(s) will be consistent with ISO 13528 and may employ appropriate test(s) such as Grubbs or standard T test (ASTM E178-80)

b) When there are fewer than 10 laboratories after the removal of outliers, one of the other techniques in ISO/IEC Guide 43 will be used to determine the assigned value. This will be documented by the proficiency testing coordinator.

6.3.2.4 All test results that are reported by the laboratory as lower than that laboratory's declared detection limit shall be treated as "non-detects". The proficiency testing provider shall not assign a z-score for those "non-detect" specific test results and the proficiency testing provider shall report those results as either "<" or "< DL".

6.3.2.5 The proficiency testing coordinator shall evaluate each laboratory's overall performance by employing the interpretation of the z-score for each measurand as follows (ISO/IEC Guide 43):

$ z \leq 2.0$	"Satisfactory" performance
$2.0 < z < 3.0$	"Questionable" performance
$ z \geq 3.0$	"Unsatisfactory" performance

6.3.2.6 The proficiency testing coordinator shall calculate the RSZ and SSZ scores as follows:

Individual z-scores for each laboratory shall be combined within each proficiency testing cycle to produce a Re-scaled Sum of Scores (RSZ) and a Squared Sum of z-Scores (SSZ) such that:

- RSZ is calculated as: $\sum z / \sqrt{n}$ (n = number of z-scores being combined)
- SSZ is calculated as: $\sum z^2$

6.3.2.7 The proficiency testing coordinator shall evaluate each laboratory's overall performance for RSZ and SSZ by employing the acceptability criteria outlined in the current version of CAN-P-1579 Appendix A ("*PSA-MA Criteria for Assessment of Proficiency Testing Performance*").

6.3.2.8 The proficiency testing provider shall issue to the laboratory a preliminary "Performance Report" (however named) within the time period specified by the proficiency testing coordinator after receipt of all laboratory reports along with any other pertinent information for each sample as outlined by the SCC MAWG.

6.3.2.9 The proficiency testing provider shall issue a final "Comprehensive Report" (however named) within the time period specified by the proficiency testing coordinator. This final "Comprehensive Report" shall detail each laboratory's specific performance evaluation results the laboratory received for the individual measurands as well as general performance evaluation information for all participants. This final report shall contain the following minimum information:

- a) "x" - the participant's result
- b) "X" - the "assigned value"
- c) σ - the standard deviation
- d) z-scores and criteria
- e) overall RSZ and SSZ scores and criteria
- f) the result of quantitative validation assessments

6.3.2.10 The proficiency testing coordinator shall provide copies of the "Performance Report" and the final "Comprehensive Report" and date of delivery of these documents to the SCC MAWG Convener and Secretary.

NOTE: The proficiency testing coordinator has no responsibility for monitoring the laboratory's proficiency testing performance as related to maintenance of SCC accreditation. This is the sole responsibility of the SCC MAWG proficiency testing sub-committee.

6.3.3 SCC responsibilities

6.3.3.1 The SCC MAWG criteria employed for the evaluation of performance in quantitative test results shall be the interpretation of the z-score for each measurand as follows (ISO/IEC Guide 43):

$ z \leq 2.0$	“Satisfactory” performance
$2.0 < z < 3.0$	“Questionable” performance
$ z \geq 3.0$	“Unsatisfactory” performance

NOTE: As outlined in section 6.3.2 above, in general the number of participants the laboratory is evaluated against shall be ≥ 10 .

6.3.3.2 The specific SCC MAWG acceptability criteria used for evaluating each laboratory’s overall proficiency testing performance are outlined in the current version of CAN-P-1579 Appendix A (“PSA-MA Criteria for Assessment of Proficiency Testing Performance”) and have been approved by the SCC MAWG.

6.3.3.3 Any appeal by a laboratory regarding the assessment of reported results by the proficiency testing coordinator will be administered by SCC through its MAWG, as explained in CAN-P-15.

6.4 Procedures for unsatisfactory laboratory performance

6.4.1 Failure of a laboratory to comply with any aspect of accreditation requirements including these requirements, may lead to suspension or withdrawal of accreditation in accordance with the standard SCC suspension and withdrawal procedures documented in CAN-P-15. In addition, the laboratory will be subject to the suspension procedures below, when its mineral analysis testing performance does not meet the specified performance criteria outlined in sub-section 6.3.3 above.

All instances of withdrawal of accreditation will be publicized by the SCC on their website. When suspension action is taken, customers, potential customers and the public will be notified by the posting of a Suspension Notice on the SCC website. In the case of accredited laboratories, this notice shall take the form of an amended scope of accreditation.

6.4.2 SCC will consider several factors in determining whether the suspension of a test from the scope of an applicant laboratory or withdrawal of accreditation of an accredited laboratory is necessary:

- a) unsatisfactory performance or failure to participate in a proficiency testing round
- b) failure to take immediate corrective action(s) on unsatisfactory proficiency testing performance(s)
- c) failure to properly correct the unsatisfactory proficiency testing performance(s) in a timely manner
- d) failure to report proficiency testing results within the timeline outlined by the proficiency testing provider

e) failure to report unsatisfactory proficiency testing results to the SCC MAWG Secretary within the timelines outlined in section 6.3.1.5

f) failure to accurately report their performance in any proficiency testing round

6.4.3 Failure of a laboratory to participate in a proficiency testing scheme round or to accurately report their performance in any proficiency testing round will result in immediate suspension. The laboratory will not be reinstated until demonstrated satisfactory proficiency testing performance is confirmed.

6.4.4 Incorrect quantitation on any proficiency testing sample are unacceptable for any measurand for which a laboratory is accredited and will result in initiation of action(s) according to clauses 6.4.5 to 6.4.7.

6.4.5 The first date of “Receipt of notification” by the laboratory of any unsatisfactory measurand result from the proficiency testing provider in that proficiency testing round places the onus directly upon the laboratory to immediately initiate corrective action(s). Any preliminary “Performance Report” issued by whatever means by the proficiency testing provider and in what ever format first reported to the laboratory constitutes this “receipt of notification” of the proficiency testing results to the laboratory.

The laboratory shall respond to the SCC MAWG Secretary (with a copy to the proficiency testing coordinator) within 10 working days from the first date of this “receipt of notification” (in what ever format first reported to the laboratory) with an initial corrective actions report (CAR). This initial CAR shall include a description of the unsatisfactory performance or failure and clearly indicating the corrective actions(s) being taken. This initial CAR shall indicate whether the laboratory has requested a “remedial” set of samples for that measurand(s).

a) if a “remedial” set of samples has been requested then:

- these “remedial” set of samples shall be a different set of samples (samples with the concentrations blind to the laboratory, in different concentrations to the original set and the same or more number of samples as in the original round) from that of the current proficiency testing round
- the “remedial” set shall be obtained from the proficiency testing provider
- upon receipt of this set of “remedial” samples from the proficiency testing provider, the laboratory shall analyze them in a timely fashion and report the results to the proficiency testing provider
- the proficiency testing provider shall judiciously evaluate the results using their normal protocols and provide a final “remedial proficiency testing performance report” (however named) to the laboratory. As outlined in section 6.3.2 above the number of participants the laboratory is evaluated against shall be ≥ 10 or another acceptable criteria defined by the proficiency testing coordinator
- the laboratory shall then report to the SCC MAWG Secretary (with a copy to the proficiency testing coordinator) within 10 working days of receiving the “remedial proficiency testing performance report” (in what ever format first reported to the laboratory) with the final CAR which shall clearly include root cause analyses and all the corrective actions(s) taken. The laboratory shall also electronically report their proficiency testing performance by providing a copy of the proficiency testing providers final “remedial proficiency testing performance report”
- this total process should generally not take longer than the normal SCC protocols for CARs (i.e. 30 working days)

b) if no “remedial” set of samples are required, the laboratory shall respond to the SCC MAWG Secretary within 30 working days with the final CAR which shall clearly include root cause analyses and all the corrective actions(s) taken.

The SCC MAWG proficiency testing subcommittee will evaluate the results of this final CAR(s) to determine if satisfactory proficiency testing performance has been achieved. The SCC MAWG may request additional information to substantiate satisfactory proficiency testing performance has been achieved.

NOTE: The proficiency testing coordinator has no responsibility for monitoring the laboratory’s proficiency testing performance as it relates to the maintenance of SCC accreditation under this PSA-MA. This is the sole responsibility of the SCC.

6.4.6 Failure to provide the final CAR response or a copy of the proficiency testing providers final “remedial proficiency testing performance report” (in what ever format first reported to the laboratory) as required by section 6.4.5 following unsatisfactory proficiency testing performance on any specific measurand/test method in any proficiency testing cycle shall result in suspension or withdrawal of accreditation. The CAR(s) shall provide sufficient objective evidence that the problem has been identified, root cause analyses has been conducted and all the corrective actions(s) taken. An unsatisfactory CAR(s) may also initiate an on-site visit by the SCC.

6.4.7 Failure obtaining satisfactory performance for any measurand in that “remedial” set of proficiency testing sample(s) shall initiate the SCC MAWG Convener to recommend to the SCC MAWG Secretary an immediate suspension of that test(s). The SCC MAWG Secretary will advise the Manager Laboratories - PALCAN to immediately suspend that test(s). The laboratory’s test(s) will not be reinstated until substantiated proof of demonstrated satisfactory performance has been confirmed by the next scheduled round of the proficiency testing providers’ proficiency testing scheme for that test(s).

6.4.8 Should the SCC initiate action to suspend the laboratory's accreditation for the specific test(s), the laboratory's official status will become "suspended" according to normal SCC protocol until such time that demonstrated satisfactory proficiency testing performance is confirmed and the suspension is lifted.

6.4.9 Continued unsatisfactory proficiency testing performance over three (3) occurrences in any proficiency testing rounds in any two (2) year period will trigger SCC to initiate withdrawal according to the procedure in the current version of CAN-P-15.

6.4.10 A laboratory whose accreditation has been withdrawn can reapply according to normal SCC protocol

BIBLIOGRAPHY

The following is an extensive list of recommended references:

APLAC PT 002:2008, Testing Interlaboratory Comparisons. (<http://www.aplac.org/index.php?id=86>)

APLAC PT 003:2008, APLAC Proficiency Testing Directory. (<http://www.aplac.org/index.php?id=86>)

APLAC PT 006:2008, Proficiency Testing Frequency Guidelines. (<http://www.aplac.org/index.php?id=86>)

CITAC/EURACHEM ECTRACE, 2003, Traceability in Chemical Measurement, a guide to achieving comparable results in chemical measurement. (<http://www.citac.cc/>)

CITAC/EURACHEM Guide, English Edition 1.0, December 1998 (currently under revision 2008), The Fitness for Purpose of Analytical Methods. A Laboratory Guide to Method Validation and Related Topics. ISBN 0-948926-12-0 (<http://www.citac.cc/>)

CITAC/EURACHEM Guide, Edition 2002, Guide to Quality in Analytical Chemistry. An Aid to Accreditation. (<http://www.citac.cc/>)

CITAC/EURACHEM Guide CG2, English Edition 2nd internet version, November 1998, Quality Assurance for Research and Development and Non-routine Analysis (CAN-P-1595 July 2001). (<http://www.citac.cc/>)

CITAC/EURACHEM Guide CG4, Second Edition, 2000 (**QUAM: 2000.P1**). Quantifying Uncertainty in Analytical Measurement. (<http://www.citac.cc/>)

ILAC Guide 13:08/2007, ILAC Guidelines for the Requirements for the Competence of Providers of Proficiency Testing Schemes.

ISO 3534-1:1993(E/F), Statistics - Vocabulary and Symbols Part 1: Probability and General Statistical Terms.

ISO 5725-1:1994(E) (amendment 1998), Accuracy (trueness and precision) of Measurement Methods and Results-Part 1: General Principles and Definitions.

ISO 5725-2:1994(E) (amendment 2002), Accuracy (trueness and precision) of Measurement Methods and Results-Part 2: Basic Method for the Determination of Repeatability and Reproducibility of a Standard Measurement Method.

ISO 5725-3:1994(E) (amendment 2001), Accuracy (trueness and precision) of Measurement Methods and Results-Part 3: Intermediate Measures of the Precision of a Standard Measurement Method.

ISO 5725-4:1994(E), Accuracy (trueness and precision) of Measurement Methods and Results-Part 4: Basic Methods for the Determination of the Trueness of a Standard Measurement Method.

ISO 5725-5:1998(E) (amendment 2005), Accuracy (trueness and precision) of Measurement Methods and Results-Part 5: Alternative Methods for Determination of the Precision of a Standard Measurement Method.

ISO 5725-6:1994(E) (amendment 2001), Accuracy (trueness and precision) of Measurement Methods and Results-Part 6: Use in Practice of Accuracy Values.

ISO 7870:1993, Control Charts - General Guide and Introduction.

ISO 7873:1993, Control Charts for Arithmetic Average with Warning Limits.

ISO 9000:2000, Quality Management Systems - Fundamentals and Vocabulary.

ISO Guide 30:1992, Terms and definitions used in connections with reference materials.

ISO Guide 33:1989, Uses of Certified Reference Materials.

ISO/IEC Guide 2:1996, General terms and their definitions concerning standardization and related activities.

ISO/IEC Guide 43-1:1997(E), Proficiency Testing by Interlaboratory Comparisons - Part 1: Development and Operation of Proficiency Testing Schemes.

ISO/IEC Guide 43-2:1997(E), Proficiency Testing by Interlaboratory Comparisons - Part 2: Selection and Use of Proficiency Testing Schemes by Laboratory Accreditation Bodies.

ISO/IEC Guide 98-1:2008 (draft 09/2007), Uncertainty of measurement – Part 1: Introduction to the expression of uncertainty in measurement.

ISO/IEC Guide 98-3:2008, Uncertainty of measurement – Part 3: Guide to the expression of uncertainty in measurement (GUM:1995).

ISO/IEC Guide 99, First edition 2007, International Vocabulary of Metrology - Basic and General Concepts and Associated Terms (VIM). (VIM) 3rd ed.:2007).

ISO Standard 13528:2005(E), 1st Edition, Statistical Methods for use in Proficiency Testing by Interlaboratory Comparisons.

ISO/IEC Standard 17025:2005, General Requirements for the Competence of Testing and Calibration Laboratories.

ISO/IEC CD 17043:2008, Conformity assessment – General Requirements for proficiency testing.

Guide to the Expression of Uncertainty in Measurement (**GUM**), 1993 (amended 1995), published by ISO in the name of BIPM, IEC, IFCC, IUPAC and OIML (ISBN 92-67-10188-9). (Note: the new ISO/IEC Guide 98-1:2008 supplement)

Harmonised Guidelines for Internal Quality Control in Analytical Chemistry Laboratories, Pure Appl. Chem., Vol. 67, pp 649-666, 1995.
(<http://www.iupac.org/publications/pac/1995/pdf/6704x0649.pdf>)

Harmonized Guidelines for Single-laboratory Validation of Methods of Analysis (IUPAC Technical Report) 2002 - Pure Appl. Chem., Vol. 74, No. 5, pp 835-855, 2002.
(<http://www.iupac.org/publications/pac/2002/pdf/7405x0835.pdf>)

Nomenclature, symbols, units and their useage in spectrochemical analysis - II Data Interpretation, IUPAC 1975, Spectrochim. Acta B 33B, 1978, p.241-245,

Quality Assurance of Chemical Measurements, John Keenan Taylor, Lewis Publishers, p85, 1987,

Standard Practice for Dealing with Outlying Observations, ASTM E178-80.

The Expression of Uncertainty and Confidence in Measurement, UKAS M3003, Edition 2, January 2007. (<http://www.ukas.com/Library/downloads/publications/M3003.pdf>)

The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories (IUPAC Technical Report) Prepared for publication by Michael Thompson, Stephen L.R. Ellison, Pure Appl. Chem., Vol. 78, No. 1, pp 145-196, 2006. (<http://www.iupac.org/publications/pac/2006/pdf/7801x0145.pdf>)