Title: Proficiency Testing – Organic Laboratories		
SOP No.: 3.18/1.0/S	Effective Date: January 4, 2010	Page No.: 1 of 4

Copy No:

Proficiency Testing – Organic Laboratories

1. Introduction and Scope

Proficiency tests (PT) define the ability of the laboratory and/or analyst to carry out the procedures of the method with acceptable precision and accuracy. Participation in a interlaboratory comparisons or proficiency test programs is a requirement of ISO/IEC 17025:2005. CALA's policy on PT states that all accredited laboratories shall demonstrate their technical competence by the satisfactory participation in a suitable proficiency testing activity. All analytes appearing in the laboratories Scope of Accreditation must be supported by PT. Typically the laboratory must demonstrate this ability twice a year. Participation in interlaboratory testing also enables the laboratory to monitor their performance and compare their results with other laboratories performing the same analysis.

CALA has 6 options for the laboratory to take to meet their PT requirements. The lab must select the first option that is applicable.

Option i: Use either a CALA PT or one from an approved PT provider that has 2 studies per year, 4 samples per study, samples are provided as ready to analyse (i.e.not concentrates).

Option ii: Use an approved PT provider and participate in 2 studies per year, one sample per study.

Option iii is no longer available.

Option iv: Participate in at least one formal study per year from a PT provider eg EPA, and establish acceptable performance criteria if flags are not assigned.

Option v: Participate in at least one less formal inter-laboratory study per year and establish acceptable performance criteria if flags are not assigned.

Option vi: Conduct an inter-analyst comparison at least once a year and establish acceptable performance criteria if flags are not assigned.

Currently, the Chemical Analysis and Methods unit has 5 Scopes of Accreditation. This SOP describes the procedures that are followed for the PT associated with each of the scopes.

2. Procedures

2.1 Method 3.02/*.*/M: Dioxins/Furans in Ambient Air

2.1.1 At this point in time, there are no PT providers for Dioxin/Furan analysis in ambient air samples and as a result AAQS has chosen Option vi to meet the requirements.

2.1.2 At least annually, or whenever there is a change in analysts, 150 mg of NIST Urban Dust 1649b is extracted in triplicate and processed following the method. The results are reviewed and archived by the supervisor. Criteria for acceptable performance includes recovery of surrogates must be within 75 to 120 % and recovery corrected values for the native compounds must be within 30 % of the NIST certified value. PT is repeated if the performance criteria are not met.

2.1.3 The results are kept on file for review during site assessments.

2.1.4 As an alternate, 300 mg of NIST Urban Dust 1649b may be used. In this event, the extract is split and analysed for PCDD/F and PAH following Method 3.03. The results are reviewed and archived by the supervisor. Criteria for acceptable performance includes recovery of surrogates must be within 75 to 120 % and recovery corrected values for the native compounds must be within 30 % of the NIST certified value. PT is repeated if the performance criteria are not met.

2.2 Method 3.03/*.*/M: Analytical Method for the Determination of PAH in Ambient Air

2.2.1 Currently AAQS is using Option ii, using an approved outside provider and participating twice a year, one sample each round. The extract provided is spiked onto a PUF which is also provided, then extracted and cleaned up following the method. The supplier is Environmental Resource Associates, catalogue number 1013, Air & Emissions - PAHs on PUF. The outcome is reported on the CALA Web-Data-Entry system.

2.2.2 The first PT was initiated in December, 2009. Prior to the availability of this PT, AAQS was using Option vi. At least annually, or whenever there is a change in analysts, 100 mg of NIST Urban Dust 1649b is extracted in triplicate and processed following the method. The results are reviewed and archived by the supervisor. Criteria for acceptable performance includes recovery of surrogates must be within 75 to 120 % and recovery corrected values for the native compounds must be within 30 % of the NIST certified value. PT is repeated if the performance criteria are not met.

2.2.3 As an alternate, 300 mg of NIST Urban Dust 1649b may be used. In this event, the extract is split and analysed for PCDD/F and PAH following Method 3.02 and Method 3.03. The results are reviewed and archived by the supervisor. Criteria for acceptable performance includes recovery of surrogates must be within 75 to 120 % and recovery corrected values for the native compounds must be within 30 % of the NIST certified value. PT is repeated if the performance criteria are not met.

2.3 Method 3.04/*.*/M: Analytical Method for the Determination of PCBs – Oils

2.3.1 The Chemical Analysis and Methods unit of AAQS is currently using Option i to meet the requirements for PT. CALA supplies 4 oil samples twice a year. Samples are initially screened using GC/ECD then the appropriate weight is withdrawn and the sample is processed through the various procedures for GC/MS analysis. Results are reported through the CALA Web-Data-Entry system.

2.4 Method 3.04/*.*/M: Analytical Method for the Determination of PCBs – Soil

2.4.1 The Chemical Analysis and Methods unit of AAQS is currently using Option i to meet the requirements for PT. CALA supplies 4 soil samples twice a year. Samples are initially screened using GC/ECD then the appropriate weight is withdrawn and the sample is processed through the various procedures for GC/MS analysis. Results are reported through the CALA Web-Data-Entry system.

2.5 Method 3.05/*.*/M: Determination of PCDD and PCDF in Pulp and Paper Mill Effluents

2.5.1 Currently AAQS is using Option ii, (using an approved outside PT provider) and participating twice a year, one sample each round. The extract provided is spiked into 1000 mL of water, then extracted and cleaned up following the method. The supplier is Environmental Resource Associates, catalogue number 857, WatR Supply Dioxin. The outcome is reported on the CALA Web-Data-Entry system.

2.5.2 In addition, at least annually, or whenever there is a change in analysts, 1000 mL of deionized water, in triplicate, is spiked with 20 to 80 pg of native PCDD/F then extracted and processed following the method. The results are reviewed and archived by the supervisor. Criteria for acceptable performance includes recovery of surrogates must be within 75 to 120 % and recovery corrected values for the native compounds must be within 30 % of the amount spiked. PT is repeated if the performance criteria are not met.

2.6 Method 3.09/*.*/M: Determination of Hexachlorobenzene, Pentachlorophenol and Octachlorostyrene in Ambient Air

2.6.1 At this point in time, there are no PT providers for Hexachlorobenzene, Pentachlorophenol and Octachlorostyrene analysis in ambient air samples and as a result AAQS has chosen Option vi to meet the requirements.

2.6.2 At least annually, or whenever there is a change in analysts, 100 mg of NIST Urban Dust 1649b in triplicate, is weighed onto an EMFAB filter and spiked with 25 ng of HCB and OCS native then extracted and processed following the method. The results are reviewed and archived by the supervisor. Criteria for acceptable performance includes recovery of surrogates must be within 75 to 120 % and recovery corrected values for

the native compounds must be within 30 % of the spiked value. PT is repeated if the performance criteria are not met.

2.6.3 The results are kept on file for review during site assessments.

3. Revisions

Dec. 2009: Author: Gary Poole; New document

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