

CALA DISPUTES - 2009

Requirement	Dispute Resolution and Explanation
<p>Ensure alternate staff are designated to substitute for key managerial positions. It was noticed that as per laboratory's SOP QP01.01.011 no staff member from the laboratory was officially designated to fill in for the Technical Manager. The staff assisting in the assessment had a lot of difficulty locating the quality management system documents.</p>	<p>Upheld</p> <p>A designate for the Technical Manager is specified in QP01.01.011, so this item will be removed from the reassessment report.</p>
<p>Ensure that the laboratory has a calibration program for its measurement and test equipment, and meets traceability requirements as per CALA Traceability Policy (A61). Records of ISO/IEC 17025 accreditation for the company performing calibration on mechanical pipettes were not available.</p>	<p>Denied</p> <p>As pipettes are being sent out to a company, the pipettes are, in fact undergoing an external calibration - not an internal calibration. CALA policy is that when sending dispensing devices out for calibration, they must be sent to an accredited calibration laboratory. This must remain as a Required "A" item.</p>
<p>Modify procedures so that there the records of method validation, specifically for precision and accuracy of pH, in which statistical calculations are performed on the actual hydrogen ion concentrations rather than on their negative logarithmic modes of expression, after which the means, RSDs, and confidence limits can be re-expressed in pH units.</p>	<p>Upheld</p> <p>The panel agreed with the laboratory's position that the current procedure is in line with industry standards and meets the needs of the laboratory. It was generally accepted that the assessors were correct in their determination from a purely scientific point of view and that there are limitations to statistical analysis for pH. However, the method references commonly used (e.g. Standard Methods, EPA) makes no reference to the use of the logarithmic modes of expression and states accuracy and precision directly in pH units.</p>

Requirement	Dispute Resolution and Explanation
<p>The use of low level duplicates to calculate method detection limits is typically be used only when it is difficult to make low level standards (e.g., conductivity and turbidity). The laboratory uses the practice of low level duplicates for all method detection limit calculations which, according to current documentation (e.g., Eurochem Guide), is not the appropriate practice for method detection limit determination. REQUIRED ACTION Submit documentary evidence to support the laboratory’s calculation procedure for method detection limit using duplicate pairs. If a change is made to use low level standards for determination of method detection limit, then modify the procedure and recalculate the method detection limits, accordingly.</p> <p>FINDING: The laboratory calculates the method detection limit using low level duplicates. This method is not consistent with the method of calculating the method detection limit used in other accredited laboratories, which use a low level standard for this calculation. Hence, the capability of the laboratory listed on the scope is not comparable with that of other accredited labs due to the methodology difference. The use of low level duplicate measurements for method detection limit determination is typically suitable for tests for which reliable low level test samples cannot be prepared (for example, conductivity and turbidity). REQUIRED ACTION: Provide documentary evidence that the process of using low level duplicates is more appropriate for calculating method detection limits for this laboratory than the standard process, or, recalculate the method detection limit for all accredited tests using test</p>	<p>Denied</p> <p>The findings are different, although the documentary evidence may be similar.</p>

Requirement	Dispute Resolution and Explanation
<p>samples with a concentration between 3 and 10 times the method detection limit.</p>	
<p>Submit documentary evidence that test reports include statement to the effect that the results relate only to the items tested for Micro Reports.</p> <p>Submit documentary evidence that test reports include a statement to the effect that the results relate only to the items tested. This is not on the Precipitation Report format.</p>	<p>Upheld</p> <p>These two findings are covered in the quality system finding 070.</p>
<p>Document and implement a procedure so that method quality control is adequate and either included or referenced in the test method; specifically, use a full list of analyzed pesticides as reference sample to monitor accuracy/recovery.</p>	<p>Denied</p> <p>The lab needs to cover far more target analytes in reference materials than are currently.</p>
<p>Submit documentary evidence that waterbaths are available and functioning properly; i.e., maintained within the specified temperature range, and temperatures monitored and recorded at least once daily. The temperature log is not present.</p>	<p>Upheld</p> <p>There is no waterbath used for this appendix.</p>
<p>Submit documentary evidence that the Quality Manual that includes or makes reference to all procedures, within a defined document structure; for example cross reference to appropriate sections of standard operating procedure Manual.</p>	<p>Upheld</p> <p>The structure of the documentation and Master Document List were included.</p>

Requirement	Dispute Resolution and Explanation
<p>Submit documentary evidence that obsolete documents are removed. A methods binder was found in the laboratory containing 3 "older" methods from 1997.</p>	<p>Upheld</p> <p>The CALA Accreditation does not extend to methods outside the accreditation.</p>
<p>Submit documentary evidence that all support equipment required for the test procedure is available and functioning properly, specifically, the oven (AGR74) used for drying at 103 to 105 deg Celsius was observed at 106.3. In the absence of a max - min thermometer this problem could be common. The max and min should be recorded to avoid the problem.</p>	<p>Denied</p> <p>The lab needs to demonstrate that equipment can maintain the required temperature for the test (e.g., submit temperature records from when the oven is in use for this test).</p>
<p>Submit documentary evidence that records of method validation include an estimate of the measurement uncertainty, specifically for VOC in water method.</p>	<p>Denied</p> <p>All tests must have a measurement uncertainty calculated as part of the method validation, even new methods. This method has been accredited for some time, so the laboratory should have sufficient data to calculate the measurement uncertainty. The laboratory can continue to collect additional data and recalculate and update the measurement uncertainty at a future date.</p>
<p>Modify procedures so that submit documentary evidence that the water used is organic free water. The water used for the procedure is DI water.</p>	<p>Denied but reworded</p> <p>As records showing the water used was "organic free" were not seen, the lab must either submit evidence that the water used is organic free, as described in the method, or modify the method to accurately describe the type of water used for this method.</p>

Requirement	Dispute Resolution and Explanation
<p>Submit documentary evidence that method quality control is adequate and either included or referenced in the test method; specifically, use control charting to monitor method quality control specifically the use of statistically derived control limits instead of the current limits stated in the method.</p>	<p>Denied but reworded</p> <p>Section 5.9.1 of ISO/IEC 17025:2005 requires that “resulting data shall be recorded in such a way that trends are detectable and, where practicable, statistical techniques shall be applied to the reviewing of the results”. If the lab has no records of nonconformances using the current limits, then there should be records of review examining this finding and some reason given why the fixed limits were kept and why there are nonconformances are not occurring (statistically some nonconformances would occur).</p>
<p>Modify procedures so that sample history requirements are adequate and either included or referenced in the test method, specifically, the reference method states the sample storage at 4 +/- deg Celsius, the method states 4 deg Celsius and the fridge (AGR 90) has a log sheet that states a temperature of 0-4 deg Celsius. A) need consistency and B) need to agree with reference method.</p>	<p>Denied</p> <p>The method and the lab practice need to agree on the temperature and any allowable temperature range.</p>
<p>Modify procedures so that sample history requirements are adequate and either included or referenced in the test method, specifically, better define the word refrigeration and update to agree with the stated reference method, Standards Methods, which states 4 +/- 2 deg Celsius.</p>	<p>Denied</p> <p>The method and the lab practice need to agree on the temperature and any allowable temperature range.</p>
<p>Submit documentary evidence that sample history requirements are adequate and either included or referenced in the test method, specifically, storage conditions in the method should be identical to the limits if the walk-in cooler (4 +/- 3 degrees Celsius).</p>	<p>Denied</p> <p>The method and the lab practice need to agree on the temperature and any allowable temperature range.</p>

Requirement	Dispute Resolution and Explanation
<p>Modify procedures so that sample history requirements are adequate and either included or referenced in the test method, specifically, the holding temperature of 4 deg Celsius does not agree with the temperature log sheet on the cold room of 4 +/- 3 deg Celsius.</p>	<p>Denied</p> <p>The method and the lab practice need to agree on the temperature and any allowable temperature range.</p>
<p>Modify procedures so that sample history requirements are adequate and either included or referenced in the test method, specifically, the storage temperature should agree with the storage temperature stated in the reference method.</p>	<p>Denied</p> <p>The method and the lab practice need to agree on the temperature and any allowable temperature range.</p>
<p>Document and implement policies and procedures to ensure corrective actions are taken and documented for non-conformances. When the quality controls of a set of samples failed twice on one instrument but passed when the sample set was then run on a second instrument with no problems, the data was approved, however, there was no documentation found of the action taken to identify the root cause of the failures on the first instrument. This was observed for more than one method.</p>	<p>Denied</p>
<p>Document and implement a procedure so that deviations from the test methods are documented, technically justified, authorized by the appropriate authority and ensure that any deviations are recorded.</p>	<p>Denied</p>
<p>The laboratory has a calibration program for its measurement but it does not have criteria for recording and addressing what to do in the case of nonconformance's for organic methods. Document in the standard operating procedure what to do in the case of a nonconformance.</p>	<p>Denied</p>

Requirement	Dispute Resolution and Explanation
<p>Document and implement a procedure so that organisms used for quality control purposes that have been isolated from the environment are properly characterized and that biochemical characteristics are checked annually. If organisms are purchased, verify that there is a certificate with the organism name, plus confirmation on selective medium.</p>	<p>Upheld</p> <p>This was in relation to sulphur-reducing and iron-reducing bacteria. While standard strains are difficult to obtain and maintain, lab needs some form of positive QC sample.</p>
<p>Submit documentary evidence that all necessary successive steps in the test procedure are adequately documented in the test method, specifically, that the procedure is based on the latest published reference method and that deviations are assessed for CAL SOP 00073 with regard to EPA 335.2 and 335.3.</p>	<p>Denied</p>
<p>The thermocouples used in the method are not calibrated by an accredited calibration laboratory. Submit documentary evidence that the thermocouples meet the requirements of the CALA Traceability Policy (A61).</p>	<p>Denied</p>
<p>Review reference methods for sample preservation and holding time requirements, specifically, (holding time for preserved samples is 28 days and unpreserved samples is 1 day according to BC MOE guidelines) to ensure method is fit for purpose.</p>	<p>Denied</p>

Requirement	Dispute Resolution and Explanation
<p>Document and implement a procedure so that resultant chromogenic reactions are properly confirmed with CFU on appropriate culture media.</p> <p>Modify procedures so that method quality control is adequate and either included or referenced in the test method; specifically, use standard strains to monitor performance of the test method. (ATCC).</p>	<p>Upheld and item removed; this requirement is not applicable to iron-reducing bacteria.</p> <p>Partially upheld; while standard ATCC strains are not available for iron-reducing bacteria, the lab needs some form of positive QC sample.</p>
<p>Document and implement a procedure so that supplies required for the test procedure are available and meet requisite requirements, specifically, inhibitory effects of filters tested by comparison of recoveries on a membrane filter and a spread plate</p>	<p>Upheld</p> <p>The current laboratory procedures more than adequately fulfill the requirements of clause 4.6.2.</p>
<p>Modify procedure to include the reference to the supporting work instructions in the test method; specifically, include reference to SOPxxx: Soil Drying and Soil Grinding and SOPyyy: Preparation of Soil Sample for Ion Measurement</p>	<p>Upheld</p> <p>Soils are not part of the accredited scope.</p>
<p>Submit documentary evidence that records are maintained related to the performance of the test method, specifically, records of volumetric traceability for the autoclaved pipette tips. The pipette tips are being autoclaved and are being used to measure sample volume. The pipette tips should be checked for volumetric traceability after they are autoclaved to ensure the process is not affecting their dispensing volume</p>	<p>Denied but reworded</p> <p>Pipette verification needs to occur after the autoclaving of the pipette and tips.</p>

Requirement	Dispute Resolution and Explanation
<p>Submit documentary evidence that all supplies are stored under appropriate conditions (eg 1-4 degrees Celsius) and in a manner which satisfies requirements for safety, security, separation or incompatible materials, and ease of retrieval, specifically, silica is activated for 2 hours at 250C but method states 16 hours. There is no log of activation of reagents in oven.</p>	<p>Denied but reworded</p> <p>The lab was not following their own procedure. The log of silica gel activation is not required.</p>
<p>Document and implement a procedure so that all supplies required for the test procedure are available and meet requisite requirements and/or specifications; specifically, check hydrophobicity of filters (e.g., charcoal test or confluent growth or other acceptable method (e.g., as per Standard Methods)).</p>	<p>Upheld</p> <p>The assessor was not familiar with the hydrophobicity test that the lab is using.</p>
<p>Modify policy or procedure so that the laboratory shall advise the customer of the subcontracting arrangement in writing; specifically, include this in policy/procedure; currently, the laboratory does this (e.g., by way of signed memorandum).</p>	<p>Denied</p> <p>The lab needs to notify its clients of the use of subcontractors regardless of whether they are asked.</p>
<p>Document and implement a procedure so that procedures for management of measuring equipment are established and include procedures for safe handling, transport, storage, use, maintenance, intermediate calibration checks and updating of documents (e.g., computer software, when correction factors are generated); specifically, document a procedure for handling and transport of equipment being sent from the laboratory.</p>	<p>Denied</p>

Requirement	Dispute Resolution and Explanation
<p>Submit documentary evidence that the laboratory has a calibration program for its measurement and test equipment, and meets traceability requirements as per CALA Traceability Policy (A61); specifically, calibrate thermometers annually according to the CALA Traceability Policy. Currently, the laboratory is using factory calibrated thermometers with 2 year recalibration date which requires less than 1/10 contribution to uncertainty (5.6.2.2.1).</p>	<p>Denied</p>
<p>Modify procedures so that records of nonconformance and corrective actions taken are maintained, specifically, document required action or reasons for no action when duplicate control limits are exceeded, specifically, a wastewater duplicate for Boron exceeded the CL at 29% over expected limit of 20%. Non-conformance was documented, however, no explanation nor follow-up occurred (run xxxxx).</p>	<p>Upheld</p> <p>The 20% limit was for water; the sample in question was a different matrix (biosolid).</p>
<p>Submit documentary evidence that there are records of method validation for GC/ECD instruments. Not all instruments have been validated to run samples.</p>	<p>Denied</p>
<p>Document the existing procedure, which identifies subcontracted results in the final report, specifically in section 8.6.1 of the QAM.</p>	<p>Upheld</p> <p>This information is already included in section 8.6.1 of the QAM.</p>

Requirement	Dispute Resolution and Explanation
<p>Submit documentary evidence that all necessary supporting work instructions are either included or referenced in the test method; specifically, include the Environment Canada prohibition against the reuse of control fish.</p>	<p>Deleted</p> <p>Error. This item relates to appendix 14, which doesn't exist on the scope.</p>
<p>Modify the method review procedures so that details are provided on editing changes to the method, such as the changes made in response to the May 2007 amendments to the reference method.</p>	<p>Upheld</p> <p>May 2007 reference method amendments were made in August 2009 revision.</p>
<p>Include the holding conditions for samples which are to be run on the day following sample receipt.</p>	<p>Upheld</p> <p>In method, section 1.4.4, holding conditions are included</p>
<p>Include the use of the randomization spreadsheet, or a cross-reference to and SOP on its use.</p>	<p>Upheld</p> <p>Randomization is not applicable to the method Microtox liquid phase.</p>
<p>Modify the method so that the EC requirement that duration and temperatures for mixing should be as similar as possible is included</p>	<p>Upheld</p> <p>In method, section 1.6.3, this requirement is included</p>
<p>Include the use of the randomization spreadsheet, or a cross-reference to and SOP on its use.</p>	<p>Upheld</p> <p>Randomization is not applicable to the method Pseudokirchneriella Subcapitata.</p>

Requirement	Dispute Resolution and Explanation
<p>Modify procedure so that the latest valid edition of AWWA Standard Method (21st, 2005) is referenced in the method, unless it is not possible or appropriate.</p>	<p>Upheld</p> <p>Not appropriate. Method is done for a specific client need.</p>
<p>Modify procedure so that analyst worksheets contain adequate information; specifically, record the balance ID for weight measurements to provide traceability to balance calibration.</p>	<p>Upheld</p> <p>Balances are not used in the preparation or analysis of water samples. A volume of sample is prepared using a volume of solvent.</p>
<p>Modify procedure so that analyst worksheets contain adequate information; specifically, record the ID of syringes for volumetric dispensing to provide traceability to syringe accuracy verification.</p>	<p>Upheld</p> <p>Each analyst has his own set of syringes that no one else is able to access. The syringes are labeled with the analyst's names. The syringe calibration verification logs identify the syringes by analyst and all analyst worksheets, bench sheets and record/log books contain the analyst's initials. This provides traceability for the syringes. The syringe inventory from calibration/verification file has been attached.</p>
<p>Given that Proficiency Testing (i.e., the annual Norwegian round robin currently participated in) is the primary means for validation this method, modify the written procedure such that it clearly outlines the importance of maintaining participation in PTs (including inter and intra laboratory comparisons).</p>	<p>Downgraded from "A" to "C".</p> <p>Proficiency testing is covered in the Quality Manual, sections 2.4.2, 2.4.4 and 2.6, with an appendix that lists the studies that laboratory regularly participates in. The Norwegian study will be added to the list and a National round robin is in the planning stages. The laboratory shall submit a new PT plan to Ken that they upgraded the Correlation Proviso from option vi to option iv.</p>

Requirement	Dispute Resolution and Explanation
<p>Submit documentary evidence that evaluation of water quality requirements (total residual chlorine ≤ 0.002 mg/L and un-ionized ammonia ≤ 0.02 mg/L) is achieved as described in the reference method.</p>	<p>Denied but reworded</p> <p>The laboratory disputed portion of item 07 pertaining to residual chlorine measurements with a LDL of ≤ 0.002 mg/L and this portion has been removed based on the fact that levels < 0.01 mg/L are satisfactory for this method.</p>
<p>Modify procedures so that method quality control is adequate and either included or referenced in the test method; specifically, use duplicates to monitor precision. Currently, MSOP14.07 specifies that duplicates will only be processed if more than one jar is received for a given sample. The laboratory, in the absence of sufficient sample, should at least be preparing two LCS and using the precision data for these analyses to meet the criteria for dups.</p>	<p>Upheld</p> <p>The lab is using a validated method, which means that they have evaluated the trueness and precision (repeatability/reproducibility) of the method through controlled studies, and has (presumably) defined their fit-for-use and level of QC they are going to use. The on-going verification of performance (trueness and precision) can be achieved through the use of the LCS; the results of which can be evaluated against the known or accepted value and also against the historical performance of the method to determine reproducibility (which includes random error). The inclusion of 2nd and 3rd LCS samples (unless specified by the method), is not providing a more robust evaluation of the overall precision; rather, it is an evaluation of the repeatability of the method, which has vary less variation than the reproducibility evaluation. And the lab has an allowance for the inclusion of duplicate samples where sample volume permits. The Advisory Panel member believes that the effort put forth is sufficient to demonstrate technical competence.</p>

Requirement	Dispute Resolution and Explanation
<p>Modify procedures so that method calibration is adequate and either included or referenced in the test method; specifically, use a reagent blank to establish the calibration baseline. Specifically, although analyzed, the instrument blank is not used in the generation of the calibration curve.</p>	<p>Upheld</p> <p>The blank value would be below the linear range of the test and should not be used in the calibration curve. The Advisory Panel member agrees with the laboratory's explanation.</p>
<p>Modify procedures so that method calibration is adequate and either included or referenced in the test method; specifically, use a reagent blank to establish the calibration baseline. CCME requires that the instrument blank be incorporated into the calibration curve for this method; however, although analyzed within the sequence, the instrument blank is not included in the calibration curve.</p>	<p>Upheld</p> <p>The blank is not included in the calibration curve. The Advisory Panel member agrees with the laboratory's explanation.</p>
<p>Modify procedures so that all necessary supporting work instructions are either included or referenced in the test method, specifically, NA-TM-1101 v01, section 11.5, does not include references to associate lab methods. Modify document to include all associated method references.</p>	<p>Upheld</p> <p>The document NA-TM-1101 refers to local equipment logs, procedures, supporting instructions and analytical records, specifically, sections 6, 7 and 11.5.</p>
<p>The CALA Uncertainty Policy (P19) Appendix 2 indicates the reported MU shall be statistically derived. The reported MU for the test method parameter(s) is different than the most recently derived MUs. Update the reported MUs to those recently derived.</p> <p>Ensure the MU includes uncertainty from the sample processing (extraction) steps, if it is determined to be significant as per CALA policy.</p>	<p>Upheld - Last sentence in finding related to MU of sample processing removed.</p> <p>The laboratory disputed the portion of the finding related to the MU from the sample processing steps. The laboratory indicated that this was already a part of their uncertainty determination and submitted records to demonstrate this.</p>

Requirement	Dispute Resolution and Explanation
<p>Submit documentary evidence that records of volumetric traceability are maintained; specifically, provide evidence that measurement uncertainty has been completed for pipettes.</p>	<p>Upheld - Laboratory submitted records to demonstrate that MU was already included in the pipette calibrations.</p>
<p>Submit documentary evidence and modify procedure so that method calibration is adequate and either included or referenced in the test method; specifically use an unseeded reagent blank.</p>	<p>Denied</p> <p>The laboratory should be running 3 quality control samples with each batch:</p> <ol style="list-style-type: none"> 1. Glucose-glutamic acid check - to monitor accuracy and precision 2. Dilution water check - to monitor dilution water quality and cleanliness of BOD bottles; DO uptake must be <0.20 mg/L 3. Seed Control - to check the DO depletion of the seed <p>The dilution water check is done to show that the DO depletion from the dilution water will not be a significant part of the DO depletion of the Seed Control. It is required for each batch of samples to incubate a minimum of one dilution water (with all nutrients and buffer but no seed or nitrification inhibitor) to monitor the quality of the unseeded dilution water and cleanliness of the sample and incubation bottle.</p> <p>The reference method states that if the DO uptake of the dilution water is >0.20 mg/L over 5 days then the results are to not be reported or marked as not meeting quality control criteria. Therefore the lab needs to perform the dilution water check without seed or nitrification inhibitor with every batch of samples.</p>