4740.2100 QUALITY CONTROL CRITERIA FOR CHEMISTRY EXCEPT RADIOCHEMISTRY.

Subpart 1. **Scope.** This part applies to laboratories performing testing under the inorganic chemistry, metals, volatile organic compounds, and other organic compounds test categories unless otherwise indicated. All requirements in this part must be incorporated into the laboratory's procedures unless otherwise directed by the approved method. The quality control requirements specified by the laboratory's standard operating procedures manual must be followed. All quality control measures must be assessed and evaluated on an ongoing basis and quality control acceptance criteria must be used to determine the validity of the data.

Subp. 2. **Method blanks.**

A. The method blank must be processed along with and under the same conditions as the associated samples to include all steps of the analytical procedure.

B. Each contaminated method blank must be critically evaluated as to the nature of the interference and the effect on the analysis of each sample within the batch. The source of contamination must be investigated and measures taken to minimize or eliminate the problem. Affected samples must be reprocessed or data must be appropriately qualified if:

(1) the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the test method or by regulation and is greater than one-tenth of the amount measured in any sample; or

(2) the blank contamination otherwise affects the sample results according to test method requirements or the individual project data quality objectives.

C. Procedures must be in place to determine whether a method blank is contaminated. Any affected samples associated with a contaminated method blank must be reprocessed for analysis or the results reported with appropriate data qualifying codes.

D. The method blank must be analyzed at a minimum of one per batch.

Subp. 3. **Laboratory control sample.**

A. A laboratory control sample (LCS) must be used to evaluate the performance of the total analytical system, including all preparation and analysis steps. Results of the LCS must be compared to established criteria and, if found to be outside of established criteria, must indicate that the analytical system is "out of control." Any affected samples associated with an out-of-control LCS must be reprocessed for reanalysis or the results reported with appropriate data qualifying codes.

B. A laboratory control sample must be analyzed at a minimum of one per preparation batch except:

(1) analytes for which no spiking solutions are available; or

(2) in instances for which no separate preparation method is used, such as volatiles in water, the batch must be defined as environmental samples that are analyzed together with the same

method and personnel, using the same lots of reagents, not to exceed the analysis of 20 environmental samples.

C. All analyte concentrations must be within the calibration range of the instrument calibration. The components to be spiked must be as specified by the permit, program, or rule requirement. In the absence of permit, program, rule, or method requirements, the laboratory must spike as follows:

(1) for those components that interfere with an accurate assessment, such as spiking simultaneously with technical chlordane, toxaphene, and PCBs, the spike must be chosen that represents the chemistries and elution patterns of the components to be reported; and

(2) the number of analytes selected is dependent on the number of analytes reported. The analytes selected for the spiking solution must be representative of all analytes reported. The following criteria must be used for determining the minimum number of analytes to be spiked:

(a) for methods that include one to ten analytes, spike all components;

(b) for methods that include 11 to 20 analytes, spike at least ten components or 80 percent of the analytes, whichever is greater; and

(c) for methods with more than 20 analytes, spike at least 16 components.

D. The results of the analytes included in the LCS are calculated in percent recovery or measure that allows comparison to established acceptance criteria. The laboratory must document the calculation. The individual LCS is compared to the acceptance criteria as published in the approved method. When there are no established criteria, the laboratory must determine its own criteria and document the method used to establish the limits or utilize client-specified assessment criteria within a permit, program, or rule requirement.

E. A laboratory control sample that is determined to be within the criteria effectively establishes that the analytical system is in control and validates system performance for the samples in the associated batch. Samples analyzed along with a LCS determined to be "out of control" must be considered suspect. The samples must be reprocessed and reanalyzed or the data reported with appropriate data qualifying codes.

Subp. 4. **Matrix spike and matrix spike duplicates.**

A. The frequency of the analysis of matrix spikes and matrix spike duplicates must be determined as part of a systematic planning process or as specified by the required approved method. The matrix spikes must be prepared from samples contained in the batch.

B. For a matrix spike, the components to be spiked must be as specified by the approved method or permit, program, or rule requirement. In the absence of specified spiking components, the laboratory may follow client instructions and then must document its criteria for quality control. In the absence of client instruction, the laboratory must spike as follows:

(1) for those components that interfere with an accurate assessment, such as spiking simultaneously with technical chlordane, toxaphene, and PCBs, the spike must be chosen that represents the chemistries and elution patterns of the components to be reported; and

(2) the number of analytes selected is dependent on the number of analytes reported. The analytes selected for the spiking solution must be representative of all analytes reported. The following criteria must be used for determining the minimum number of analytes to be spiked:

(a) for methods that include one to ten analytes, spike all components;

(b) for methods that include 11 to 20 analytes, spike at least ten or 80 percent of the analytes, whichever is greater; and

(c) for methods with more than 20 analytes, spike at least 16 components.

C. The results from matrix spikes and matrix spike duplicates must be expressed as percent recovery, relative percent difference, absolute difference, or other measure. Results of matrix spikes and matrix spike duplicates must be compared to the acceptance criteria as published in the approved method. When there are no established criteria, the laboratory must determine its own criteria and document the procedure used to establish the limits or utilize client-specified assessment criteria within a permit, program, or rule requirement.

Subp. 5. **Surrogate spikes.**

A. This subpart applies to the analysis of organic compounds.

B. Except when the matrix precludes their use, or when not available, surrogate compounds must be added to all samples, standards, and blanks for all appropriate test methods.

C. Surrogate compounds must be chosen to represent the various chemistries of the analytes in the method. When specified, the surrogates mandated in the method must be used.

D. The results from surrogate spikes must be expressed as percent recovery. Results of surrogate spikes must be compared to the acceptance criteria as published in the approved method. When there are no established criteria, the laboratory must determine its own criteria and document the method used to establish the limits or utilize client-specified assessment criteria within a permit, program, or rule requirement.

Subp. 6. **Internal standards.**

A. When internal standards are recommended or required by the test method, such as mass spectrometry techniques, a laboratory must add the internal standards to all samples, standards, blanks, and quality control samples before analysis.

B. When specified in the test method, a laboratory must use the internal standards mandated in the test method. If internal standards are not recommended in the method, then the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest and not expected to be found in the samples otherwise.

C. A laboratory must monitor and document the results from analysis of internal standards.

D. Results of internal standards must be compared to the acceptance criteria as published in the approved method. When there are no established criteria, the laboratory must determine its own criteria and document the procedure used to establish the limits or utilize client-specified assessment criteria within a permit, program, or rule requirement.

Subp. 7. **Detection limits.**

A. A laboratory must utilize a test method that provides a detection limit that is appropriate and relevant for the intended use of the data. The detection limit, such as method detection limit (MDL), must be determined by the protocol in the approved method or applicable regulation. If the protocol for determining detection limits is not specified, the selection of the procedure must reflect instrument limitations and the intended application of the test method.

B. The commissioner shall not require a detection limit study for any component for which spiking solutions or quality control samples are not available.

C. A laboratory must initially determine the detection limit for the compounds of interest in each test method in a matrix in which there are not target analytes or interferences at a concentration that would impact the results or the laboratory must determine the detection limit in the matrix of interest.

D. A laboratory must determine the detection limits each time there is a change in the test method that may affect how the test is performed or when a change in instrumentation occurs that affects the sensitivity of the analysis.

E. A laboratory must include all sample processing steps of the analytical method in the determination of the detection limit.

F. A laboratory must document all procedures used to determine the detection limit, including the matrix type of the sample and all supporting data.

Subp. 8. **Reporting limits.**

A. A laboratory must document all procedures used to determine the reporting limit.

B. A laboratory must establish reporting limits for each field of testing. The reporting limits must be greater than detection limits.

C. A laboratory must verify the reporting limit each time the instrument is calibrated, or monthly at a minimum. The laboratory must analyze a verification standard with a concentration at or below the reporting limit. The percent recovery of the standard must fall within plus or minus 40 percent of the true value.

D. If the percent recovery of the reporting limit verification standard is outside the acceptance criteria, a laboratory must elevate the reporting limit for the associated samples to the concentration of the lowest point, above the zero blank, that meets the acceptance criteria defined in item C. The laboratory must report all samples analyzed after the failed reporting limit check using the elevated reporting limit until a new calibration curve and reporting limit verification standard meet the acceptance criteria.

Subp. 9. **Selectivity.**

A. Absolute retention time and relative retention time aid in identifying components in chromatographic analyses and evaluating the effectiveness of a chromatographic medium to separate constituents. A laboratory must develop and document acceptance criteria for retention time windows if the acceptance criteria are not specified in the approved method.

B. A confirmation must be performed to verify the compound identification when positive results are detected on drinking water. The confirmations must be performed on organic tests, such as pesticides, herbicides, or acid-extractable compounds, or when recommended by the analytical test method, except when the analysis involves the use of a mass spectrometer or Fourier transform infrared spectrometer (FTIR). All confirmations must be documented.

C. A confirmation must be performed to verify the compound identification when positive results are detected on a sample from a location that has not been previously tested. The confirmations must be performed on organic tests, such as pesticides, herbicides, or acid-extractable compounds, or when recommended by the analytical test method, except when the analysis involves the use of a mass spectrometer or Fourier transform infrared spectrometer. A confirmation is not required on positive results for samples analyzed for diesel range organics and gasoline range organics under the underground storage tank program. All confirmations must be documented.

D. A laboratory must document acceptance criteria for mass spectral tuning. The laboratory must ensure that the tuning criteria meets the specifications in the approved method or as established by the client, whichever is more stringent.

Subp. 10. **Manual integrations.** If the integrations are not calculated by the equipment's software, a laboratory must document acceptable use of manual integrations and must have in place a system for review of manual integrations performed to verify adherence to the policies and procedures of the laboratory.

Subp. 11. **Constant and consistent test conditions.**

A. A laboratory must ensure that the test instruments consistently operate within the specifications required of the application for which the equipment is used.

B. A laboratory must ensure that glass and plastic containers are cleaned so that they meet the sensitivity of the test method. Any cleaning and storage procedures that are not specified by the test method must be documented in laboratory records and the laboratory standard operating procedures manual.

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