4740.2093 REQUIREMENTS FOR INSTRUMENT CALIBRATION.

Subpart 1. **Scope.** This part applies to all devices that are the actual test instrument used to quantify the test results.

Subp. 2. **Requirements.**

A. Equipment must be operated by trained personnel. Up-to-date instructions on the use and maintenance of equipment, including any relevant manuals provided by the manufacturer of the equipment, must be readily available for use by the appropriate laboratory personnel.

B. All equipment must be properly maintained, including inspection, calibration, and cleaning. Maintenance procedures must be documented. Calibration of balances, weights, temperature recording devices, light sources, and detectors must be appropriate to the required precision and accuracy of the method. Calibrations must be performed at least annually and must be traceable to appropriate standards.

C. Records must be maintained for each major item of equipment, including software. The records must include:

(1) the identity of the item of equipment, including software;

(2) the manufacturer's name, type identification, and serial number or other unique identification;

(3) documentation that equipment complies with the manufacturer's specification;

(4) the current location within the laboratory;

(5) the manufacturer's instructions, if available;

(6) dates, results, and copies of reports and certificates of all calibrations, adjustments, and acceptance criteria and the due date of the next calibration;

(7) the maintenance plan and maintenance carried out to date, documentation on all routine and nonroutine maintenance activities, and reference material verifications;

(8) any damage, malfunction, modification, or repair to the equipment;

(9) date received and date placed in service or the date on which its first use or repair was recorded; and

(10) if available, condition when received, such as new, used, or reconditioned.

Subp. 3. **Initial calibration.**

A. Sufficient records must be retained to permit reconstruction of the instrument calibration, such as calibration date, approved method, instrument, analysis date, each

analyte name, the manual or electronic identification of the analyst performing the test, concentration and response, calibration curve or response factor, or unique equation or coefficient used to reduce instrument responses to concentration.

B. Sample results must be quantitated from the most recent instrument calibration and may not be quantitated from any instrument calibration verification unless otherwise allowed by permit, program, or rule.

C. All instrument calibrations must be verified with a standard obtained from a second source. Traceability must be to a national standard, when available.

D. Criteria for the acceptance of an instrument calibration must be established, such as correlation coefficient or relative standard deviation. The criteria used must be appropriate to the calibration technique employed and must be documented in the laboratory's standard operating procedure.

E. If allowed in the permit, program, or rule, results of samples outside of the concentration range established by the calibration must be reported with defined qualifiers, flags, or explanations estimating the quantitative error.

F. The following must occur for methods employing standardization with a zero point and a single point calibration standard:

(1) before the analysis of samples, the linear range of the instrument must be established by analyzing a series of standards, one of which must encompass the single point quantitation level;

(2) a zero point and a single point calibration standard must be analyzed with each analytical batch;

(3) a standard corresponding to the reporting limit must be analyzed with each analytical batch and must meet established acceptance criteria as specified under part 4740.2100, subpart 8;

(4) the linearity must be verified at a frequency established by the method or the manufacturer; and

(5) if a sample within an analytical batch produces results above its associated single point standard, then:

(a) a standard with a concentration at or above the analyte concentration in a sample must be analyzed and must meet established acceptance criteria for validating the linearity;

(b) the sample must be diluted such that the result falls below the single point calibration concentration; or

(c) the data must be reported with an appropriate data qualifier or an explanation in the test report.

G. If the instrument calibration results are outside established acceptance criteria, corrective actions must be performed and all associated samples reanalyzed. If reanalysis of the samples is not possible, data associated with an unacceptable instrument calibration must be appropriately qualified on the test report.

H. Calibration standards must include concentrations at or below the limit specified in the permit, program, or rule.

I. If an approved method does not specify the number of calibration standards, the minimum number is three, one of which must be at the reporting limit, not including blanks or a zero standard, with the exception of instrument technology for which it has been established by methodologies and procedures that a zero and a single point standard are appropriate for calibrations. The laboratory must document in its standard operating procedures how it determines the number of points required for the instrument calibration employed, and the acceptance criteria for calibration.

Subp. 4. **Calibration verification.**

A. When an instrument calibration is not performed on the day of analysis, the instrument calibration must be verified before analysis of samples by analyzing a calibration standard with each batch.

B. If calibration verification is not described in the approved method, a calibration verification must be repeated at the beginning and end of each batch.

C. Sufficient raw data records must be retained to permit reconstruction of the calibration verification, such as test method; instrument; analysis date; each analyte name, concentration, and response; calibration curve or response factor; or unique equations or coefficients used to convert instrument responses into concentrations. Calibration verification records must explicitly connect the verification data to the instrument calibration.

D. Criteria for the acceptance of a calibration verification must be established and evaluated using the same technique used to evaluate the instrument calibration.

E. If the calibration verification results obtained are outside established acceptance criteria, corrective actions must be performed. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then the laboratory must either demonstrate performance after corrective action with two consecutive successful calibration verifications or perform a new instrument calibration. If the laboratory has not demonstrated acceptable performance, sample analyses must not occur until a new instrument calibration is established and

verified. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:

(1) when the acceptance criteria for the calibration verification are exceeded high (high bias) and all associated samples contain analytes below the reporting limit, then those sample results may be reported; and

(2) when the acceptance criteria for the calibration verification are exceeded low (low bias), the sample results may be reported if the concentration exceeds a maximum regulatory limit as defined by the permit, program, or rule.

F. When allowed by permit, program, or rule, verification procedures may result in a set of correction factors. If correction factors are employed, the laboratory must have procedures to ensure that copies of all data records, such as in computer software, are correctly updated.

G. Test equipment, including both hardware and software, must be safeguarded from adjustments that would invalidate the test results.

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