

NR 149 Changes



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NR 149 Workgroup-May 2014

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What If...

- Proposed 149.02(7)(f)

When a laboratory incorporates a procedure that is neither expressly permitted nor prohibited by the method, the department shall retain the authority to determine the acceptability of the practice.



Definitions

- Current 149.03(12)

“Aqueous” means a certification or registration matrix designating any aqueous sample that is not a drinking water, and samples with no more than 15% settleable solids.

Note: Samples with more than 10% settleable solids may also be classified as solid.

Definitions

- Proposed 149.03(13)

“Aqueous” means an accreditation matrix that is a single phase water sample and that is not drinking water.



Fees

- Current: NR149.21(4)(c)

Note: Considering base fees, matrix fees, analytical technology fee maximums, and the analytical class fee maximum, this effectively establishes a maximum annual fee “cap” of 100 RVUs for any laboratory.



- Proposed 149.21(5)

(language specifying maximum fees is removed)

Category	Number of Labs	Projected Number of Labs with potential fee increase	% of projected number of labs with potential fee increase	Average fee increase
Commercial	71	23	32%	\$948
Industrial	45	0	0%	-
Municipal	220	0	0%	-
Public Health	15	1	7%	\$894

Max fee increase: \$1,222

Category	Number of Labs	Projected Number of Labs with potential fee decrease	% of projected number of labs with potential fee decrease	Average fee decrease
Commercial	71	48	68%	\$124
Industrial	45	45	100%	47
Municipal	220	220	100%	62
Public Health	15	14	93%	\$78

Max fee decrease: \$266

Laboratory Ethics

- Proposed 149.365



All of the following practices are prohibited and may result in enforcement action as presented in s NR 149.10:

- (1) Fabrication, falsification, or misrepresentation of data.
- (2) Improper instrument clock setting, termed time traveling, or improper recording of date or time.
- (3) Unwarranted manipulation of samples, software, peak integration, or analytical conditions.
- (4) Concealing or failing to report a known improper or unethical behavior or action associated with sample analysis.

Corrective Action

Proposed 149.38 (1)(added)

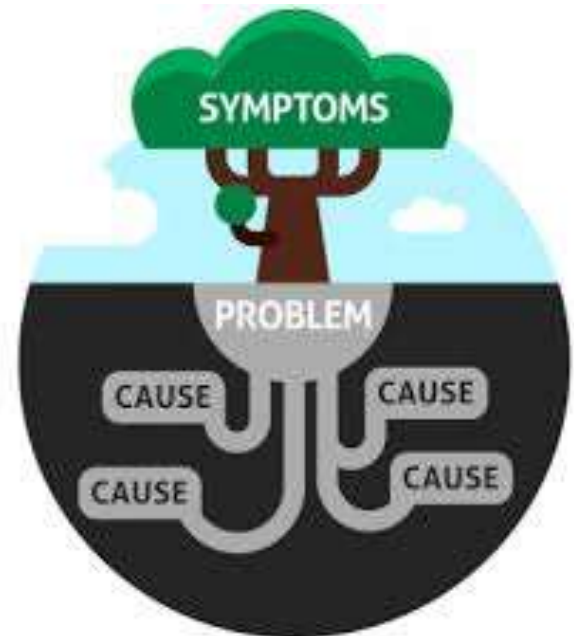
(1)The laboratory shall document, step by step, the procedures that will be taken for corrective action when a quality control sample fails.

- (2)(b) Quality control samples exceed acceptance limits, unless reanalysis of the affected sample resolves the issue.

Corrective Action

Proposed 149.38 (6 added)

(6) Analysts may not always be able to identify the source of single event failures of quality control samples. However, **root cause analysis** shall be performed when there is recurrence.



Reagents & Standards

Proposed 149.39(3)(d) (added)

- The laboratory may not use any standards and reagents beyond the expiration dates, unless the laboratory is using the standard and reagents for qualitative determinations.



Method Selection

Proposed 149.41(3 & 4) (added)

When using methods associated with the methods compendium document, “Test Methods for Evaluating Solid Waste,” the laboratory shall **comply with the minimum requirements** of the methods as written and **state which options** are being implemented when options exist.

The department reserves the right to determine whether or not a **modification** is within the scope of a method.

Cal. & Verification of Support Equipment



- Current 149.44(3)(g)

Analytical balances that have been used at least once in a month shall be checked monthly with at least **2 certified weights**, one weight in the gram range and one weight in the milligram range.

- Proposed 149.44(3)(d)3.

Monthly: balances, with **one weight in the expected range** of use.

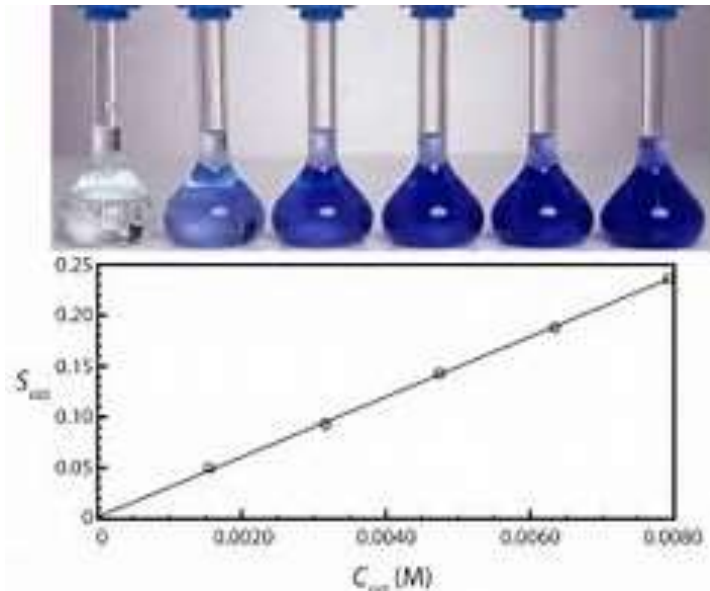
Laboratory Analytical Instruments



- Current 149.44(4)(d)

When analytical instruments **leave the direct control** of the laboratory for maintenance or for any other reason, the laboratory shall **ensure** that the functional and **calibration status** of those analytical instruments are checked or demonstrated to be satisfactory before the instruments are returned to service.

Laboratory Analytical Instruments



- Proposed 149.44(4)(d)

When analytical instruments leave the direct control of the laboratory for maintenance or for any other reason, the laboratory shall ensure that the instruments are functional and that a new initial calibration has passed, to demonstrate that the instruments are in satisfactory working order before returned to service.

Instrument Calibration



- Current 149.44(5)(a)

All analytical instruments shall be calibrated at least **once in any year** in which they have been used, and shall be calibrated or their calibration verified before they are used to provide any quantitative results.

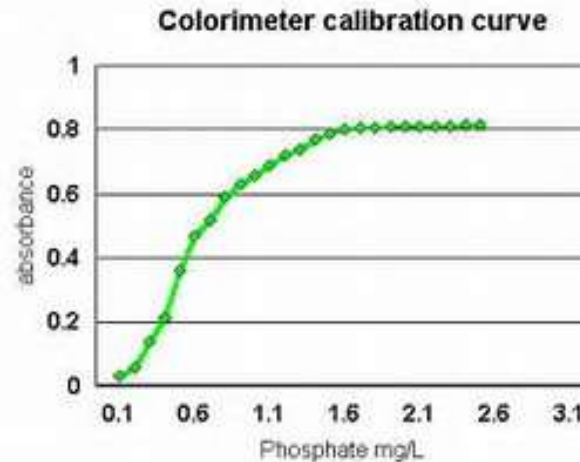
Instrument Calibration



- Proposed 149.446

When an initial instrument calibration is not performed on the day of analysis, the continuing validity of the initial calibration **shall be verified** prior to analyzing any batch quality control or environmental samples...

Instrument Calibration



- Current 149.44(6)(f)(2) (highlight deleted)

The laboratory shall use the simplest linear calibration function unless it has documentation that a non-linear function provides a statistically improved definition of the calibration range.

Non-linear functions may not be used to compensate for instrument saturation, insensitivity, or malfunction.

Instrument Calibration



- Current 149.44(6)(g)5.(h)

The laboratory shall establish procedures for **zeroing an instrument** and the treatment of calibration blanks, when the referenced analytical method used by the laboratory requires the response of a calibration blank to be part of a calibration function.

Instrument Calibration

- Proposed 149.444(1)(e)

For **colorimetric** technologies, the laboratory **may not use a method blank to zero** the instrument.

Note: For colorimetric technologies, other than those based on chemistries when the instrument response varies inversely with concentration, the instrument **should be zeroed with the solvent of interest which is generally reagent water.**

Instrument Calibration

- Current 149.44(6)(i)

...verify initial calibrations... with a second source standard, unless ...

- Proposed – Remove exceptions

(1)An instrument is calibrated by tuning it to conform to a universally accepted scientific law or scale, as is the case with pH meters, ion selective electrodes and dissolved oxygen meters.

Instrument Calibration



- Current 149.44(6)(i)
...verify initial calibrations... with a
second source standard, unless ...
- Proposed – Remove exceptions
(2)The laboratory analyzes **quality control standards** for the
analyte or analyte group involved and evaluates them ...

Instrument Calibration



- Proposed 149.444(1)(f)

The laboratory **may not utilize pre-programmed initial calibrations**, provided by the instrument manufacturer, for compliance testing.

Instrument Calibration



- Proposed 149.444(1)(h & i)

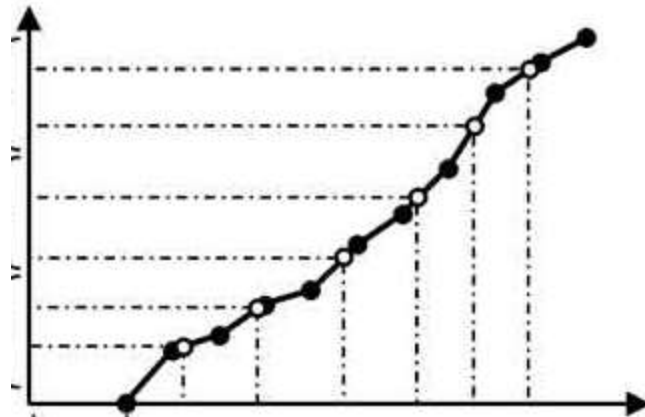
For initial calibrations used to determine the **LOD**, the laboratory shall **use the same number and concentration levels of standards, as** that used for **routine sample analysis**, except as allowed in s NR 149.444 (5). [when to exclude calibration points]

When required by method, the laboratory shall **process each calibration standard in the same manner** as samples.

Instrument Calibration

- Proposed 149.444(1)(j)

Point to point calibrations are **not allowed** unless otherwise specified in this chapter.



Initial Calibration-Minimum Number of Standards

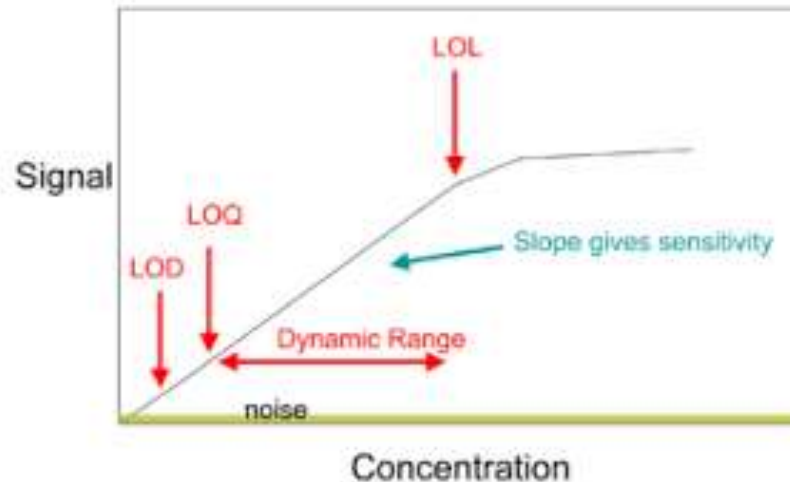
- Current 149.44(6)(d)

The minimum number of non-zero standard concentrations selected to establish calibration shall be three except for all of the following:

- Proposed 149.444(2)(b) (added conductivity)

Conductivity meters, for which the minimum shall be one. Conductivity meters shall be calibrated by verifying the cell constant or adjusting the meter based on the analysis of a potassium chloride standard solution.

Initial Calibration



- Proposed 149.444(4)(e)

The lowest concentration standard in the initial calibration cannot be more than two times the concentration of the LOQ.

Initial Calibration

- Proposed 149.444(5)

INITIAL CALIBRATION – EXCLUDING CALIBRATION

POINTS. If one or more calibration standards are excluded from the calibration all of the following criteria shall be met:

- (a) The rationale for the exclusion is documented.
- (b) Any regulatory required detection limits can still be met.
- (c) If the standard being excluded is not the highest standard, it shall be removed for all analytes in that standard.
- (d) If the highest calibration standard is removed, the linear range shall be limited to the remaining high standard concentration.

Sample Preservation

- Proposed: 149.442(3)(b) (highlight added)
...a non-drinking water sample... The preservation status of a non-drinking water sample may be recorded as “received on ice” only if solid ice is present around the sample when the sample is received at the laboratory. The preservation status of a sample preserved with ice packs, such as “blue ice,” may not be recorded as “received on ice.”



Reports



- Current: 149.47 (1)(e)1

The **name**, **address** and **telephone** of the laboratory where tests were performed, as well as the name of a **contact**.

- Proposed: 149.47(2)(d)1

The **name of the laboratory** where the tests were performed.

Reports

- Current: 149.47 (1)(e)

7. The date of receipt of the samples.

8. For samples submitted to pretreatment steps, such as digestions or extractions, with identified holding times in department regulations, the date in which such steps were performed.

9. The date of analysis.

- Proposed: 149.47(2)(d)6

The dates of analysis, extraction, or digestion, when a holding time has been established for the preparation step.

Reports

- Current: 149.47 (1)(e)10.b

For sample results reported on a **dry weight** basis, the **solids content** and a statement **or flag** indicating that results have been adjusted for the solids content of the corresponding samples.

- Proposed: 149.47(2)(d)7

When **non-aqueous sample results** are reported, the laboratory shall indicate whether the non-aqueous sample results were reported on a **dry weight or wet weight** basis.

Reports



- Current: 149.47 (1)(e)12

The names and signatures of **responsible parties** authorizing reported results.

- Proposed: removed

Quality Control Requirements



- Proposed: 149.48(1)(c) (added)

A laboratory shall **document deviations** from the laboratory's quality system or exceedances of quality control samples, and to the extent the department's data systems allow, be **communicated with the results** to the department.

Limit of Detection

- Current: 149.48(2)(d)



Limits of detection shall be determined at least **annually** unless a laboratory can verify the continued applicability of a previously determined limit of detection by an established and defensible protocol.

Limit of Detection

- Proposed: 149.48(2)(c,f)

The LOD shall be adjusted when the sample amounts used are different than those used for the LOD determination.

- The laboratory shall have a procedure to verify that the LOD is realistically achievable.
- Note: See the determinative LOD guidance provided on the Wisconsin department of natural resources laboratory accreditation program website.

Limit of Detection

- Proposed: 149.48(2)(g,h)

If an annual LOD verification determines that the LOD is within 0.5 to 2 times the existing LOD the laboratory may continue to use the existing LOD.

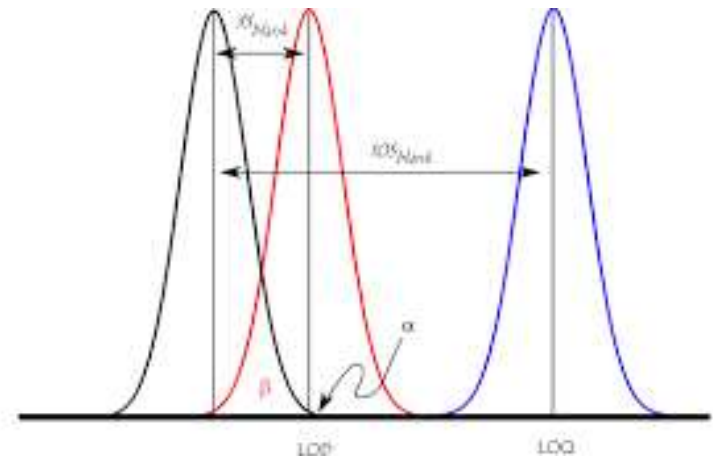
Limit of Quantitation

- Current: 149.48(2)(f&g)

Laboratories shall establish procedures to relate limits of detection to limits of quantitation.

Established limits of quantitation shall be above determined limits of detection.

Limit of Quantitation



- Proposed: 149.48(3)(a,b)

A laboratory shall establish the LOQ for all tests performed except for those exempted from an LOD under s NR 149.48 (2) (a).

The LOQ shall be established as $10/3$ the LOD.

Reporting Limit

- Proposed: 149.48(4)
- Reporting limits are reserved for those analytes for which LODs are not appropriate.
- For biochemical oxygen demand and carbonaceous biochemical oxygen demand, the minimum reporting limit is 2 mg/L which is based on a 300 mL sample volume. When no dilution is equal to 300 mL, the reporting limit shall be adjusted based on the lowest dilution reported.
- For total suspended solids, the reporting limit shall be determined using the following formula: Reporting Limit (mg/L) = $1000 / (\text{sample volume filtered in mL})$.

Method Blanks

- Proposed: 149.48(5)(b)

The laboratory shall process method blanks at a frequency of at least one per preparation batch up to 20 environmental samples in a batch. When samples are analyzed by methods that do not require a preparation step before analysis, a blank shall be analyzed at the frequency of one per analytical batch up to 20 environmental samples.

Method Blanks

- Proposed: 149.48(5)(c)

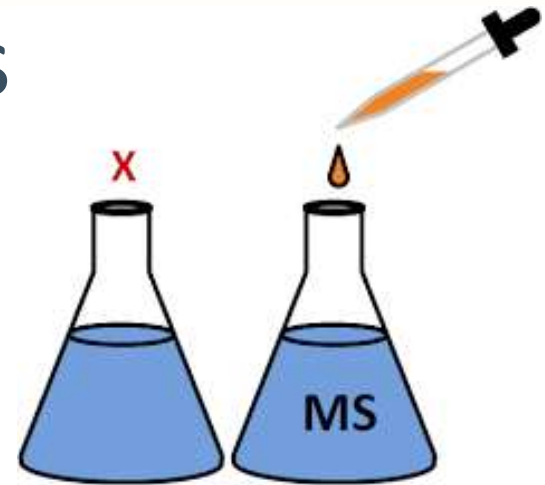
Whenever the **absolute value** of the concentration of the method blank contains analytes of interest **greater than the LOD**, the laboratory shall evaluate the nature of the interference and its effect on each sample in a preparation batch.

Laboratory Control Samples

- Proposed: 149.48(6)(a)

Unless otherwise exempted by this subsection, the laboratory shall process an LCS at a frequency of at least one sample per preparation batch up to 20 environmental samples, along with and under the same conditions as the associated samples in a preparation batch. These conditions shall include all sample preparation steps, except for leaching procedure extractions.

Matrix Spikes



- Current: 149.48(4)(d)

Matrix spikes or certified reference materials may be processed for all reported analytes, at the frequency described in par. (a), in place of laboratory control samples...

- Proposed: 149.48(6)(d)

The **LCS** shall be fortified with the analytes specified by regulation, method, covered program or all reported analytes, except as allowed in par (e).

Laboratory Control Samples – Acceptance Criteria



- Proposed: 149.48(7)(b) (new)

For **biochemical oxygen demand** and carbonaceous biochemical oxygen demand, the LCS is prepared based on a mixture of 150 mg/L each, of glucose and glutamic acid. The acceptance criteria for the LCS shall be 198 plus or minus 30.5 mg/L or **167.5 to 228.5** mg/L.

Laboratory Control Samples – Acceptance Criteria

- Proposed: 149.48(7)(c) (new)

When the method, regulation, or covered program do not specify acceptance limits, the laboratory shall evaluate LCS recoveries and generate control limits, following exclusion of outliers using a recognized statistical technique, using the mean plus or minus 3 times the sample standard deviation.

Laboratory Control Samples – Acceptance Criteria

- Proposed: 149.48(7)(d,e) (new)

Annually, the laboratory shall review its in-house generated quality control limits and update those limits whenever the performance characteristics change. When a laboratory generates its own quality control limits using historical data, the laboratory shall perform a recognized statistical outlier determination and any identified outliers shall be removed from the data set prior to calculating quality control limits.

In lieu of using calculated quality control limits, the laboratory may opt to use the CCV standard limits.

These all removed:

- 149.48(5) Quality Control Standards
- 149.48(6) Matrix Spikes and Matrix Spike Duplicates
- 149.48(7) Sample Replicates
- 149.48(8) Surrogate Spikes



New Section: Technology Requirements

BOD: 149.50(1)

- 17 to 23 degrees
- Assess supersaturation (and treat) each day of analysis
- Wide bore pipet tips
- GGA – no averaging
- Seed disinfected and inhibited samples

BOD (continued)

- Dilutions to expect 2 mg/L depletion
- Equipment with multiple D.O. probes – calibrate each
- Optical D.O. probes – calibrate each day
- Barometric pressure – local, not adjusted to sea level
- Chlorine strips to 0.1 mg/L

Technology: Colorimetric or Turbidimetric 149.50(2)

- Initial calibration – use cal blanks, assign the measured response
- Digestion, closed vial – 150 for 30 min
- Digestion, open vial – boil 30 min or reduced by 80% without going dry
- No method blank subtraction
- Cl₂ strips 0.1 mg/L, sulfide strips to 10 mg/L

Colorimetric or Turbidimetric (continued)

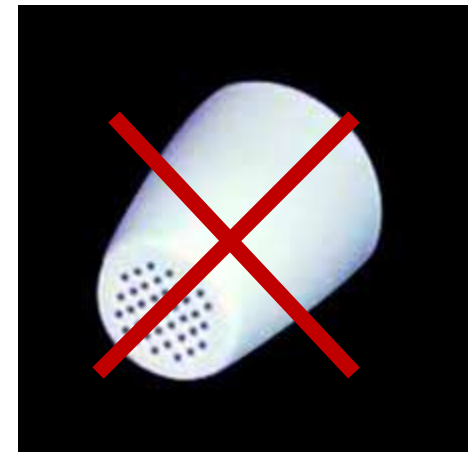
- Do not dilute samples after adding color reagent

Technology: Ion Selective Electrode 149.50(3)

- Initial calibration each day of analysis
- Use an Initial Calibration Verification to verify each Initial Calibration, except for pH
- Slope criteria 54-60 mV
- Slope criteria for high performance probes is 54-65 mV
- Conductivity-determine cell constant annually

Technology: Gravimetric-Solids 149.50(4)

- No Buchner funnels or Gooch crucibles for TSS or TDS
- Use wide-bore pipet tips



Technology: Titration 149.50(6)

If method required standardization of titrants, standardize monthly, unless:

- Unused titrant is never poured back into original container
- Titrant is always protected from light
- LCS recovery of 90 to 110% is achieved

Summary

- Balances – 1 weight in expected range
- Removing cap on RVUs for calculating fees
- Instrument calibration – verify prior to use, do not need to recalibrate yearly
- Do not use a method blank to zero your instrument

Summary

- No pre-programmed instrument calibrations
- Calibration low standard – no more than 2X the LOQ
- If certified, we removed some required elements on the results report
- Realistic LODs – guidance on our website

Summary

- Don't have to change your LOD every year, if 0.5 – 2X current LOD
- LOQ is $10/3$ of the LOD
- A batch is defined as 20 samples. New method blank every 20 samples.

Summary

- Pay attention to negative numbers on Method Blanks. Absolute value must not exceed LOD.
- BOD – GGA criteria 198 ± 30.5 , and no averaging. One GGA is sufficient.

Possible Timeline

- February 2018 - To DNR Board for approval
- Spring 2018 – Legislative approval
- September 1, 2018 – Effective date