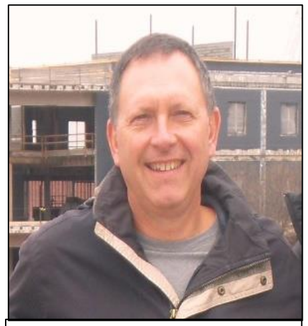
WISCONSIN DEPARTMENT OF NATURAL RESOURCES

NR 149 UPDATES *for* Certified labs

July 2021 DNR.WI.GOV

Intro to Lab Certification Staff



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“New” NR 149

Published NR 149 in March 2021

Effective July 1, 2021

Enforcing September 1, 2021

Website: **https://dnr.wisconsin.gov/topic/LabCert**

Out with the OLD and in with the NEW

As technology and standards change…

…some things are no longer useful and have been removed

The new stuff –

- More direct language
- Changing with the times

We'll start with the **NEW** stuff, so labs are aware of upcoming changes and have time to prepare.

NR 149 changes for CERTIFIED labs

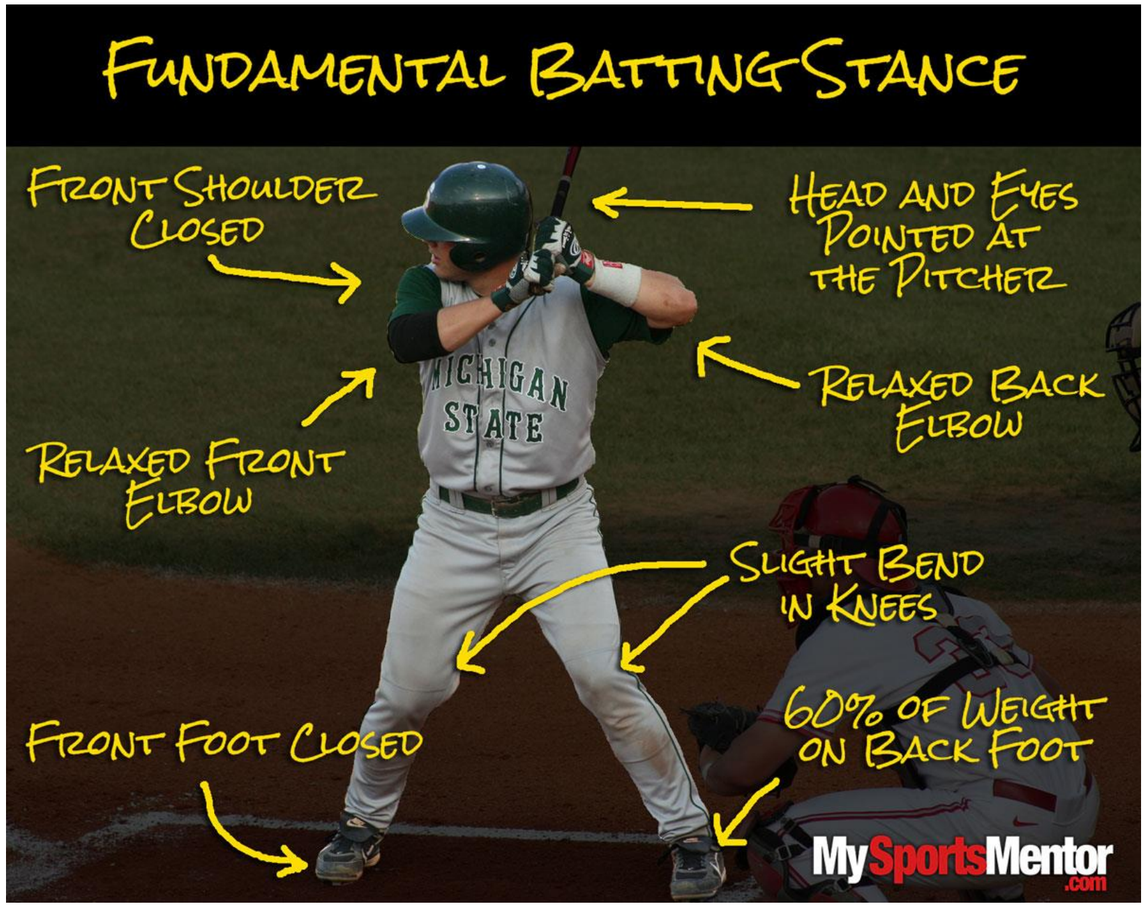
If a laboratory analyzes compliance samples “for hire” or analyzes drinking water compliance samples (NR 809 or NR 812), the laboratory must be certified, not registered.

The changes presented in this PowerPoint apply to **certified** laboratories.

*There is a different presentation specific to **registered** wastewater laboratories on the DNR website.*

So what do LABs need to do to prepare?

- Sample Handling
- PTs
- Calibrations
- LOD/LOQ/RL
- Quality Systems
- Test Reports
- Technology Info



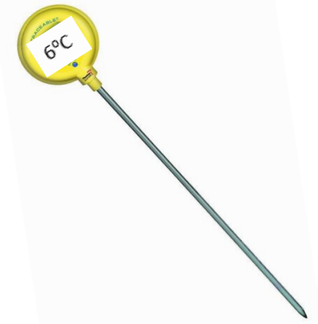
Sample Handling

Most of the sample acceptance and handling requirements in the previous code are the same in the 2021 update, but there are a few differences.

The temperature check requirement may be **NEW** to some labs that accept compliance samples:

NEW

Temperatures must be taken when samples are received **if** thermal preservation is required.



Check and document the temperature. Temp °C:_____

Sample Handling - Thermal Preservation

What to do if the temperature upon receipt is above 6°C:

If the samples are received at the lab on the same day the samples were collected, if they are received on ice (ROI), and if they were put on ice at collection, then the results do not need to include a qualifier.

- ❑ 1. Document if ice present (ROI).
- ❑ 2. Know that samples were placed on ice at the time of collection.

If either 1 or 2 is not true, then….

Sample Handling - Thermal Preservation

NR 809: If the sample is a public water supply drinking water compliance sample, the result may not be used for compliance; therefore, it is likely that a new sample will need to be collected.

Not NR 809: If the sample is **not** an NR 809 drinking water compliance sample, the lab may report the non-drinking water sample results if the client wants the results, and the results are qualified. For example…

"The temperature requirement upon receipt of above freezing to 6°C was not met."

Sample Handling - Hold Time/Receipt Time

NR 809: If the sample is a public water supply drinking water sample, a new sample will need to be collected if the hold time is not met.

NR 812: If the sample is a potable well compliance sample, a new sample will need to be collected if the sample is not received at the primary lab within 48 hours of collection.

Not NR 809 or 812: The lab may report the results when the client wants the results, and the results are qualified. For example…

"The sample holding time of 48 hours was not met."

Sample Handling

How to find the hold time and preservation regulations :

NR 809 (public drinking water): Go to NR 809.113, Table B, NR 809.203 Table D, and NR 809.311 (3).

https://docs.legis.wisconsin.gov/code/admin_code/nr/800/809/

NR 812 (private wells): Go to NR 812.46.

https://docs.legis.wisconsin.gov/code/admin_code/nr/800/812

NR 219 (WW, biosolids, and other covered programs): Go to NR 219.04 (2) and Table F.

https://docs.legis.wisconsin.gov/code/admin_code/nr/200/219.pdf

Proficiency Testing (PT) Updates

PT Updates

DUE DATE IS AUGUST 31 … *get 16 more days*

Need to report the correct / proper method code.

If the method code is not correct, that counts as a **FAIL…**

…which means you'll need a **NEW PT.**

WP PT Method Code Info (3/17/21)

The following tables have methods codes for some common analyte/method combinations for wastewater for **WP** (water pollution) PT sample results.

If you are not using any of these methods or have other questions, please contact Tom Trainor to get the appropriate method code.

WP PT Method Code Info (3/17/21)

BOD Method	Code
SM 5210B (21st Edition)	20135006
SM 5210B (22nd Edition)	20135017
SM 5210B – 2001	20135255
SM 5210B – 2011	20135266

TSS Method	Code
SM 2540D (21st Edition)	20051007
SM 2540D (22nd Edition)	20051018
SM 2540D – 1997	20051201
SM 2540D – 2011	20051212
USGS I-3765-85	40011209

Typical methods for WWTPs

WP PT Method Code Info (3/17/21)

Phosphorus Method	Code
SM 4500-P E (20th Ed)	20123802
SM 4500-P E (21st Ed)	20124009
SM 4500-P E (22nd Ed)	20124010
SM 4500-P E (23rd Ed)	20124032
SM 4500-P E – 1999	20124214
SM 4500-P E – 2011	20124225
Hach 10210 TP 2008 5th Ed	60005121
Hach 10210 TP 2014 8th Ed	60005143
Hach 8190 TP 5th Ed	60003909
EPA 365.3 – 1978	10070801

Ammonia Method	Code
SM 4500-NH3 D (21st Ed)	20109200
SM 4500-NH3 D (22nd Ed)	20109211
SM 4500-NH3 D – 1997	20109404
SM 4500-NH3 D – 2011	20109415
Hach 10205 NH3 2008 5th Ed	60005007
Hach 10205 NH3 2014 8th Ed	60005018
EPA 350.1 – 1993	10063602

Typical methods for WWTPs

PT Updates

The previous exception to requiring PTs for FLAA and low-level analytes has been removed.

If the lab is certified for **Hg by CVAFS** or **metals by FLAA, the lab must analyze and pass a PT each year** from an approved PT provider.

As a reminder, WP PTs still cover the requirements for non-aqueous matrices.

Ignitability requires a RCRA/HW program PT – this PT will be a solvent, not solid.

PT Method Code Info: general (3/17/21)

Method codes for other analytes may be found on the website below; however, please feel free to contact Tom Trainor.

https://lams.nelac-institute.org/TestMethods

PT Updates - WP (Aqueous and Non-Aqueous Matrices)

Repeating…repeating…PT failures *for renewal*

If there are **3** water pollution (WP) PTs in a row that did not pass, that means you need to pass **2 PTs** that are…

- **2 passes in a ROW**
- **2 unique WP studies**
- **10 days apart**
- **Not in the same batch**

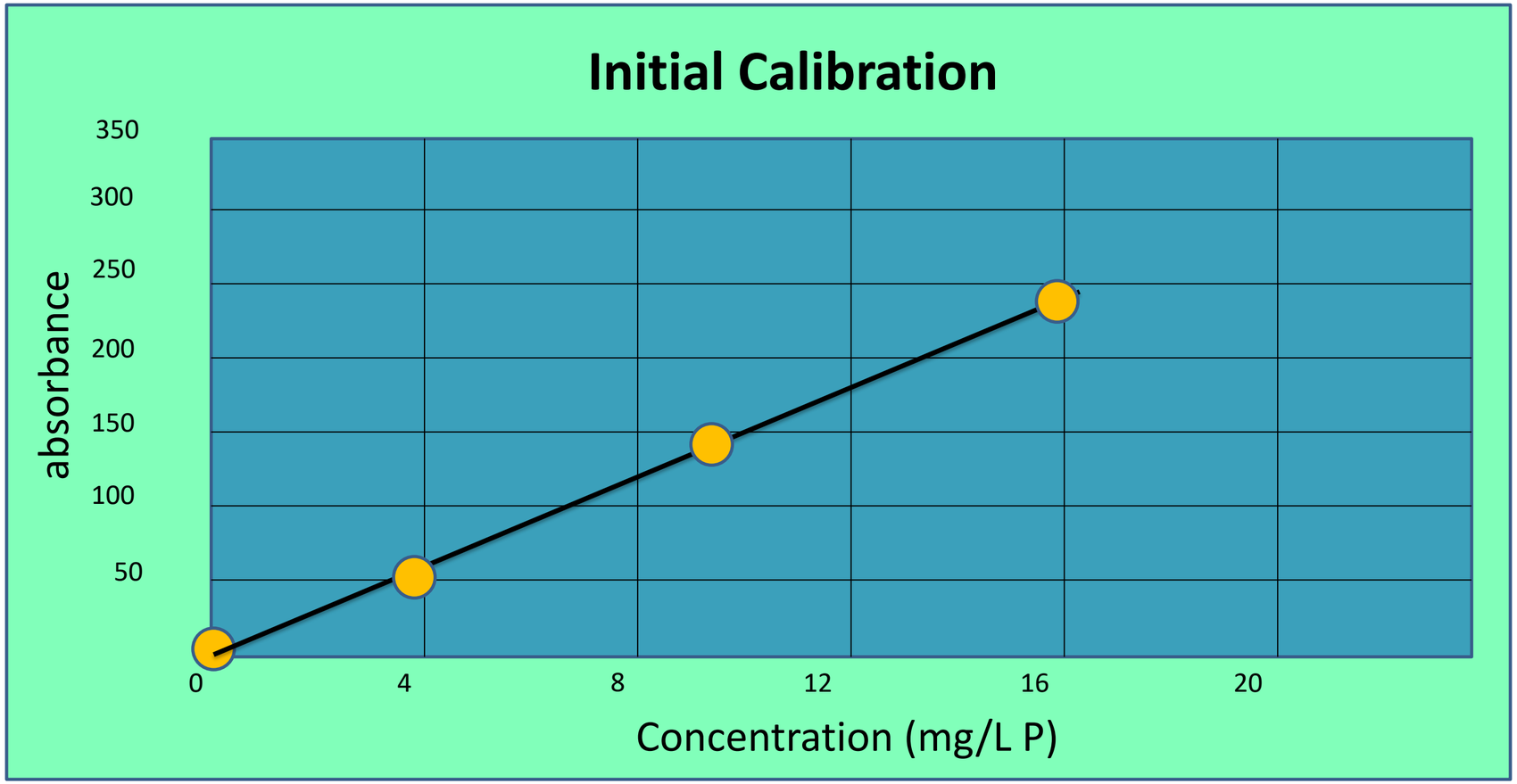
PT Updates - WS (Drinking Water Matrix)

PT failures *for renewal of certification for Drinking Water methods*

If there are **2** water supply (WS) PTs in a row that did not pass, that means you need to **pass 2 WS PTs** that are...

- ❑ From 2 unique WS studies
- ❑ Received at least 10 days apart
- ❑ Reported consecutively
- ❑ Not prepared/analyzed in the same batch

Calibration (initial and continuing)



Calibration information applies to both certified and registered laboratories

Initial Calibration (curves)

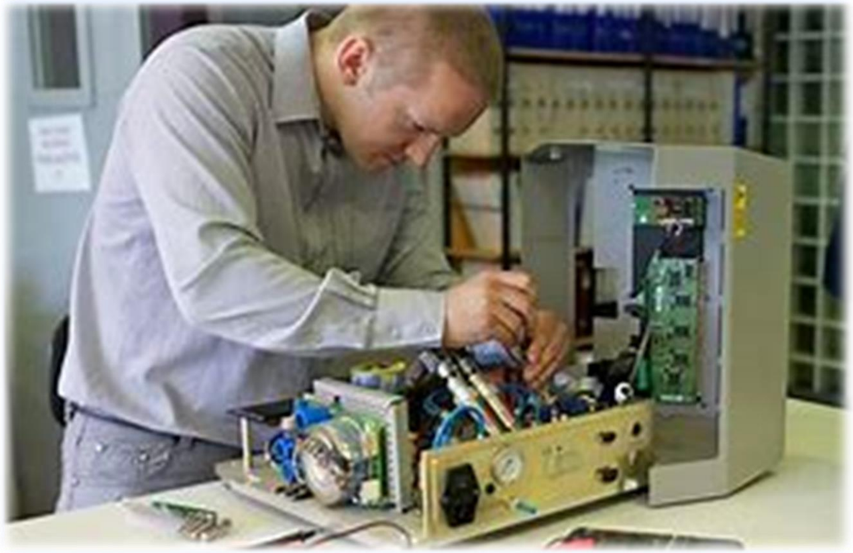
NEW

Calibration curves do **NOT** need to be done annually.

Factory calibration curves are (still) **NOT** acceptable for accredited tests for compliance.

Need a new calibration curve if there is **NON-ROUTINE MAINTENANCE** or the instrument behavior has changed.

Will need a new calibration curve if the instrument leaves the laboratory.



Calibration

Need a new calibration curve if the continuing calibration verification **(CCV)** standard **FAILS**

You have the option to **rerun the CCV, but…**

- it has to be analyzed immediately and under the same conditions, and…
- if the 2nd CCV fails, then **(NEED A NEW CALIBRATION CURVE).**

Calibration - Ion Selective Electrode (ISE)

Calibration Curves required **DAILY** – applies to <u>all</u> ISE: BOD, NH_3, pH, etc.

Calibration Std Concentrations (mg/L)	Measured mV
0.20	116.8
2.00	60.8
20.00	3.4

NEED **3** calibration standards for ISE *(except for BOD and pH)*

NEED a **2nd** source initial calibration verification **(ICV)** standard (*except for BOD and pH)*

NEW

Calibration (some EXTRA stuff)

Calibration

NEW

The ICV and CCVs need to be treated like the calibration standards. If the initial calibration was processed, process each calibration standard, ICV, ICB, CCV, and CCB in the same manner. *Applies when ICBs, CCVs, and CCBs are required.*

Calibration blanks:

The ICB needs to be less than the LOD.

The CCB needs to be processed at same frequency as the CCV and evaluated similarly to the MB (<LOD, <5% regulatory limit, or <10% sample concentration).

Applies when ICB or CCBs are required.

Excluding calibration points: Need to have a documented reason explaining why a point was removed, still need to meet regulatory limits, and need to limit the linear range for that calibration to the (next) highest standard.

Calibration

Reduction of calibration data includes the following:

*(these would apply **unless** the approved method has different criteria)*

- When average response factors are used, the RSD of the response factors may not exceed 20%.
- When linear regression or least squares analysis is used, the correlation coefficient (r) shall be at least 0.995 for inorganic analytes or 0.99 for organic analytes.
- When quadratic or cubic analysis is used, the coefficient of determination (r^2) shall be at least 0.995 for inorganic analytes or 0.99 for organic analytes.

Calibration

NEW

Evaluating calibration – additional and optional criteria:

(unless the approved method has different criteria pertaining to RSE and to residuals)

- When the x-intercept is used, the value may not exceed the LOD.
- When RSE is used, the RSD may not exceed 15% for inorganic analytes or 20% for organic analytes.
- When residuals of each calibration standard are used for inorganic analytes, the recovery must be 90-110% recovery, except for the low standard which must be 80-120%.
- When residuals of each calibration standard are used for organic analytes, the recovery must be 70-130% recovery, except for the low standard which must be 50-150%.

Calibration

What do residuals (read backs) look like?

Example includes criteria for inorganic analytes (optional):

Sample	pH adjust 6-8 (Y/N)?	Sample Vol. (mLs)	Sample + DI Vol. (mLs)	Dilution Factor	Absorbance	Total Phosphorus (mg/L)	Final Total Phosphorus (mg/L)	True Value (mg/L)	Quality Control
Calibration Blank	**Y**	0	2	1	**0.066**	***-0.012***		**0.00**	
Standard 1	**Y**	0.05	2	1	**0.113**	***0.054***	*RF= 2.260*	**0.05**	108.8%
Standard 2	**Y**	0.1	2	1	**0.148**	***0.104***	*RF= 1.480*	**0.10**	103.7%
Standard 3	**Y**	0.3	2	1	**0.296**	***0.312***	*RF= 0.987*	**0.30**	104.1%
Standard 4	**Y**	0.5	2	1	**0.427**	***0.497***	*RF= 0.854*	**0.50**	99.3%
Standard 5	**Y**	0.8	2	1	**0.633**	***0.787***	*RF= 0.791*	**0.80**	98.4%
Standard 6	**Y**	1	2	1	**0.790**	***1.008***	*RF= 0.790*	**1.00**	100.8%

Calibration

Unless otherwise required by method or other regulation…

Once a calibration model/function is selected for use, the lab may not change the model after the associated samples have been analyzed without performing a new initial calibration.

The NR 149 recovery limits for the ICV and CCV apply when the method does not specify limits.
For inorganics, the recovery limit is 90-110%.
For organics, it is 80-120%. NEW

Also, CCV standards need to contain all the analytes that will be reported except for multi–peak analytes – multi-peak CCVs are needed when the analyte is detected in the sample. *This is required for compliance samples and may be in addition to the method specifications.* NEW

Calibration

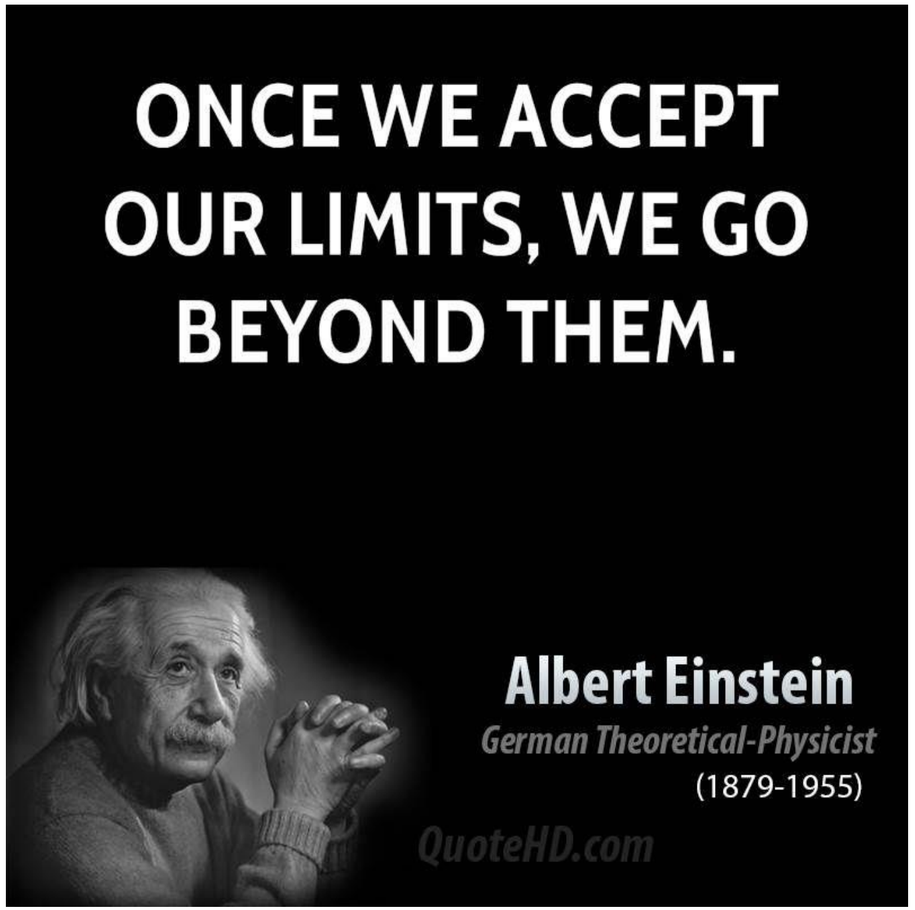
Continuing calibration verification (CCV) shall be performed after the consecutive analysis of each group of 20 environmental samples. *Unless otherwise required in the method/regulation.*

Quadratic model calibration functions require only 1 CCV now, instead of 2 CCVs at two different concentrations.

Cubic models will require 2 CCVs at <u>two different concentrations</u> instead of 3.

Easy....

LOD, LOQ, and RL Updates



E = ?

LOD, LOQ, and RL Updates

Regulatory limits, where possible, must be met for the LOD and LOQ.

For example, if the permit limit is 0.040 mg/L (40 ug/L), the limit of detection needs to be 0.040 mg/L or lower.

Certified laboratories that analyze drinking water compliance samples for analytes with a maximum contaminant level (MCL) must be able to meet those limits. Refer to the website below to access the drinking water and groundwater limits:
https://dnr.wisconsin.gov/topic/DrinkingWater/HealthAdvisoryLevels.html

The required LOD for vinyl chloride in drinking water has been updated to 0.0002 mg/L (the previous code LOD limit was 0.0003 mg/L by mistake).

LOD and LOQ Updates

1. Limit of Detection (LOD) – determined by "new" procedure

If the sample is adjusted from how the LOD procedure was done, then the LOD shall be adjusted.

2. Limit of Quantitation (LOQ)

- The LOQ shall be equal to 10/3 x LOD **or** set to the lowest concentration standard in your curve.
- For single point calibrations for ICP and ICPMS, the LOQ shall be either 10/3 the LOD **or** the lower limit of quantitation.
- Adjust the LOQ by dilution factors when samples are diluted.

Adjustments are any dilutions or sample amounts that were used differently than used during the LOD studies.

Reporting Limit (RL) Updates

RL

3. Reporting Limits (RL) – applies to BOD and TSS

The BOD RL is equal to 2 mg/L if a 300 mL sample was run.

If a 300 mL sample was run and there was no measured BOD above 2 mg/L, then report <2 mg/L.

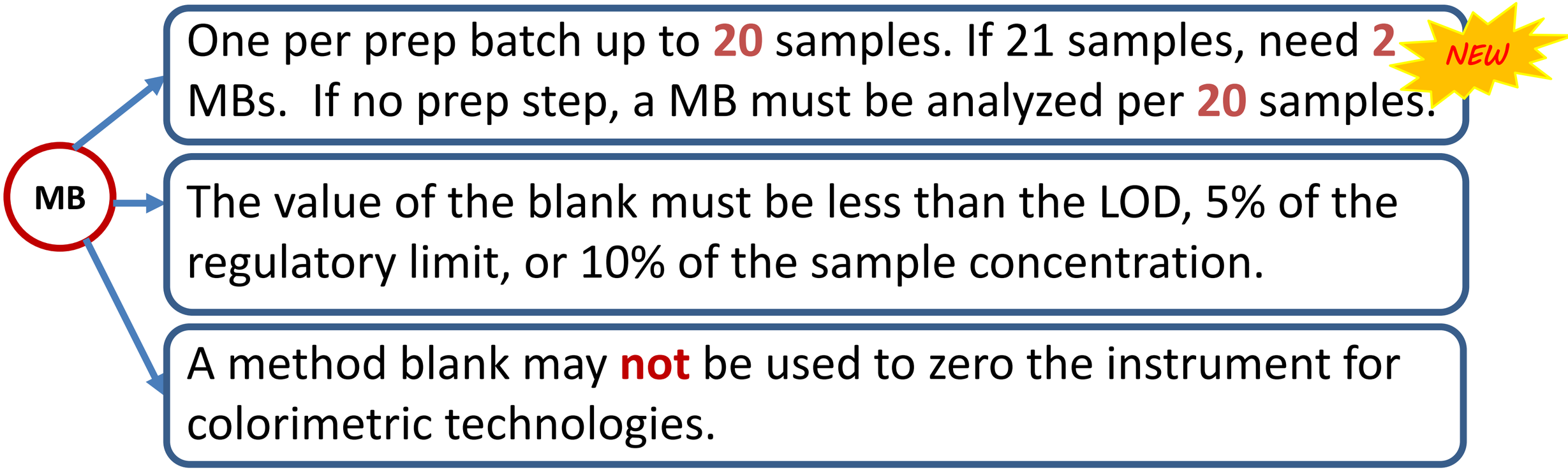
The TSS RL is equal to 2 mg/L if a 500 mL sample was run (RL = 1000/sample volume in mL).

If a 500 mL sample was run and the result is less than 2 mg/L, then report <2 mg/L.

Method Blanks, LCSs, Quality Systems

Method Blanks

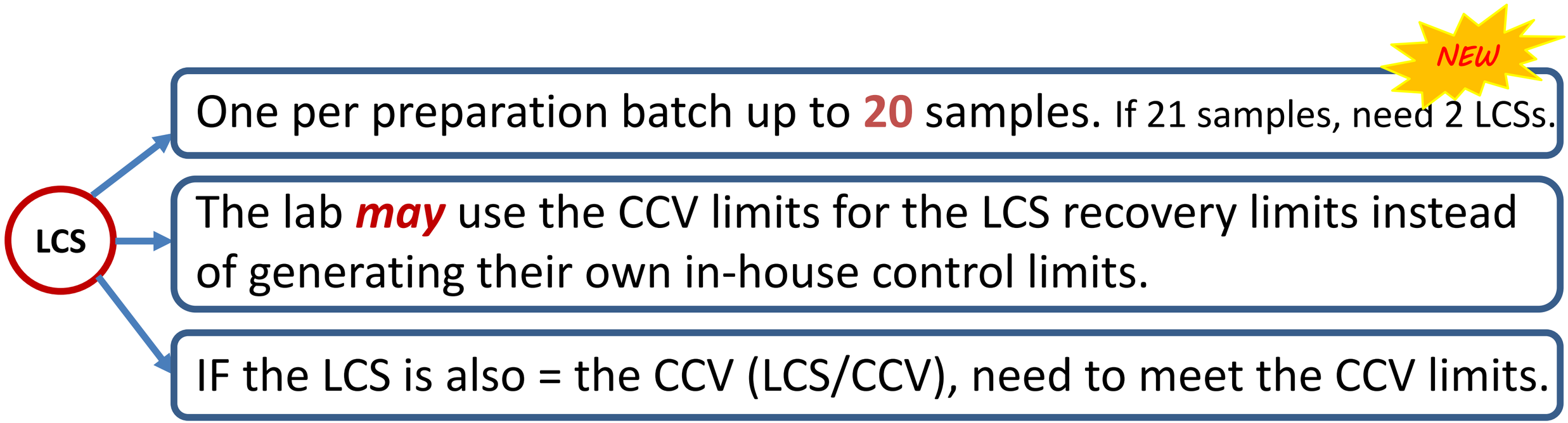
Method Blank (MB) – *not required for pH, alkalinity, acidity, conductivity, or solids (except a MB for alkalinity is required for WET testing)*



LCSs

Lab Control Sample (LCS) – *not required for pH, solids, chlorophyll, color*

(BOD: if ≤20 samples in a week, need 1 GGA/wk; if >20 samples in a week, need to process 1 GGA per analytical batch)



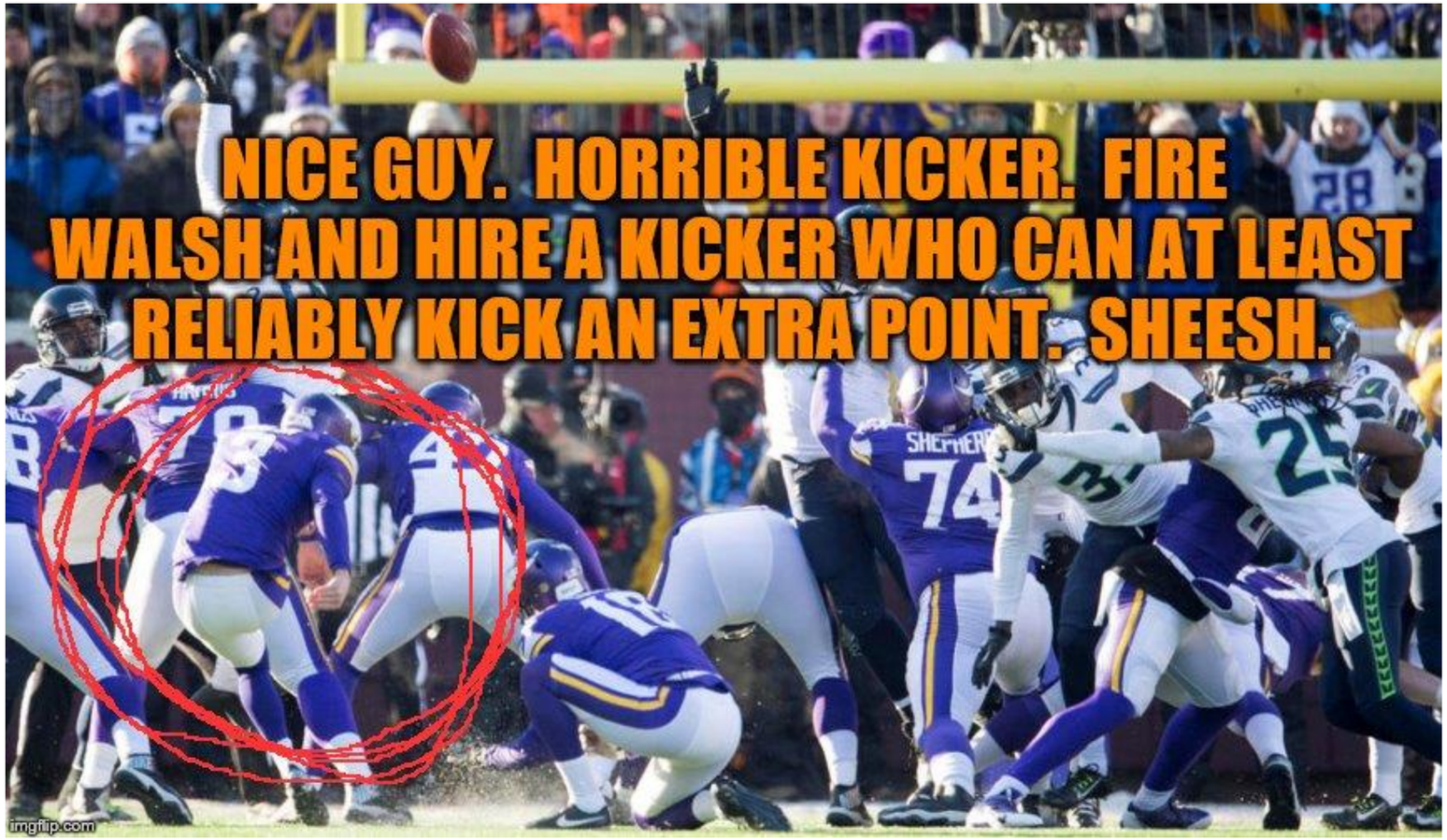
Quality Systems

Corrective Action:

Root cause analysis shall be performed when there is **recurrence.**

At times, all labs may experience repeating issues.

2 PTs in a row fail?
GGAs repeatedly fail each spring?
Oven can't keep temperature?



Quality Systems

Remember the slide about the multiple (3) PT failures? If that was more thoroughly investigated as to what the cause of the failures were, it likely would have prevented a 3rd PT failure.

If previous corrective actions have not prevented these repeated problems, then a more in-depth investigation is needed.

Remember, the Lab Cert staff are here to help.

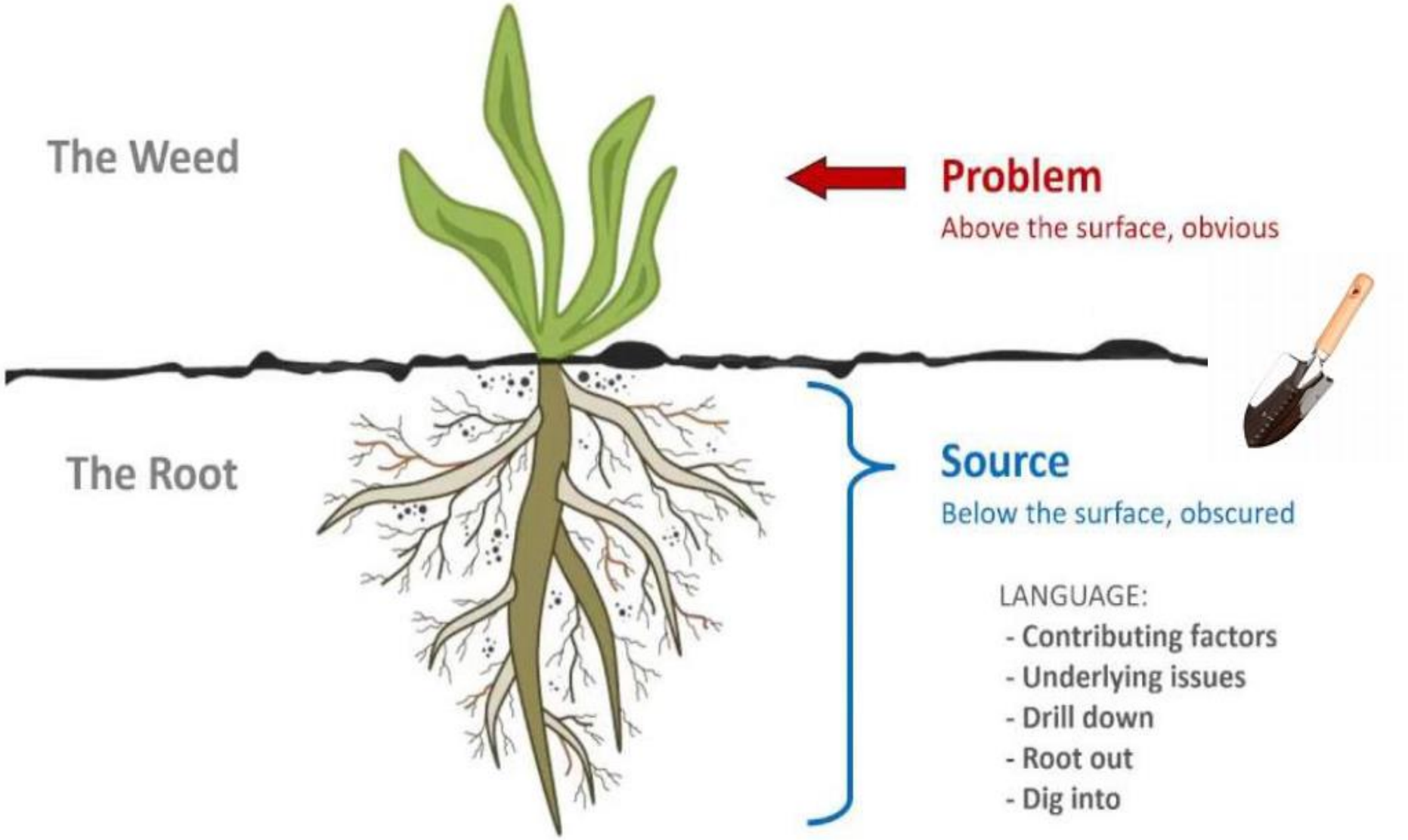
Root cause – in depth…

Quality Systems

Root Cause

Why?
Why?
Why?
Why?
Why?

Root Cause Analysis - The Concept



Quality Systems

Problem: **The total phosphorus PT failed twice in a row.**

1.Why? 1. After looking at the data, both results had absorbances above those of the highest standard in the curve.
2.Why? 2. The analyst didn’t dilute and rerun the samples to get the response within the curve.
3.Why? 3. The analyst was new and didn’t know that was needed.
4.Why? 4. During training, the new technician does not remember being told that this was needed.
5.Why? 5. The requirement to dilute and rerun wasn’t in the SOP.

Corrective action: **Update the SOP with this requirement.**

Quality Systems

Root causes – things to keep in mind: When doing a root cause investigation (such as the “5 Whys”), keep in mind what the problem could be related to…

- raw data and calculations
- chemical reagents used in the test
- the expiration dates of calibration and check standards
- instrument calibration
- instrument responses
- instrument maintenance
- laboratory reagent water
- sample handling (Was it preserved? Was it compromised somehow?)
- staff training and capability
- standard operating procedures
- data entry / data review
- undue job pressures /distractions
- workplace cleanliness

Quality Systems

SOPs

Include both ***preparation*** and analysis procedures.

Include potential interference(s) and how they are **treated.**

NEW

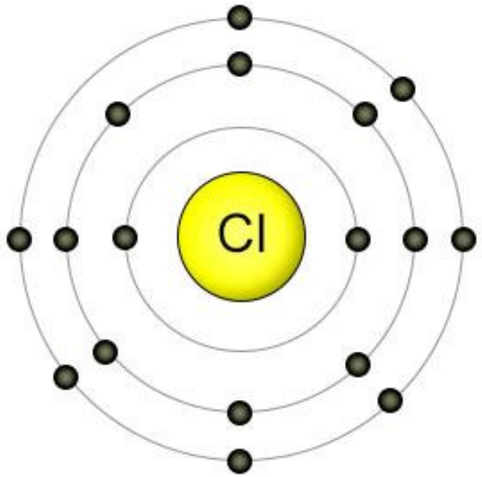
Not this kind of interference…

Quality Systems

Addressing interferences in the SOP: include potential interference(s) and how they are **treated.**

Often, this info is in the reference method.

For example, an interference for BOD could be residual chlorine. Treat with sodium sulfite.



For cyanide by colorimetry, both chlorine and sulfide interfere. Refer to the approved method for how to treat these.

Quality Systems

Labeling bottles – Reagent and standard containers shall be labeled with: expiration date *(if provided by manufacturer),* chemical name, and concentration.

NEW

Weights – Just need **1** weight, but it needs to be the correct class. For analytical balances, typically, an ASTM class 2 or better is needed.

NEW

There are some certified laboratories that have a system in place to use NIST traceable weights to verify the working weights. This has been added as an acceptable practice.

NEW

Quality Systems

ETHICS – things that are prohibited:

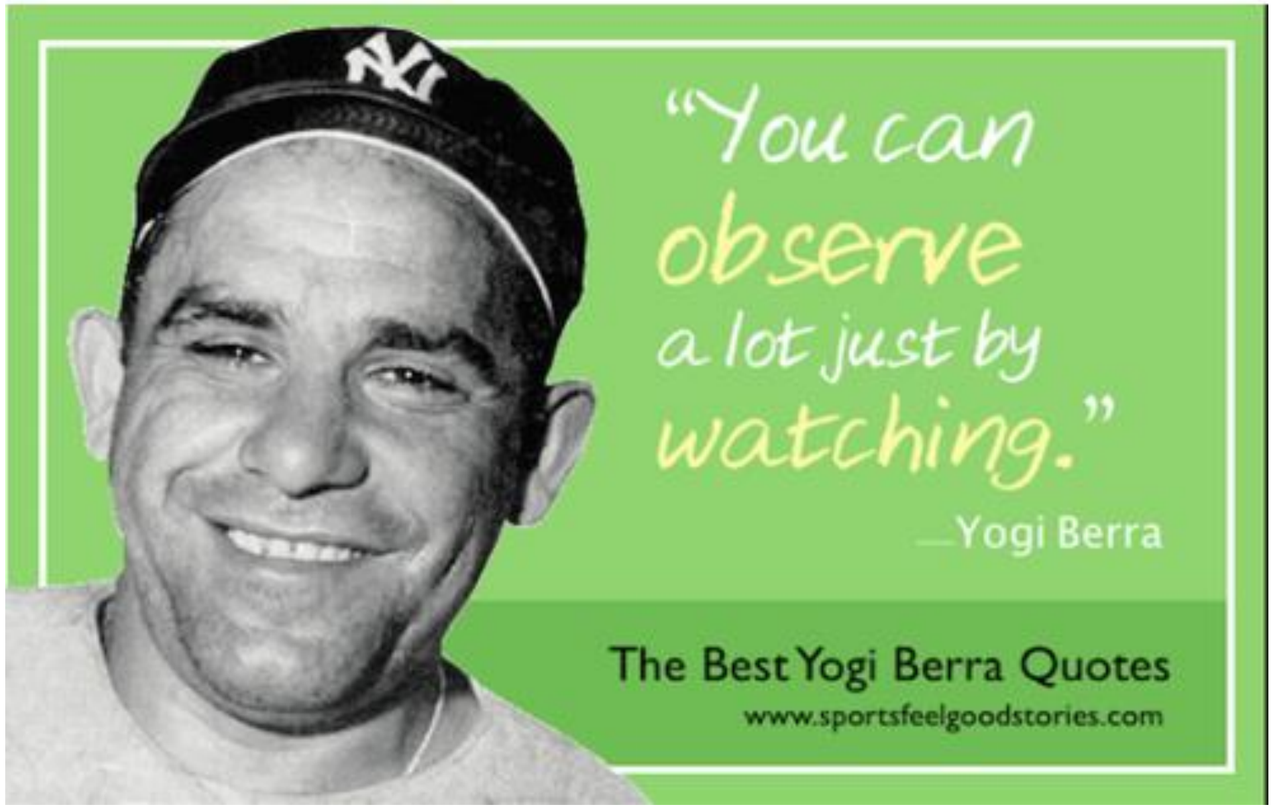
NEW

- Fabrication, falsification, misrepresentation of data
- Time traveling (i.e., recording of dates improperly)
- Unwarranted manipulation of samples, software, or analytical conditions
- Concealing or failing to report a known improper or unethical behavior or action associated with sample analysis

While it is not required, adding ethics to the Quality Manual helps train all staff of the prohibited list.

Quality Systems

ETHICS



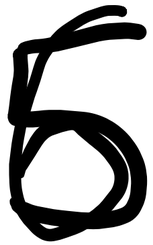
Let staff know whom to report issues to, and that lab cert is here to help.

Quality Systems

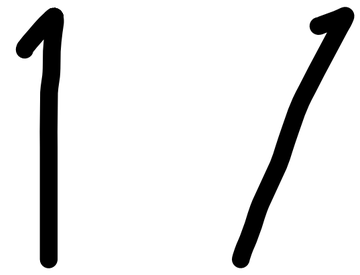
Need to make sure documentation is ~~legible~~. **LEGIBLE**

Can another person decipher the numbers and letters written?

~~149.39~~ (g) – avoid scribbles



– avoid overwriting



Is this a “one” or a “seven”?
…based on a true story ☺

Quality Systems

When specific **temperatures** are required, the operating temperature shall be checked and documented (solids, BOD, total phosphorus, COD etc.).

CCVs (known standards) are analyzed **before** analyzing any environmental samples or batch QC samples (i.e., on non-calibration days, run the CCV *then* the method blank).

Adjust the LODs and LOQs when the samples are diluted.

This applies if the sample is adjusted (different amount used from the LOD study) prior to the preparation or just prior to analysis.

Indicate in the test report if LOD and/or LOQ have been adjusted.

This is not applicable to HRGC/MS analysis.

Whew!

Test Reports

These apply unless other WDNR programs have specific reporting requirements or if the laboratory has a written agreement with a client that certain elements in NR 149.47 are not needed.

Date: ***IF*** there is a specific holding time for preparation, then include the date of preparation (extraction, digestion, distillation, concentration).

NEW

Time: ***IF*** the holding time is in hours, include the times of collection, preparation, and analysis.

NEW

Include the preparation method (in addition to the analytical method).

NEW

MCL exceedances for NR 809 public water supplies (drinking water) need to be reported within 48 hours of completing the sample results.

Test Reports - Qualifiers

As indicated earlier, results or samples must be qualified for issues related to holding time and preservation requirements AND…

Also qualify affected results/samples when there are problems meeting requirements related to: collection, insufficient sample received to complete the analysis, or inappropriate containers.

Include any qualifiers with the reported results.

For multi-analyte analyses, if an analyte does not perform as well as most of the analytes in the initial calibration, analysis may continue, and the laboratory may report the analyte results - *if* qualified.

Test Reports - Qualifiers

When the RPD is >40% for dual column/detector systems, report the higher of the two results, unless the higher result is biased due to interference. If so, the lab can report the lower result with a qualifier or can report both results.

NEW

When reporting to the LOD for ICP analysis, if the non-spiked elements in a required interference standard are not less than the LOQ or 10/3 the LOD, reanalyze the sample or report with a qualifier.

NEW

Technology - NEW specifics to NR 149

Ok, so it’s not as exciting as the **NEW** Mars Rover Concept Car…

Technology - Colorimetric/Turbidimetric

- Calibration blanks are required in the initial calibration (curve). Use the measured response (it is NOT always = “0”). *Calibration blanks are not required for inverse chemistries.*

- If performing total phosphorus digestion with a hot block using closed vials, heat at 150 ± 2°C for at least 30 min.
- Do not dilute samples after adding color reagent.
- Hexavalent chromium standards must be treated the same as the samples.
- When using sulfide strips to check for sulfide interference, the strips need to be able to detect 10 mg/L.

Technology - Gravimetric

- **<u>Residue analysis:</u>**
  - Use wide bore pipets.
  - *<u>Don't</u>* use Buchner funnels.
  - *<u>Don't</u>* use Gooch crucibles.

- **<u>Oil and Grease analysis:</u>**
  - When using the SPE extraction technique, do not allow polar solvents to contact the sample.
  - The laboratory shall use <u>activated</u> silica gel for silica gel-treated determinations.

Technology - BOD, cBOD

- Maintain room temperature at 17 to 23 °C.
- Use the theoretical oxygen saturation point.
- Calibrate the meter at or near oxygen saturation point…

…based on temperature and barometric pressure,
on each day of analysis to assess supersaturation
(note – take these measurements from the DO meter).

- Assess (and treat) supersaturation each day of analysis.
- Use samples volumes to expect **2** mg/L depletion.

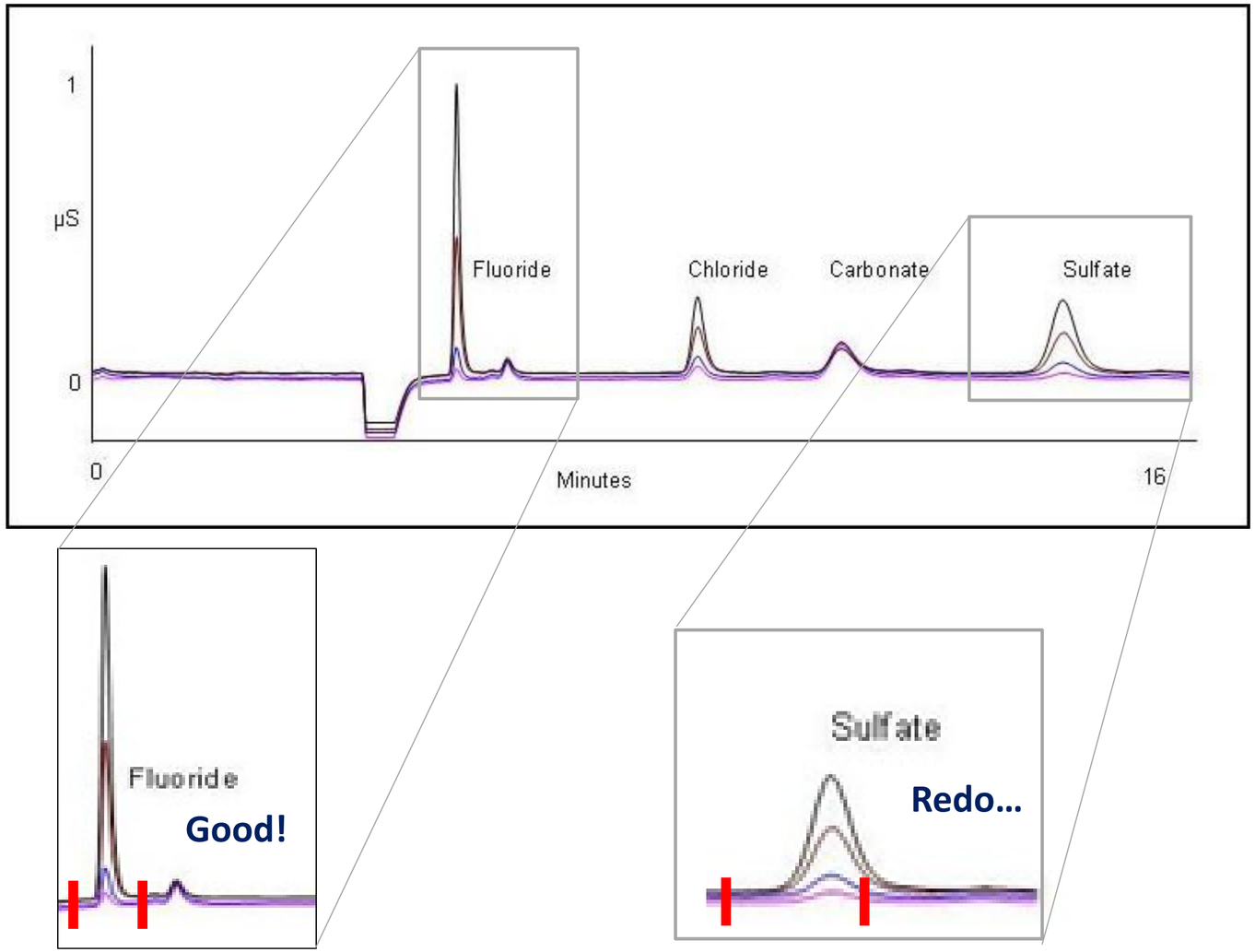
Technology - BOD, cBOD

- Optical DO probes – calibrate each day (if the lab has more than one, each needs to be calibrated)
- Barometric pressure – local, not adjusted to sea level
- Chlorine test strips – must be able to detect 0.1 mg/L residual chlorine
- Pipets or tips – wide bore pipets or tips
- GGA – no averaging (each must pass or qualify the data)
- Method blanks – no averaging (each must pass or qualify the data)
- Seeding – seed samples that have been disinfected or inhibited
- Inhibitor – Do NOT add inhibitor to GGA, method blanks, or seed material

Technology - Ion Chromatography

- The retention time (RT) window width used to identify the analytes **must be based on RT measurements of standards over the course of a day…**

  …unless analyst experience provides for another defensible procedure.



Technology - Titration & NDIR or Microcoulometry

- **Titration/Potentiometric assays**
  - Labs shall standardize the titrants monthly when required in the method unless:
    - Unused titrant is never poured back into the original container.
    - Titrants shall always be protected from light.
    - LCS recovery control limits achieved are 90 to 110%.

- **TOC by NDIR/Microcoulometry**
  - Complete an inorganic carbon removal check with each analysis batch.
  - Perform duplicate injections until the relative percent difference is 10% or less for aqueous samples when results are > LOQ.

Technology - FLAA and GFAA

- **Flame AA analysis:**
  - Use at least 2 consecutive readings for all samples and standards (report the average).
  - The RPD limit is 10% between the readings when results are > LOQ.
  - Use the same acid matrix for both the calibration standards and samples.

- **Graphite Furnace AA analysis:**
  - Use at least 2 consecutive firings for all samples and standards.
  - The RPD limit is 10% between the readings when results are > LOQ.
  - Use the same acid matrix for both the calibration standards and samples.
  - For wavelengths < 200 nm, use Zeeman or an equivalent background correction.

Technology - CVAA

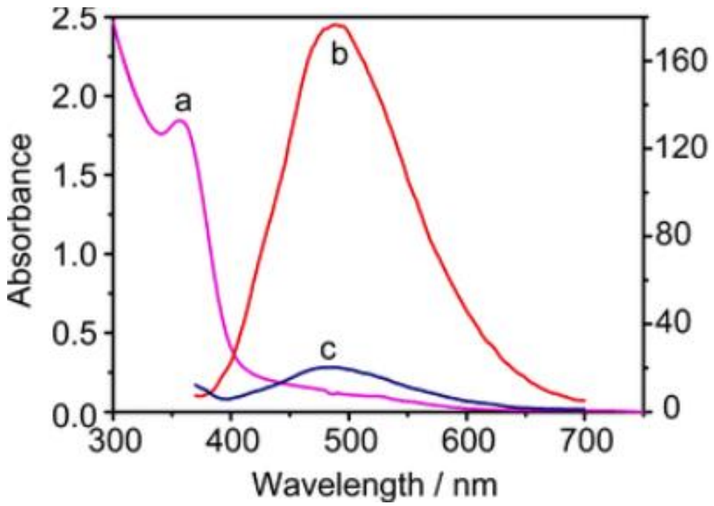
- The laboratory shall ensure that potassium permanganate is present **<u>after</u>** the two−hour digestion for mercury analysis. *This is a clarification of the method requirements.*

  - If not, re-digest the sample using a smaller sample amount so that the potassium permanganate remains in excess.

  - Alternatively, add more potassium permanganate to the affected samples and method blank, and digest for an additional two hours.

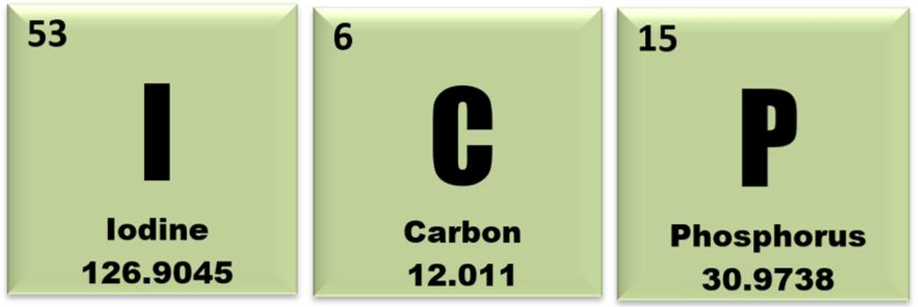
Technology - ICP

Identify the interferences:



- Analysis of each of these single element standards is required to determine if there are spectral interferences: Ag, Al, As, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Se, SiO_2, Sn, Sr, Ti, Tl, V, and Zn.
- This is required for each instrument, mode, and wavelength used for quantitation.
- When the lab identifies other spectral interferences, those element(s) must also be analyzed.
- The concentration of the single element standards must be as high as the analyzed sample concentrations.

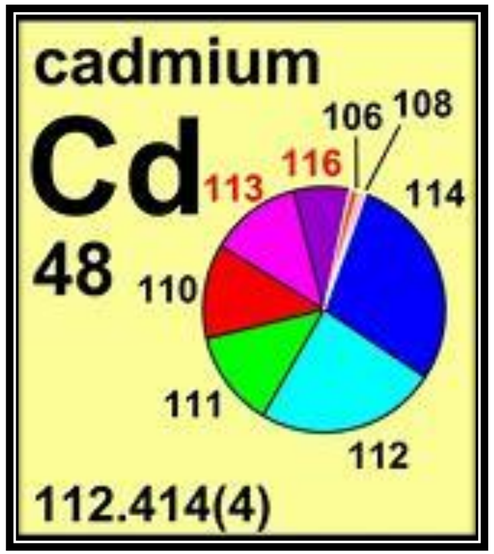
Technology - ICP



Verify the corrections

- Each day before analyzing samples, analyze interference check standard(s) to verify the interference and background corrections.
- The interference check standards need to include levels of interferents at the maximum concentration of the quantitated sample results.
  - If sample concentrations are higher than the interference standards, dilute the sample, ***or*** analyze a single interferent standard to verify there are no interferences affecting the analyte to be reported.
- The non-spiked target elements in the interference check standard need to be < 10/3 the LOD when reporting results to the LOD (or the affected analyte result must be qualified).
- If the background correction is **adjusted**, the lab needs to assess if there was an effect on any associated interference correction factors.

Technology - ICPMS



- Only the masses listed in the approved methods may be used for identification and quantitation…

  …unless the laboratory has data specifying interferences for other masses, and if correction equations are needed, they are utilized.

- All QC samples need to be performed on the isotope used for identification and quantitation.

Technology - GC and GC-MS

- **GC Analysis:**
  - Non-Aqueous samples: calibration standards need to include the same preservation type as the samples for VOC analysis.
  - The SOP for multi-peak compounds (Aroclors, toxaphene, chlordane) needs to include which peaks are used to identification; how quantitation is done when there is weathering, degradation or positive interference; and for Aroclors, how quantitation is done when there is more than one Aroclor detected in the sample.

- **GC-MS Analysis:**
  - Non-Aqueous samples: calibration standards need to include the same preservation type as the samples for VOC analysis.
  - Full scan tunes must pass prior to analyzing samples by selective ion monitoring (SIM).

Technology - Hazardous Waste Characteristics & Preparation Methods

- **<u>Hazardous Waste Characteristics:</u>**
  - **TCLP:** When measuring pH to determine the TCLP leaching fluid to use, the lab must stir the samples. *(This was clarified with the EPA.)*
  - **Ignitability:** A flashpoint standard is required with each batch.

- **<u>Preparation:</u>** (*Unless otherwise required by the method)*
  - Fortify (spike) the QC samples prior to adding the preparation reagents.
  - When microwave technology is used, it must use temperature feedback control.

Removed (no longer needed - these are OUT)

Removed (no longer needed - these are OUT)

- Don’t need certificates displayed
- Cap on technology fees has been removed (allows for some savings for WWTPs)
- Chain of custody (COC) references have been removed
- Since the methods dictate QC requirements, matrix spikes, duplicates, replicates, and QCS were removed from NR 149 (*follow method requirements)*

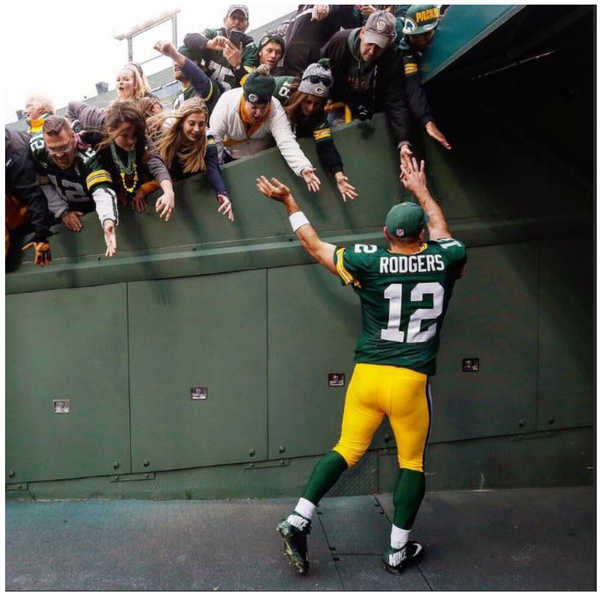
Removed (no longer needed - these are OUT)

Quality Manual – these details are not required for NR 149:

- Organization and management structure
- List of major analytical instruments and support equipment
- Procedures for reviewing analytical data and reporting results

SOPs – these details are not required for NR 149:

- Analytes (unless multi-analyte)
- Applicable matrices
- Method sensitivity



Removed (no longer needed - these are OUT)

DON'T need to *(but is still acceptable to)*:

- Use **2** weights to check balances monthly *(do need just 1 in the range)*
- Perform a carboy blank to show they are free of contamination *(however, labs must not use inappropriate containers)*
- Perform quarterly verifications for burettes or auto-titrators
- Calibrate annually
- Dilute samples by the least amount possible
- Include a standard in the curve near the LOQ *("near" can be subjective)*
- Document the date of receipt for standards and reagents
- Assign an expiration date when one is not provided by the vendor

Removed…

- The requirement for the LOD to meet 10% of the MCL *(the lab's LODs must still meet state and federal requirements)*
- The requirement to analyze at least one precision sample (duplicate or MSD) per batch for drinking water samples *(this is still needed when required in the method*)
- The requirement to use the simplest calibration function
- The allowance to use a QCS instead of a PT sample for metals by colorimetry, metals by FLAA, and low-level mercury *(instead, labs will need an applicable PT)*
- The allowance to prove standards and reagents are valid after they have expired *(labs must not use expired reagents or standards)*
- All references to EPA monitoring trigger limits for drinking water
- The requirements for test reports (per NR 149) to include specific dilution factors, the date of receipt, or signatures.

Other Changes

- Clarified that certified laboratories, when there are chemical preservation requirements, need to check the pH of each sample that will be analyzed.
- Laboratories must meet the minimum requirements specified in SW-846 methods.
- Updated the programs that are covered by NR 149 and changed some fees.
- Changed “solid” to “non-aqueous” as a description of a matrix type.
- Leachates from solid waste leaching procedures, biosolids, and sludge are included in the non-aqueous matrix type.
- “Waste Characterization Assays” is now “Hazardous Waste Characteristics.” TCLP is included in this group.
- There have been other category/class changes like the items above. For example, a “Gaseous Hydride AA” category was added. For another example, there is no longer a category, “ultra low-level metals” – this was included in a “CVAFS” category.

Questions?

Hooray, you made it!

Thank you!

Please keep in mind this information is meant to inform the laboratories of the changes. *It is possible* some changes were not covered here.

This presentation **is not** reflective of all the necessary requirements.

Please refer to our website for the entire code and additional helpful information.

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