

WISCONSIN DEPARTMENT OF NATURAL RESOURCES

NR 149 UPDATES *for* Registered Labs *BASE 4 tests*

March 2021 DNR.WI.GOV



Intro to Lab Certification Staff



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“New” NR 149



Publishing NR 149 in March 2021

IMPLEMENTING by September 1, 2021

Website: <https://dnr.wisconsin.gov/topic/LabCert>

Out with the OLD and in with the NEW



As technology or standards changes...

...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.

Who do these changes apply to?



The summary of the changes in this presentation are for **registered laboratories** that analyze TSS, BOD, along with ammonia and total phosphorus (base 4 tests).

There is a different presentation with the changes that pertain to **certified wastewater treatment plants** and **commercial laboratories**.

So what do **LABs** need to do to prepare?



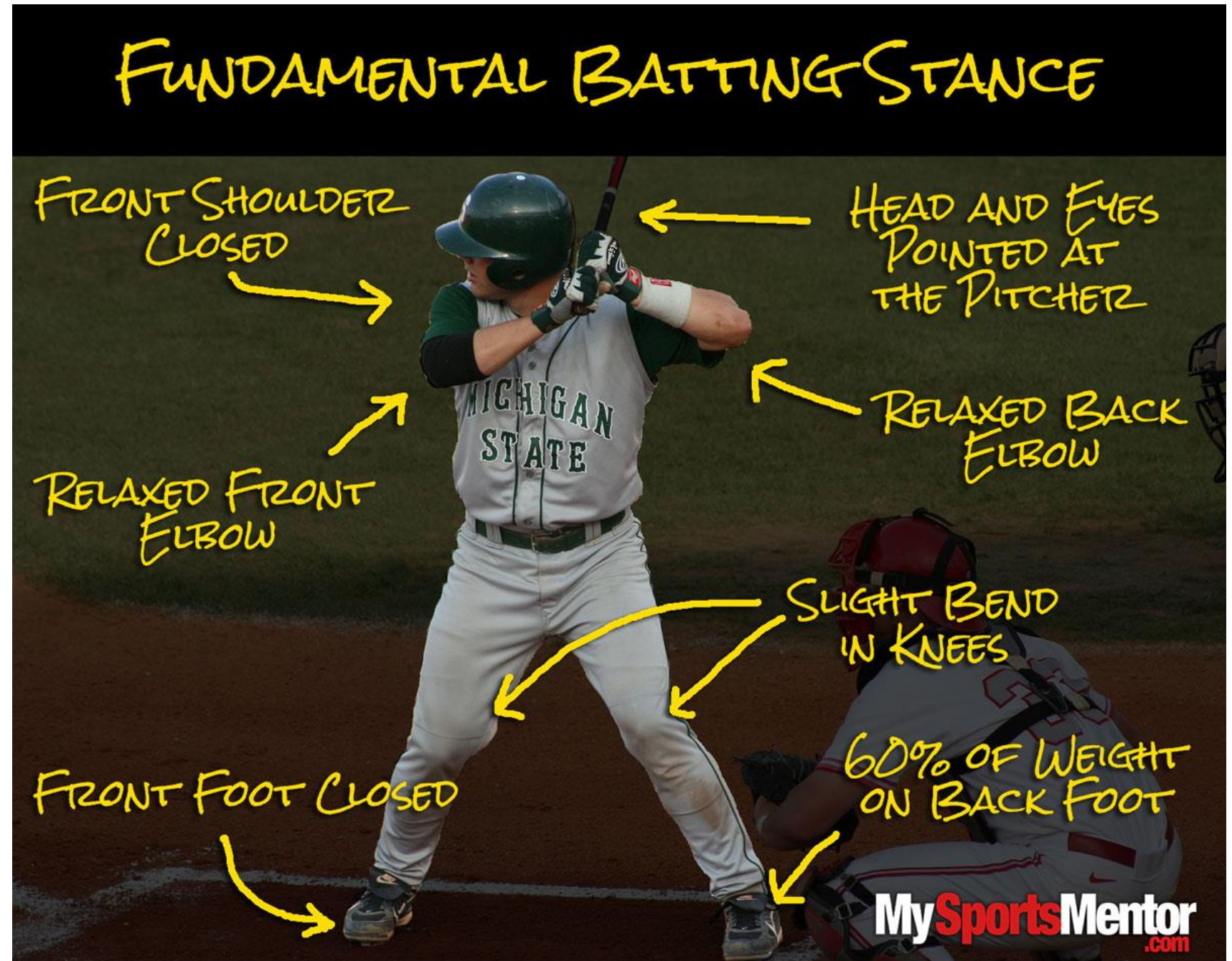
PTs

Calibrations

LOD/LOQ/RL

Quality Systems

Technology Info



Proficiency Testing (PT) Updates



Environmental Proficiency Testing



2021 Catalog

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PT Updates

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

NEW

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.**



PT Method Code Info (3/17/21)

The following tables have the methods codes that are acceptable to report with your PT samples. These are not the only methods that are approved, but these are by far the most commonly used.

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



PT Method Code Info (3/17/21)



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

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

PT Method Code Info (3/17/21)

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

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



PT Updates



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

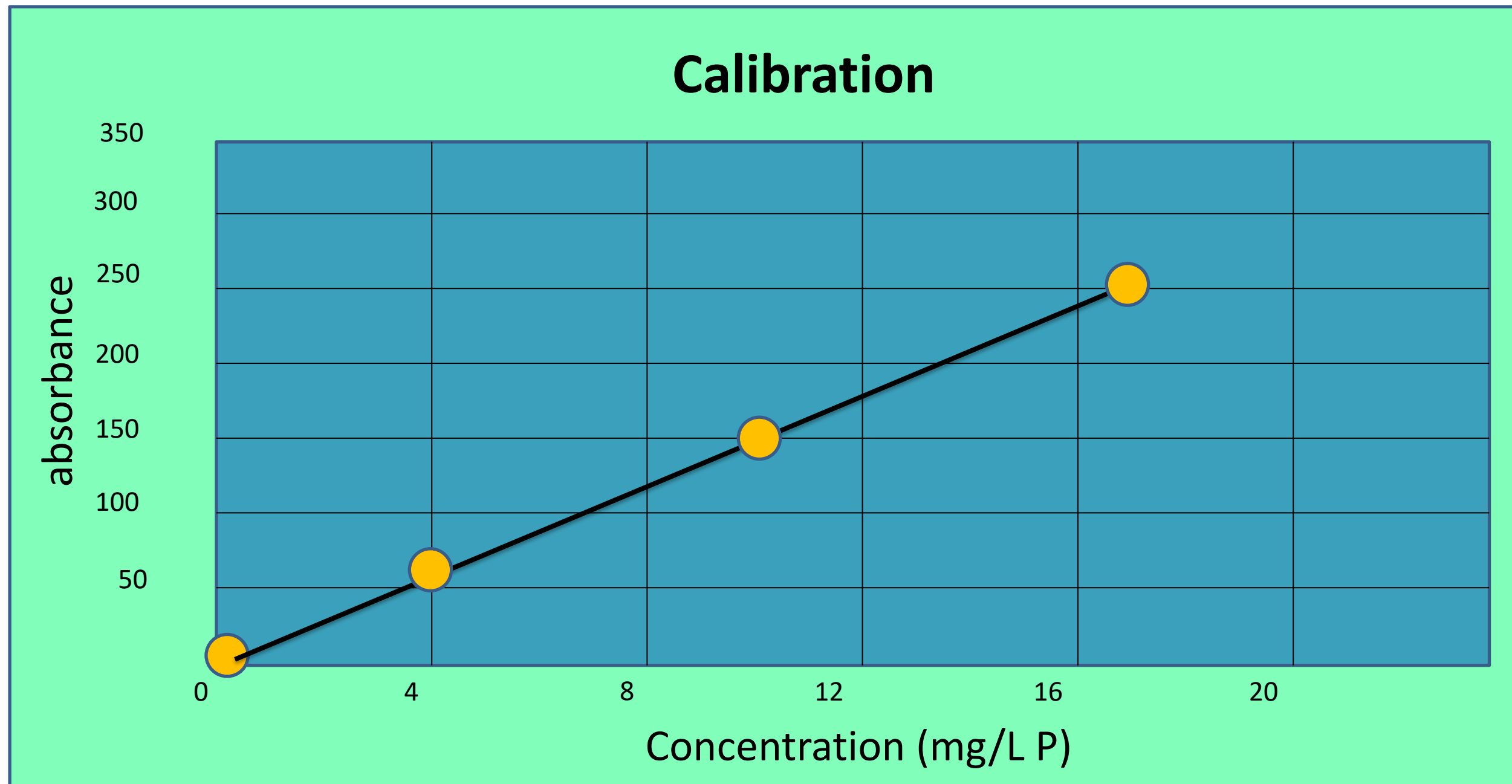
Different
WP
studies

10 days
apart

Not run
in the
same
batch



Calibration Curves (initial and continuing)



Calibration Curves



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

Factory calibrations (still) **NOT** acceptable for accredited tests for compliance.

Need a new calibration 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 Curves



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

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

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



Ion Selective Electrode (ISE) Calibration (added)

Calibrate **DAILY** – applies to all ISE:
BOD, NH₃, & pH

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, pH)*



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

NEW

Calibration (some EXTRA stuff)





Calibration (some EXTRA stuff)

The ICV and CCVs need to be processed like the calibration standards. If the initial calibration was processed or when required by the method, process each calibration standard, CCV, and ICV in the same manner as samples.

(not typically an issue for registered labs)

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

(not typically an issue for registered labs)



Calibration (some EXTRA stuff)

How to determine if the curve passes:

Adding additional/*optional* ways to assess the calibration.

NEW

- X-intercept \leq LOD
- Residuals must be 90-110% except low standard must be 80-120%
- Linear must have correlation coefficient $(r) \geq 0.995$

For example:

If the laboratory **decides** to use calibration point residuals (read backs) to evaluate the calibration curve, then the recovery **for each point must be 90-110%** except for the **lowest point, which is 80-120%**.

Calibration (some EXTRA stuff)



What do **residuals** (read backs) look like?

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%

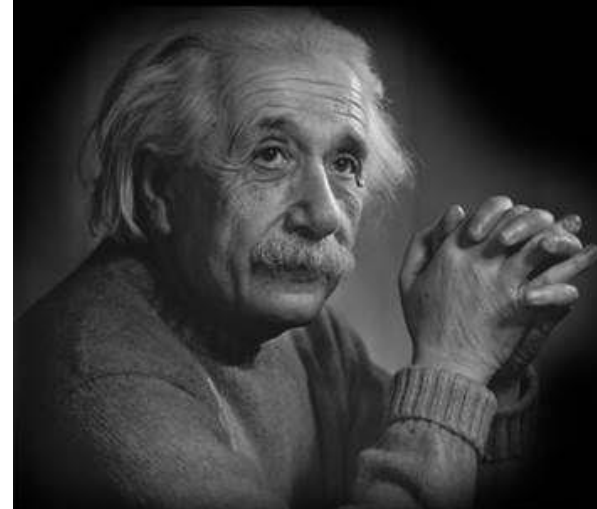
Easy...



LOD, LOQ, and RL Updates



ONCE WE ACCEPT
OUR LIMITS, WE GO
BEYOND THEM.



Albert Einstein
German Theoretical-Physicist
(1879-1955)

QuoteHD.com

$$E = ?$$

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.

LOD and LOQ Updates



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

LOD update: If the sample is adjusted (diluted) from how the LOD procedure was done (blank spikes), then the LOD **shall be adjusted**. *(May happen if reporting influent results).*

LOD

2. Limit of Quantitation (LOQ)

The LOQ shall be equal to $10/3 \times \text{LOD}$ **or** set to the lowest concentration standard in your curve. Adjust the LOQ by dilution factors when samples are diluted.

LOQ

NEW

Reporting Limit (RL) Updates



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

RL

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 there was no measured residue, then report <2 mg/L.

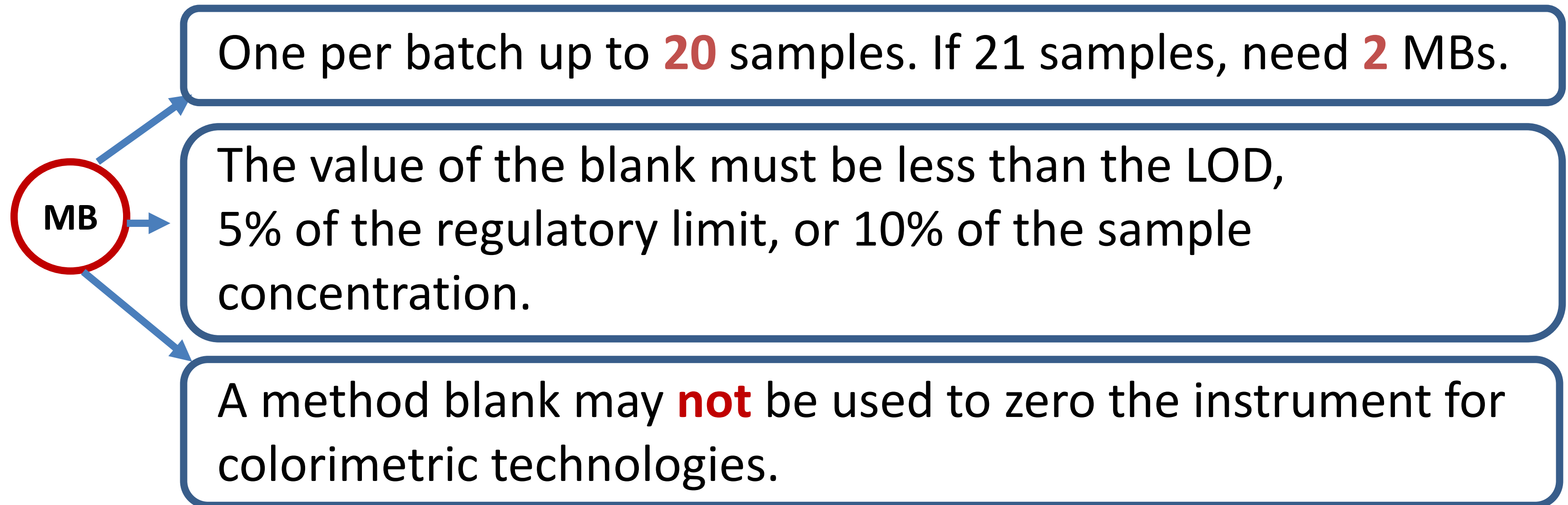
Method Blanks, LCSs, Quality Systems



Method Blanks



Method Blank (MB) – *not required for pH or solids*



LCSs



Lab Control Sample (LCS) (*BOD: if <20 samples in week, need 1 GGA LCS/wk*)
– *doesn't apply to TSS*

One per batch up to **20** samples. If 21 samples, need **2** LCSs.

The lab **may** use the CCV limits for the **LCS recovery limits** instead of generating their own in-house control limits – could apply to NH₃.

IF the LCS is also = the CCV (LCS/CCV), need to meet the CCV limits.

LCS

Quality Systems



Corrective Action:

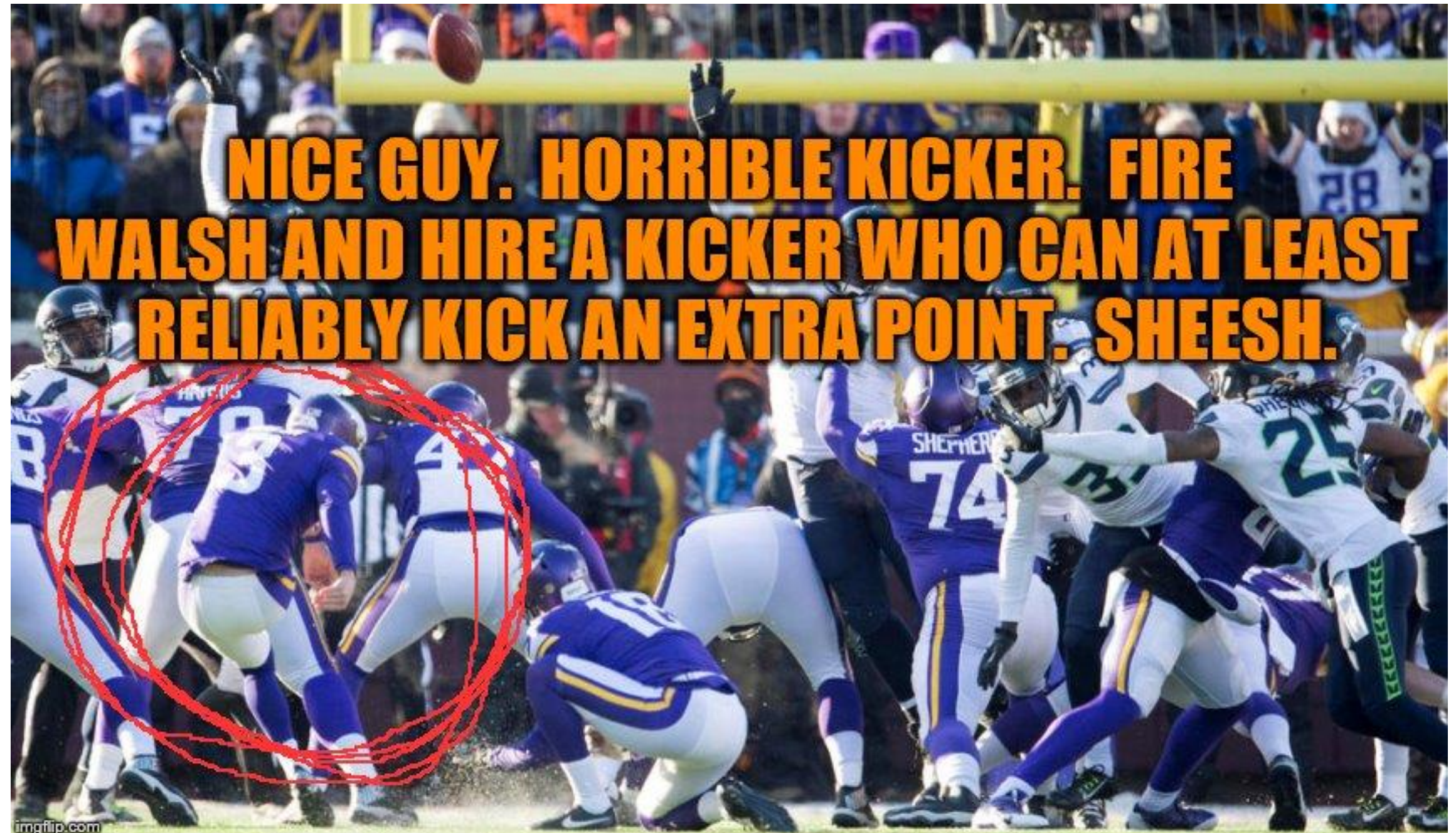
Root cause analysis shall be performed when there is **recurrence**.

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 investigated better 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.

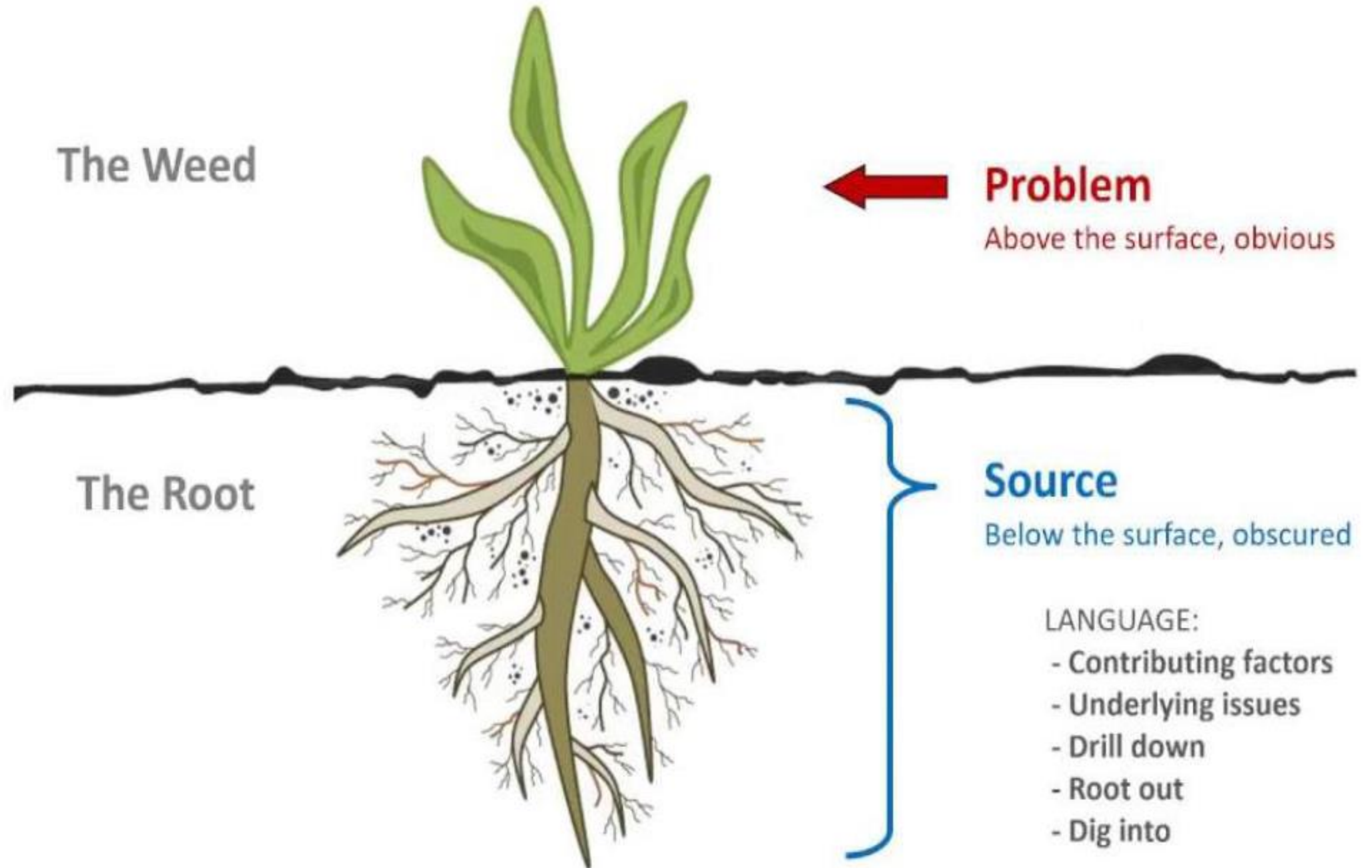
Root cause – in depth...

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

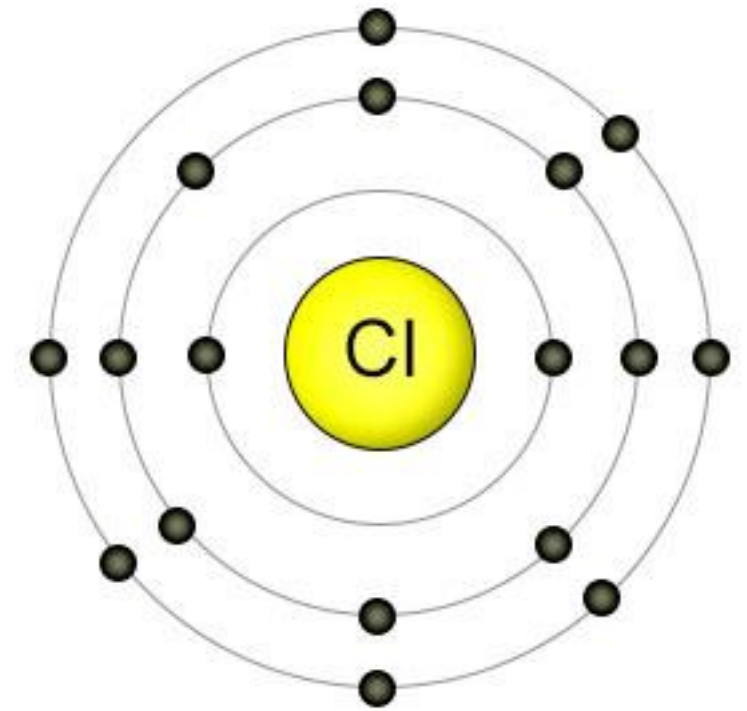


Addressing **interferences** in the **SOP**: include potential interference/s and how they are **treated**.

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

For TSS, it could be large floating goo. Avoid the goo.

Often, this info is in the reference method.



Quality Systems



Labeling bottles – Reagent and standard containers shall be labeled with: expiration date, chemical name, and concentration.

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

NEW



Quality Systems

NEW

ETHICS – things that are prohibited:

- 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



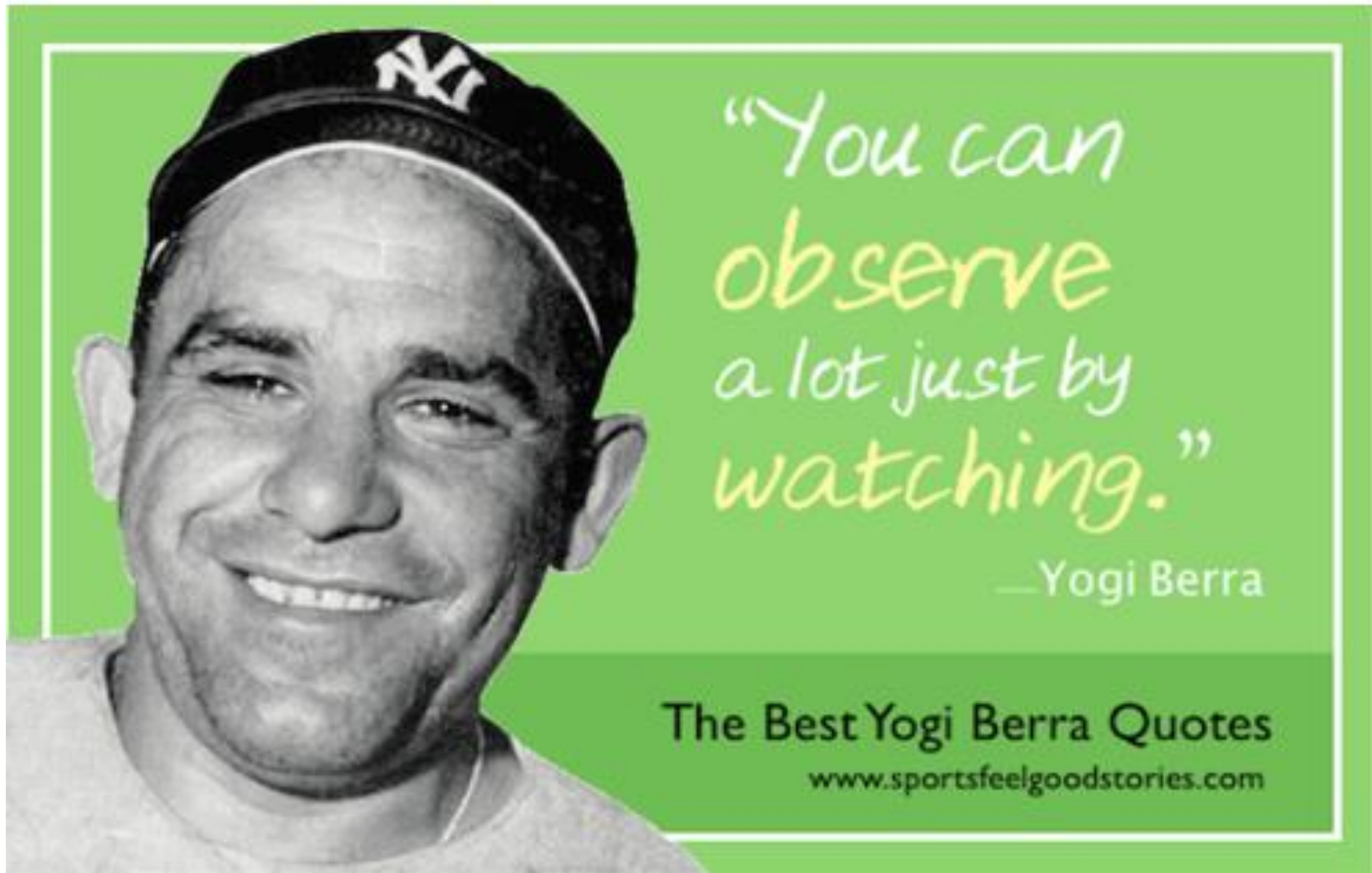
While it is not required, adding ethics to the Quality Manual helps train all staff of the prohibited list.



Quality Systems



ETHICS



Quality Systems (minor)



Need to make sure documentation is *legible* • **LEGIBLE**

Can another person decipher the numbers and letters written?

1 7

Is this a “one” or a “seven”??
...Based on a true story 😊

Quality Systems (minor)



When specific **temperatures** are required, the operating temperature shall be checked and documented (TSS, BOD, TP).

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).

For TP and NH₃, LOD and LOQs must be adjusted for:

- sample dilutions performed just before analysis, and
- sample dilutions performed by using a smaller amount of sample at the beginning.

Whew!



Technology - NEW specifics to NR 149

Ok, so it's not as exciting as the **NEW** Mars Rover Concept Car...



Technology - Colorimetric and Solids



Colorimetric:

Initial calibration (curve): when using calibration blanks, be sure to use the measured response (it is NOT always = "0" just because it is a blank).

TP Digestion with hotblock using closed vials: heat @ 150 +/- 2°C for at least 30 min.

Do not dilute samples after adding color reagent.

Solids:

Use wide bore pipets,
don't use Buchner funnels, and
don't use Gooch crucibles.



Technology - BOD, CBOD

Maintain room at 17 to 23 °C.

Use the theoretical saturation point.

Calibrate 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
- Barometric pressure – local, not adjusted to sea level
- Chlorine strips must test down to **0.1** mg/L
- 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)
- Seed samples that have been disinfected or inhibited
- Do NOT add **inhibitor** to GGA, method blanks, or seed material



Removed (no longer needed - these are OUT)



Removed (no longer needed - these are OUT)



- Don't need certificates displayed
- Cap on technology fees is 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



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
- Applicable matrices
- Method sensitivity



Removed (no longer needed - these are OUT)



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

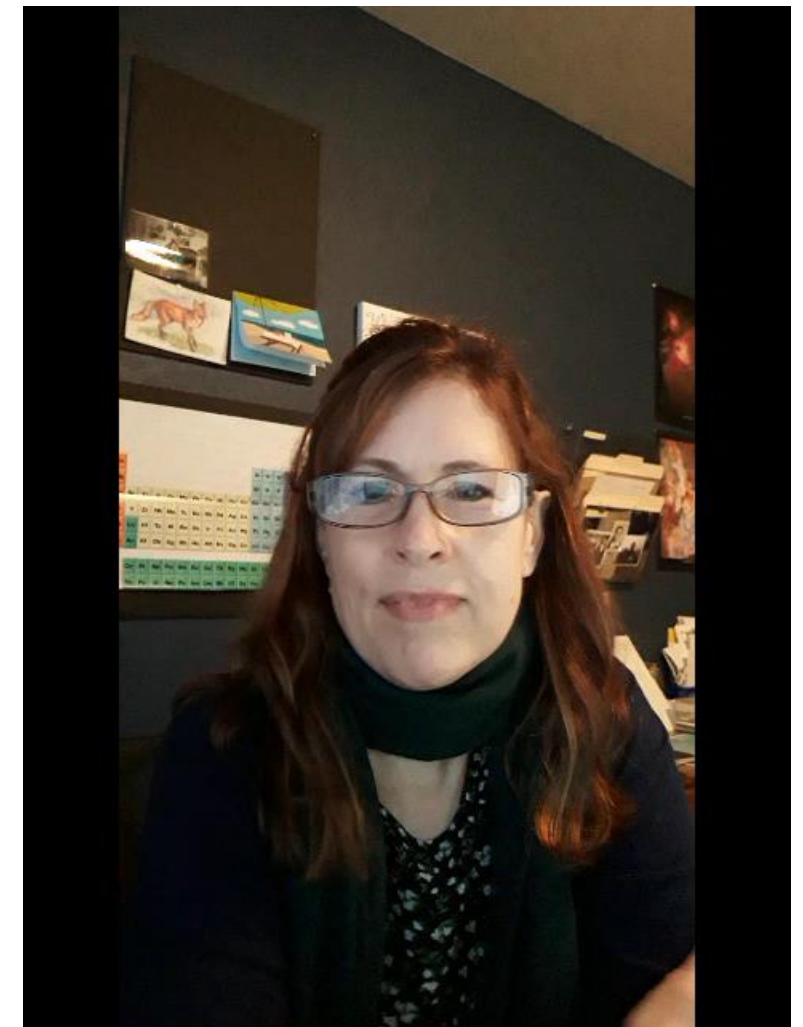
- Use 2 weights to check balances monthly (*do need just 1 in the range*).
- Perform a carboy blank to show they are free of contamination.
- Calibrate annually.
- Dilute samples by the least amount possible.
- Include a standard in the curve near the LOQ (*“near” can be subjective*).
- Document the date of receipt for standards and reagents.
- Assign an expiration date when one is not provided by the vendor.

Questions?

A question from Brooke



Answer from LabCert



Let's keep working together to find solutions.

Thank you!

Special thanks for inviting us to your meetings –

WI Wastewater Operator's Association WWOA

WWOA Southern Region

WWOA North Central Region



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Thank you! The end.



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OFF THE RECORD"