

WISCONSIN DEPARTMENT OF NATURAL RESOURCES

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

May 2021 DNR.WI.GOV



“New” NR 149

Published March 2021

IMPLEMENTED by September 1, 2021

This presentation is for registered laboratories that analyze
TSS, BOD, NH₃, TP

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 no longer required:

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

SOPs – these details are no longer required:

- Analytes
- Applicable matrices
- Method sensitivity



Removed (no longer needed - these are OUT)

- Don't need 2 weights to check balances monthly, now only need 1 weight.
- Do not need to perform a carboy blank.
- Do not need to calibrate annually just because a year is up.
- Do not need to document the date of receipt for standards and reagents.
- Do not need to assign an expiration date when one is not provided by the vendor.

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)

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



Calibration curves do NOT need to be done annually.

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

Will need a new calibration curve if the CCV fails.

Ion Selective Electrode (ISE) Calibration (added)

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

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

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



NEW

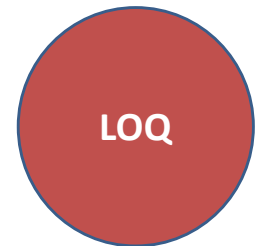
LOD and LOQ Updates

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



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.



Reporting Limit (RL) Updates

RL

Reporting Limits (RL) – applies to BOD and TSS

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

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

Method Blanks

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



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

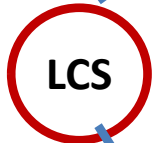
MB

A method blank may **not** be used to zero the instrument for colorimetric technologies.

LCSs

Lab Control Sample (LCS)

LCS



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

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

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

SOPs

Include both preparation and analysis procedures.

Include potential interference/s and how they are **treated**.

NEW



Not this kind of interference...

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



Colorimetric TP and NH₃

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.

TSS

Use wide bore pipets

don't use Buchner funnels

don't use Gooch crucibles



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