LABNOTES
News & Updates of the LabCert Program

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Join our Team!

Interested in seeing what life is like on the other side of your DNR audit? DNR is hiring an Audit Chemist to replace Tom Trainor starting sometime in January.

The Audit Chemist is responsible for evaluating commercial, industrial, public health, and wastewater laboratories both within and outside of Wisconsin. Candidates are eligible to be based out of any DNR service center or field office across the state. Auditors can work some of the time from home when they are not on the road.

Extensive travel is required: 2-4 audits per month and about 6 out-of-state audits per year.

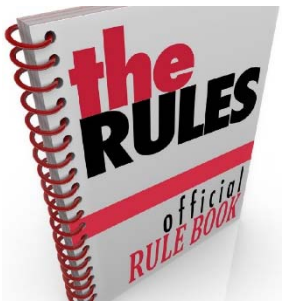
Through the diligent work of the Audit Chemists, the State of Wisconsin ensures that accredited laboratories have the appropriate systems in place to generate reliable data through environmental testing.

If you're a chemist with a passion for environmental testing and quality assurance, then this is the perfect position for you!

Please see our posting on the State's Job Site for further details on how to apply. ❋
http://wisc.jobs/public/job_view.asp?annoid=96555&jobid=96069&org=370&class=41563&index=true

NR 149 Revised Code Update

The Program and NR 149 Workgroup have considered the over 300 individual comments received on the rule during the public comment process and have completed the final version of the proposed rule. A complete response to the comments will be included in the "green sheet" package that will be sent to the Natural Resources Board (NRB) when we request promulgation of the final rule at the NRB meeting on December 12, 2018 in Madison, WI. If approved by the NRB, we then wait for approval by the legislature. If approved by the legislature, we project an effective date of September 1, 2019 which coincides with the annual renewal date for all laboratories. A summary of the changes will be available on the Laboratory Certification Website in the summer of 2019.



You can follow progress of the rule (and view all documents filed) by accessing the Wisconsin State Legislature's Clearinghouse Rule Page at http://docs.legis.wisconsin.gov/code/chr/all/cr_17_046. ❋

Using Vendor Methods to Analyze Drinking Water Compliance Samples

The Laboratory Certification Program has been in contact with EPA Region 5's drinking water Program several times in 2018. EPA Region 5 has made it clear to the Program that the only vendor methods that are acceptable for use in drinking water compliance analyses are:

- Methods listed in 40 CFR Part 141
- Methods approved under the EPA NPDWR Alternative Test Procedures

Any method that does not fall under these two options is unacceptable for compliance drinking water analysis regardless what the vendor may claim. ❋

Oil and Grease Analysis – No Partial samples allowed

When performing oil and grease analysis (as hexane extractable materials, HEM), the entire volume of the sample received in a single container must be analyzed.

EPA Method 1664B states:

1.7.2.3 *Due to the non-homogeneous nature of wastewater samples and the high probability that extractable material may contact and adhere to a wide variety of surfaces (including substances in the sample as well as the sample bottle and cap) and be non-uniformly distributed throughout the sample, sub-sampling or analysis of less than the total collected volume of sample is not allowed*.

It is very likely that HEM will adhere to glassware and other surfaces with which it comes in contact. Therefore, the bottle must not be pre-rinsed with sample before collection. This is also why the sample bottle and bottle cap must be rinsed thoroughly to quantitatively transfer all the HEM to the separatory funnel or filter funnel.

If the entire sample in the bottle is not used, it is not possible to do this critical hexane rinse step.

Side Note 1: because the hexane rinse of the sample bottle is critical, the sample blank must be prepared in a clean sample bottle. The blank must be subjected to the same procedural steps as a sample.

Side Note 2: the spike for the matrix spike must be added to the sample in the bottle it was received in. The recovery of the spike would be affected if the hexane rinse step is not thorough enough (see EPA 1664B 1.7.2.5).

What can you do if you have a really oily or greasy sample?

- The methods do allow for collecting and analyzing a smaller sample volume. If a sample is suspected to contain >500 mg/L of HEM, a smaller sample volume may be collected (refer to EPA Method 1664A 8.1.2 and EPA Method 1664B 1.7.1.5 and 8.1.2).
- For SPE, extract multiple sample portions, and combine the extracts during concentration. Be sure to use quantitative rinses.
- Use separatory funnel extraction.

HEM is a method-defined analyte, so the lab must adhere strictly to the method. As stated in the first line of the procedure section in 1664A and B, "This method is entirely empirical. Precise and accurate results can be obtained only by strict adherence to all details." There are several modifications that are expressly allowed and some that are expressly not allowed in EPA 1664B. If you are unclear about modifications you want to make, contact us!

The New LOD Procedure – Basics for the Wastewater Lab

If you have been analyzing your quarterly spiked blanks and recording your Method Blanks this past year, Congratulations! You now have enough data to calculate your Initial LOD under the new procedure. Hopefully you have had a chance to try the LOD spreadsheet on our website to help you with the calculations:

The LOD spreadsheet is here:
https://dnr.wi.gov/regulations/labcert/

If you have not started the new LOD procedure yet, keep reading.

[Note that nothing changes for BOD and TSS. Those limits are determined by sample size, like they always have been. The LOD procedure is for your ammonia and phosphorous tests, assuming you run those.]

The **"Initial" LOD** requires at least 7 Spiked Blanks and 7 Method Blanks prepped and analyzed over 3 separate days. Use the same spike concentrations you normally use for your LOD studies. Enter those data points into the spreadsheet from our website. If any of your blanks are negative, include the negative value for those blanks in the spreadsheet. Not too difficult, right? Including Method Blank data into your LOD determination might make a difference in your LOD if you frequently have Method Blanks greater than your LOD. Our spreadsheet calculates your LOD both ways and will use the higher of the two LODs. **Please call if you have questions.**

Now, for the "Ongoing" LOD:
Once you have your Initial LOD, it is time to schedule your 2 spiked blanks every quarter. There will be at least 8 spiked blanks analyzed over the year, plus ALL your Method Blanks throughout the year. If you plan and make sure all your Method Blank results are documented in one place, it will be easier. Track them as you go by putting them in our spreadsheet – otherwise, you must go back through an entire year of records to get all the results.
You will need 2 spiked blanks run in each **quarter**…and those 2 spiked blanks must be prepared and analyzed over 2 different days.

Tip: add **reminders** onto your calendar for when you need to run the spiked blanks, so you don't get busy and forget.

Enter those results into the WDNR spreadsheet (remember to do this in the "Ongoing" LOD tab).

Remember to collect all your normal Method Blank data. Don't throw out any data unless there is an obvious explanation for a bad result.

After you have 4 quarters of results, calculate the new ***Ongoing LOD***…this may seem complicated, but it's all in the spreadsheet—so use it! The LOD_s (spiked blank) and LOD_b (Method Blank) results are compared and the highest is your **new LOD.**

But ***HOLD ON***, you now have a choice to make. EPA understands that it is often a burden to change your LOD every year. So, they are allowing you to keep your previous LOD, ***AS LONG AS*** it is not too much different than the LOD you just got off our spreadsheet. How much is too much? If your new LOD is within 0.5 – 2 times your old LOD, you may keep the old one (and if less than 3% of the method blanks are higher than the old LOD). OR, you can decide to adopt the new one – **YOUR CHOICE**! The spreadsheet will let you know if you may keep the old one or if you must use the new one.
Good luck! We want to hear from you on how it's going, and if you have any questions on the procedure or our spreadsheet. The important message we want to convey is that it is **TIME TO GET STARTED**! We will help guide you through it!
By the way, if you are a commercial lab, there are nuances to be aware of, such as the requirements if you are using multiple instruments for an analysis. Make sure you read the EPA procedure to see if there is an option that applies to your situation. We also have a spreadsheet for commercial labs on our website that you may find helpful.

Thank you, Rick Mealy!

After 26 years in the Lab Certification Program, Rick Mealy is moving on to the greener pastures of retirement.

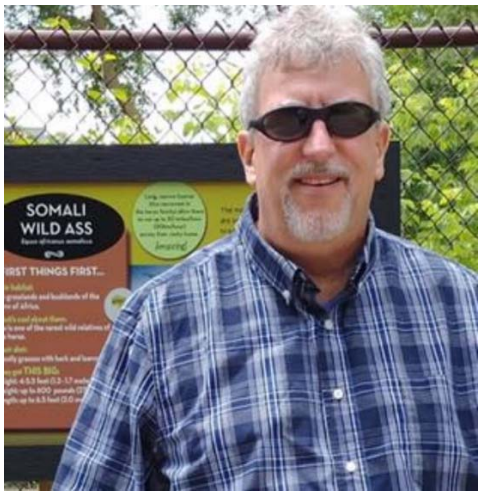
Rick joined the DNR as an auditor in 1993 after stints at several commercial labs on both coasts and in Denver, CO. During some staffing cutbacks in 1995, Rick slid into the Program Chemist role and began pulling the levers behind the curtain on PTs, renewals and applications, and updating NR 149 code.

Rick took the Program Chemist position to higher levels on his personal mission to train wastewater operators on improving their lab techniques, troubleshooting methods, and cutting wasted effort. Those of you who have been around a while will fondly recall the famous travelling Rick & George Show.

Rick's training mission extended to commercial labs as well, leading efforts to improve ICP interferences, PCB identification, and methods for hazardous waste classification. Internally, Rick has been a tremendous resource, offering chemistry expertise to DNR staff in our customer programs.

Even though Rick is moving on from DNR, I predict his passion for lab analysis will continue, and I suspect you will continue to see him around, especially if you attend the WWOA annual conference.

In the meantime, Tom Trainor has agreed to move into the Program Chemist position and will try to fill those big shoes! Good luck with that!



From the Lab Cert Program, we wish Rick an adventure-filled retirement, and offer our heartfelt thanks from all the DNR staff he's influenced over these past 26 years!

Rick's last day in the office will be November 6th, if you want to catch him before he leaves.

This document is intended solely as guidance. facts

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