**NEW LOD PROCEDURE – the BASICS for the Initial LOD**

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

It’s a good idea to save it to your computer, open it up & follow along as you read through both sets of the 6 steps.

There is an **“Initial” LOD** and an **“Ongoing” LOD**. Each are explained, see the next article for the Ongoing LOD.

The **“initial” LOD** is run over at least 3 days.

The initial LOD typically is done when a new procedure is being set up, or there has been another change. If you did the 7 spiked blanks already over 3 days, then proceed with step 4 below using method blank data you already have. FYI, “spiked blanks” are what used to be called LOD standards.

There needs to be at least **7** spiked blanks and at least **7** method blanks. The preparation must be done over at least 3 days and the analysis done over at least 3 days. An example for Total P is below:

1. Pre-planning: Estimate what spike level for the 7 standards spiked blanks will be used. A good idea is to use the concentration used in the past. You *may* adjust the concentration prior to running the initial LOD, and **usually if it is close to the low calibration standard**. The spreadsheet has a tab for estimating that concentration (it is the first tab).

In the example below, I am assuming that the spiked blank (LOD standard) concentration is 0.2 mg/L.

1. Day 1: Prepare 3 spiked blanks at a level of 0.2 mg/L. Prepare 3 method blanks. Digest the spiked blanks and the method blanks. Analyze them on your current calibration curve.
2. Day 2: Prepare 3 spiked blanks at a level of 0.2 mg/L. Prepare 3 method blanks. Digest the spiked blanks and the method blanks. Analyze them on your current calibration curve.
3. Day 3: Prepare 2 spiked blanks at a level of 0.2 mg/L. Prepare 2 method blanks. Digest the spiked blanks and the method blanks. Analyze them on your current calibration curve.
4. Calculate both the LOD types by entering the data…One for the **s**piked blanks (called the LOD**s**) and one for the **b**lanks (LOD**b**). On our spreadsheet use the tab labeled “Initial LOD (8 MB).”



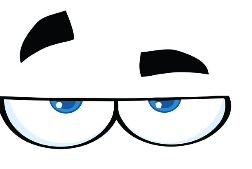
The standard column in on the left for 8 standards and the blank column for 8 blanks is on the right. Remember if some of your blanks are negative, include the negative value for those blanks in the spreadsheet.

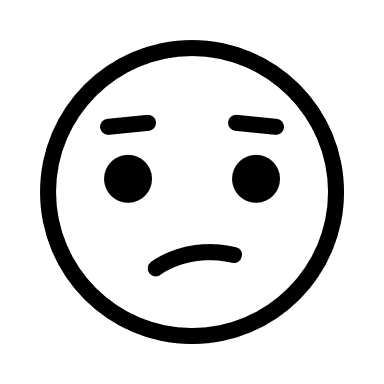
 

The higher of each of these is your **new LOD**.



Look at your LOD result…

Does your LOD make sense? Good! **Great**! Proceed to #6.

OR does something maybe not look right ? Now you’ll need to take a better look at why…

Some scenarios for this are discussed at the end of the second article.

1. The **LOQ** will be calculated using the same 10/3 factor as in the past.

**What’s next? The ongoing LOD! Please see the next article.**

**NEW LOD PROCEDURE – the BASICS for the Ongoing LOD**

**Please see the previous article about the Initial LOD.**

The ***“ongoing” LOD*:** There will be at least 8 spiked blanks analyzed over the year. If you plan ahead 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 the spreadsheet – otherwise you have go back through an entire year to get all the results.

1. Pre-planning: You will need 2 spiked blanks run in each **quarter**…and those 2 spiked blanks must be prepared and analyzed over 2 different batches.

Just like in the initial LOD, you will need to use a spiked blank at the concentration that makes sense. If you have an acceptable initial LOD then use that same spiked blank concentration for the ongoing LOD. If you want to adjust the concentration from the past study, then you need to do a new initial LOD (see above).

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

1. First Quarter …For example, Oct-Dec (2018): If you analyze TP three times per week or once a week, then you could (digest and then) analyze the first spiked blank on Monday with your samples.

Then on the following Monday, digest and analyze the second spiked blank (with your samples). Enter those results into the WDNR spreadsheet (remember to do this in the “ongoing” LOD tab).



These two results can go into the spreadsheet as Q1 A and Q1 B.



Remember to collect all your normal method blank data.



Keep in mind, if there is a problem on one of the Mondays (for example, the QC failed and you had to rerun the samples), then you’ll need to also re-digest and rerun the affected spiked blanks.

1. Quarter two…Jan-Mar (2019): Repeat the scenario from the first quarter (the old ‘rinse and repeat’ like the old-time shampoo instructions): Monday, spike blank 1, and the following Monday, spike blank 2. Remember, collect all those method blanks in the spreadsheet!
2. Quarter three…Apr-Jun (2019): Repeat steps from above.
3. Quarter four…July-Sept (2019): Repeat from above.
4. Calculate the new ***ongoing LOD***…this may seem complicated, but it’s all in the spreadsheet -so use it! The two LODs and LODb results are compared and the highest is the **new LOD.**

Some things I will point out: The spreadsheet is set up to use the spiked blanks from the previous year (2018) and also, -in this futuristic case, the current year (2019). The new method says to use the last 24 months for all spiked blanks that are the same concentration.

There are also options for the method blanks. I’ll explain it this way: if you have less than 100 method blanks in a year, then the mean+standard deviation of all your 2019 method blanks is the LODb. If you have less than 100, **skip** to #6.

*If* you have more than 100 method blanks, then you may use one of these method blank results: You can choose to use the 99th percentile or the standard deviation result. This is all in the spreadsheet; it does the math for you.

For either method blank option, these are calculated:



1. Once you are ready, use the same 10/3 factor you did in the past to calculate your LOQ.

LOD x 3.33 = LOQ.



What’s next? The **NEXT *ongoing LOD***!

This thing never ends (*just like the old procedure*).

In our case above, the 2018 and 2019 standards spiked blanks were used to generate the LOD…a similar thing happens in the next year. But *this time* it is the data from 2019 and 2020 (24 months *if the standard spiked blank concentrations used are the same*). Plus, all the method blanks (up to 24 months worth), but remember you have options with this if you run a lot of batches each year). The higher of the two (LODs & LODb) = LOD.

So now you have an LOD from 2019 and one from 2020. You have a **choice** to make… for example, the LOD from 2019 was 0.026. And the LOD from 2020 is 0.035. Which do you use? With the new procedure, you may keep the 2019 LOD because it did not change by more than a factor of **2**, or you can use the new one (if no more than 3% of your blanks failed). It’s totally up to you! However, you must use the new LOD if it is more than a factor of 2 different.

Wait…I have questions! These address situations that could affect either the Initial LOD or the Ongoing LOD.

Q. But what if you recalibrate, then what?

A. This is one change that doesn’t require you to start over. This assumes the calibration has the same calibration standard concentrations for the curve you have used previously. Just keep processing the quarterly spiked blanks *as if nothing is different*.

Q. What if something looks wrong, what if the blank LOD (LODb) is higher than the spiked blanks (LODs)…is that normal?

A. It could be just fine. The new procedure accounts for blank contamination and variability.

Q. What if one of the spiked blanks is really different, or if one of the method blanks is really out of whack?

A. Take a close look at what caused that - those results cannot be removed unless there is a specific reason for doing so (as in I forgot to spike that spiked blank, or the vial was leaking).

If you have more questions, then please call or email your friendly neighborhood auditor. Contacts for program staff are on our website: <https://dnr.wi.gov/regulations/labcert/>

