# NR 149 Update for Certified Labs – 7.21.21 Presentation – Questions/Answers

*Not every answer provided may be applicable to your specific issue or question. Please don't hesitate to contact Lab Cert staff if you have any questions at all!*

**Question (Q): Why wasn't the presentation provided before the 7/1/21 effective date?**
**Answer (A):** Due to resource issues, the webinar was not completed until 7/21/21; however, we are not requiring the updated code be implemented until 9/1/21.

**(Q): Can you explain when CCVs need to be processed?**
**(A):** CCV standards shall be treated the same as the standards used in the initial calibration. **NR 149.446 (1)(b)**

If the method does not require the calibration curve standards to be processed (extracted, digested, distilled, etc.) and the laboratory does not process the calibration curve standards, then the CCV does not need to be processed. However, if the laboratory does choose to process the calibration curve standards when it is not a method requirement, then the CCV must also be processed the same way.

For example, SM 4500-Norg D indicates that digesting the calibration curve standards is optional. If the lab does digest the calibration curve standards, the CCV must be digested as well.

Another example would be EPA 548.1. This method requires that the calibration curve standards be derivatized, and in this case, the CCV would also need to be processed (derivatized) like the calibration curve standards.

Also, when the method requires that the standards be treated the same as samples, the CCV standards shall be performed with the associated batch so that the CCV standards and samples are all processed together.

**(Q): When are the ICV and CCV limits in NR 149 of ±10% for inorganics and ±20% for organics required? What if the method has different limits?**
**(A):** Unless otherwise specified in the method, regulation or covered program, the NR 149 recovery limits will apply. **NR 149.444 (7)(a), (b); NR 149.446 (4)(a), (b)**

NR 149 limits apply when the method does not specify the recovery limits for the ICV and/or the CCV. If the method includes ICV and/or CCV limits, the lab needs to be able to meet the method limits. For example, the method may have tighter limits, such as 95-105%, so those limits must be used. Some methods may allow wider limits, in that case, the lab may use those wider limits.

**(Q): When data is required to be reported to the department electronically, what are my options for qualifying sample results?**
**(A):** The department requires that results submitted to the department be qualified when anomalies or deviations from requirements are encountered. **NR 149.03 (55), NR 149.47 (2)(d)(13)**

When results are only submitted to the department electronically, there are three options to provide qualification of results:

1. The results can be qualified using a qualifier code placed in a qualifier field that is available in the EDD.

2. The results can be qualified using a written statement placed in a field that is available in the EDD.

3. If neither 1 or 2 above is possible, the results can be submitted by EDD and a written statement of qualifications can be sent separately by mail or email to the department.

The bottom line is that results requiring qualification must be received by the department as qualified.

**(Q): What is needed when a PT supplied by the vendor does not include an analyte for which the laboratory has accreditation?**
**(A):** When a lab is certified for individual compounds, those compounds need to be fortified (spiked) in the PT if the department requires a PT for the compound. **NR 149.22 (1)(a)**

The laboratory certification website includes helpful information on PT topics:
https://dnr.wisconsin.gov/topic/labCert/program.html

**(Q): For PCB Aroclors, when is a CCV required for Aroclors other than 1016 and 1260?**
**(A):** For PCB Aroclor analysis, instruments are frequently calibrated and verified with a mixture that is made with 1016 and 1260 Aroclors. However, whenever other Aroclors are detected in a compliance sample, such as 1254 and 1248, a CCV for the Aroclors detected, such as 1254 and 1248, is required. **NR 149.446 (1)(a)**

**(Q): For an example, if Aroclor 1254 is detected, can the CCV be analyzed after the sample has been analyzed?**
**(A):** Yes, that is reasonable. Keep in mind, analyze the CCV on the same instrument and with the same instrument conditions as was used for the sample.

**(Q): For PCB Aroclors detected in samples that are not 1016 or 1260, is one CCV per instrument acceptable? What if there are more than 20 samples analyzed?**
**(A):** Unless otherwise required by the method or program, one CCV on the same instrument and the same instrument conditions used for the sample is acceptable.

**(Q): For PCB Aroclors detected in samples that are not 1016 or 1260, could the LCS or matrix spike be used as verification?**
**(A):** If the laboratory's LCS included the non-1016/1260 Aroclor and CCV recovery limits were met, this would be acceptable. However, there is not an allowance to use the matrix spike to meet calibration verification requirements.

**(Q): If I dilute a sample (for example, by a factor of 2), do I need to analyze my LOD study using a dilution factor of 2?**
**(A):** No, the LOD (MDL) study does not need to be completed using sample dilutions. For example, if a sample for benzene was diluted by a factor of 2 because it was over range, the LOD reported for benzene needs to be adjusted by the dilution factor of 2. **NR 149.48 (2)(d)**

**(Q): If I concentrate the sample (for example, by a factor of 2), can I adjust the LOD by that same factor?**
**(A):** During the live Q&A discussion on 7/21/21, it was indicated that this would be an option. However, after more consideration and research, it was determined that the laboratory must be able to verify the new, lower LOD that utilized the concentration step. **NR 149.48 (2)(b), (d), (f)**

If concentrating the sample is performed on a routine basis then the initial and ongoing LOD studies must incorporate this concentration step.

If concentrating the sample is performed on a non-routine basis, then the laboratory must complete a one-time initial LOD study over 3 days with at least 7 LOD spikes and 7 method blanks all performed with the concentration step. If the concentrated procedure LOD is less than or equal to the normal LOD adjusted for the concentration factor, then the lab has completed the necessary verification and may use it. In this situation, the method blank and LCS must be treated the same way as the concentrated sample to demonstrate acceptability of the concentration step.

**(Q): For cBOD, since we add inhibitor to the samples, why can't we add inhibitor to the GGA?**
**(A):** The GGA limits from the method are based on BOD. This is the reason to not add inhibitor to the GGA standard and why it is specifically added to the updated code. **NR 149.50 (1)(h)**

**(Q): Do calibration curves which use a quadratic function no longer need 2 different CCVs?**
**(A):** That is correct, only one CCV is needed. If the calibration function is *cubic*, then the lab would need two CCVs at two different concentrations. **NR 149.446 (3)(a), (b)**

**(Q): In a case where the beginning CCV passes, then 10 samples are analyzed, then a CCV fails, then 10 more samples are analyzed and the ending CCV passes, can we accept the data, or must a passing CCV immediately follow the failure?**
**(A):** A passing CCV must immediately follow any failed CCV or the samples analyzed before and after any failed CCV must be reanalyzed or qualified appropriately. **NR 149.47 (5) (b)**

**(Q): Could you confirm that sulfide interference techniques (when required) need to be able to detect 10 mg/L or less? For chlorine, interference techniques need to detect 0.1 mg/L or less?**
**(A):** Both of the detection levels indicated above are correct. **NR 149.50 (1)(m), (2)(c)**

**(Q): Can you confirm that we are no longer able to use expired standards and reagents – even if we demonstrate they are still good? This may become an issue for us if we continue to see issues with obtaining supplies due to supply chain issues.**
**(A):** The allowance for using expired standards and reagents has been removed from code. Chemicals must be used by the specified expiration date (either as specified by the manufacturer or as specified by the method). If the laboratory has contacted all vendors and is still not able to find a supplier of the standard or reagent, please email us with the affected chemical(s) along with all the vendors that were contacted, and we will try to help find a solution. **NR 149.39 (3)(d)**

**(Q): Are blank verifications for new lots of consumables required or can we use the method blank to demonstrate cleanliness instead?**
**(A):** There is not a specific requirement in the updated code that requires this; however, if there is something required in the method, continue to complete those verifications. For example, oil and grease by EPA 1664 methods have a requirement for bottle blanks, and in this case, the method blank and bottle blank could be run as one QC sample. For this method, there is also the ability to use the COA if the vendor supplied checks meets the limit needed. If there is some advice needed for a specific method, please feel free to contact us.

**(Q): What is the lower temperature limit for samples received at the laboratory? Is it a temperature above 0°C, or is it above freezing?**
**(A):** For samples requiring preservation at ≤6°C, the requirement for the lower limit is above the sample's freezing point. If the temperature on receipt is less than 0°C but the laboratory determined the sample has not begun to freeze, then a qualifier for this would not be needed. If the temperature is measured to be 0°C or colder with no indication of freezing started, record that freezing was not observed to document that the sample did meet preservation requirements. Freezing has started if ice crystals are visible in the samples. **NR 149.442 (2)(c)**

**(Q): Can I check the melt water in the cooler to determine the temperature upon receipt?**
**(A):** Yes, either a sample, a temperature blank that was supplied with the samples, or the melt water would be acceptable. Make sure that each shipping container/cooler used for samples that require thermal preservation is checked and documented. **NR 149.442 (2)(d)**

**(Q): For our laboratory, the people who collect the samples place the samples in our walk-in cooler. In this case do we need to measure the temperature of the samples when we take them out of the cooler?**
**(A):** The temperature upon receipt needs to be checked when thermal preservation is required. Either the lab staff or the samplers will need to check the temperature and document it before placing the samples in the walk-in cooler. **NR 149.442 (2)(b)**

There is an exception to this if the samples are placed in the cooler in the laboratory within 15 minutes of collection. **NR 219 Table F Footnote 2**

**(Q): If we document from our own samplers that samples were received on ice and were put on ice at the time of collection, can we avoid taking the temperature upon receipt?**
**(A):** No, the laboratory would still need to take the temperature of these samples. See the exception for this in the previous question. **NR 149.442 (2)(b)**

**(Q): The requirement to add the resolution of interferences to the SOPs is quite significant. Do we need to update all the SOPs prior to 9/1/21?**
**(A):** We understand updating the SOPs can be time consuming, just do your best. It is suggested to start with the SOPs for those tests where there have been known interferences encountered by the lab. Once those are complete, continue the updates for the SOPs in which encountering an interference is not typical. **NR 149.40 (5)(c)**

**(Q): Is it correct that when relative standard error (RSE) is used, that the requirement is within 15%?**
**(A):** Remember, these new assessment tools (x-intercept, RSE, and residuals) are optional. If the laboratory does use RSE to evaluate the calibration curve, the RSD limit is 15% for inorganic analytes and 20% or organic analytes – UNLESS the method has a different limit. **NR 149.444 (6)(b)**

**(Q): If I am evaluating a linear calibration, I just need to meet the required r value, correct? I do not need to check the x-intercept, RSE, or residuals?**
**(A):** Yes, that is correct *(unless the method has additional or more stringent criteria).* These calibration assessment tools are options the lab may use. **NR 149.444 (6)**

**(Q): PT requirements for flame AA and LL Hg are changing on 9/1. Am I ok for this year (2021) if I don't have a PT?**
**(A):** Yes, for renewal of accreditation **this year,** the PTs for flame AA or low-level Hg are not required. Starting January 2022, make sure the lab analyzes a PT from an approved provider.

**(Q): If I have a 54-place digestion block and digest 50 samples in it, why shouldn't that be one preparation batch?**
**(A):** In order to set a limit for the number of samples in a preparation batch, the program decided to limit a preparation batch of samples to 20 environmental samples. That is consistent with many methods and is considered the industry standard. **NR 149.48 (5)(b)**

**(Q): The LOD has to be adjusted by the dilution factor, but the dilution factor is not required to be on the report?**
**(A):** That is correct – the reported LOD and LOQ do need to be adjusted when the amount of the sample used is different than what was used to determine the LOD (for example, if a dilution was performed during preparation and/or a dilution was performed during analysis). Because of the new requirement to adjust these for dilutions, there would not need to be a dilution factor supplied on the report, but of course, it's fine to include it. **NR 149.47 (2)(c)(10), NR 149.48 (2)(d)**

**(Q): The analysis <u>times</u> are required in the report?**
**(A):** That is correct but only *when the holding time for the analyte is in <u>hours</u>*. If the department requires reporting the results electronically, analysis times are only required when there is a field available to use in the EDD. **NR 149.47 (2)(d)(6)**

**(Q): In terms of the test report, if the preparation method and the analytical method are the same, can just the analytical method be listed?**
**(A):** That would be acceptable if the preparation and analytical method are the same and the lab does not perform multiple preparations for that analyte. If the department requires reporting the results electronically, preparation method and analytical method are only required when there are fields available to use in the EDD. **NR 149.47 (2)(d)(4)**

**(Q): This wasn't part of the webinar, but I thought I noticed that we have to update the department when there is a change in lab staff. Would you please include what is required for lab staff changes?**
**(A):** The requirement applies to lab director, lab manager, QA Manager, or whole effluent toxicity technical expert. If these key personnel change, notify the department within 30 days of that change (email notification is acceptable). This is an example of a detail that was not discussed in the webinar (not all changes were addressed). **NR 149.155 (4)**

**(Q): "The laboratory shall perform microwave preparations with instruments that utilize temperature feedback control." I am also not sure what temperature feedback control means when using the microwave instrument.**

**(A**): When using a microwave for digestion, the primary way to demonstrate acceptable performance is to have a temperature feedback control mechanism as part of the instrument capability. What that means is that the instrument must have the capability of sensing the temperature of each vessel during the extraction and automatically adjusting the microwave field output power within 2 seconds of sensing if that temperature measured is not within 2.5 °C of the required extraction temperature. A microwave that has this capability and one that can demonstrate that it is performing as required is needed. **NR 149.50 (17)(b)**