Total Phosphorus - Hach 10210 (TNT 843, 844) Method Checklist * REV. 11/17/23

Based on NR 149 (2021), NR 219 (2022), and Hach 10210 [equivalent to Standard Methods 4500-P B. (5), E. (1999, 2011)]

Some questions may not be applicable to every lab. If applicable, all answers must be "yes" to be in compliance.

***This checklist was created for the aid of registered laboratories. It is only an internal audit guideline; it is not meant to be comprehensive of all regulatory requirements, to dictate DNR audit format, or to include all acceptable method options. Laboratories must comply with all applicable code and method requirements whether listed on this checklist or not. Additional general NR 149 requirements are on a separate checklist.**

	Sample Storage and Handling	Y	N	Notes	Reference
1	If preparation/analysis is not started immediately (≤15 minutes), are the samples stored at ≤6°C (but above its freezing point) prior to analysis?				NR 219 Table F; NR 149.442 (4)(b)
2	If preparation/analysis is not started immediately (≤15 minutes), are the samples preserved using sulfuric acid?				NR 219 Table F
3	When samples are preserved, is the pH measured to ensure it is <2?				NR 149.442 (2)(e)
4	Are preserved samples analyzed within the hold time of 28 days?				NR 219 Table F

	Reagents and Standards	Y	N	Notes	Reference
5	Are Hach vials, reagents, and standards unexpired?				NR 149.39 (3)(d)
6	Are reagents properly labeled (with chemical name, concentration, and expiration date)?				NR 149.39 (3)(a)
7	If different types of vials (e.g., 843 and 844) are used for compliance samples, are separate calibration curves used?				NR 149.444 (1), NR 149.48 (2)(b)

	Equipment	Y	N	Notes	Reference
8	Is the temperature of the block digestor verified annually?				NR 149.44 (3)(d)(1)
9	Is the block digestor able to maintain 148 - 152°C during digestion?				NR 149.44 (1), 149.50 (2)(b)
10	Is absorbance measured at 650 or 880 nm?				Hach 10210 (2.1)

	Digestion and Sample Measurement	Y	N	Notes	Reference
11	Are samples and standards warmed to room temperature just before analysis?				Hach 10210 (11.2.2.2)
12	If acid preserved, are samples and standards neutralized (i.e., pH of 6 - 8) with NaOH before aliquoting?				Hach 10210, Test Kit Instructions, Sample collection, preservation, and storage section
13	Is the sample volume accurately measured with a verified mechanical pipette or class A pipette?				NR 149.44 (1)
14	If using 843 vials, is 2 mL of sample added to the vials? If using 844 vials, is 0.5 mL of sample added to the vials? If using ULR vials, is 3.5 mL of sample added to the vials?				Hach 10210 (11.2.2.4); Hach Application Note LIT2097
15	Is the DosiCap Zip then added to the vial?				Hach Method 10210 Revision 2 (11.2.2.5)

16	After the vial is capped tightly and shaken, is it then placed in the pre-heated block digester at 150 ºC ± 2 °C for 30 minutes?				NR 149.50 (2)(b); Hach 10210 (11.2.2.6 - 11.2.2.7)
17	After the vial is cooled to room temperature, is Reagent B added, the DosiCap from Bottle C attached, and the vial shaken 2-3 times?				Hach Method 10210 Revision 2 (11.2.2.8 – 11.2.2.10)
18	After the vials are shaken, are the samples read on the spec after 10 minutes? *(Try to be consistent for all samples and QC.)*				Hach Method 10210 Revision 2 (11.2.2.11)
19	Is an instrument blank (without color reagent) used to zero the spectrophotometer before standards or samples are measured?				NR 149.444 (1)(e)
20	If the sample response is above the response of the highest calibration standard, is a new portion of the sample diluted in a new vial and re-analyzed?				NR 149.47 (1)(c), NR 149.50 (2)(d)
21	Are the concentration results calculated from the measured absorbance (and not from the barcode reading)?				NR 149.444 (1)(f)
22	If a sample is highly colored or turbid, is the absorbance of the sample without color reagent measured and recorded and then the concentration subtracted from the reacted sample concentration?				NR 149.45
23	Is it understood that the method blank result cannot be subtracted from the sample result?				NR 149.48 (1)(b)

	Calibration	**Y**	**N**	**Notes**	**Reference**
24	Is the calibration curve performed by the lab (i.e., not the vendor's or built-in calibration)?				NR 149.444 (1)(f)
25	Is the calibration curve redone if there are two consecutive CCV failures, the instrument leaves the lab, after non-routine maintenance, or conditions change the expected behavior of the instrument?				NR 149.44 (4)(d), NR 149.444 (1)(c), NR 149.446 (5)
26	Are at least 3 non-zero standards used for a linear calibration curve or at least 5 non-zero standards used for a quadratic curve?				NR 149.444 (2), (2)(e)
27	Is the concentration of the highest calibration standard at or below the top range of the Hach vial? (i.e., highest standard is ≤ 1.5 mg/L for 843 vials, ≤ 5 mg/L for 844 vials, and ≤ 20 mg/L for 845 vials)				Hach 10210 (1.3), (7.2)
28	Does the curve include a calibration blank that is treated the same as the other calibration standards and uses the instrument response?				NR 149.50 (2)(a)
29	Is the calibration curve not forced through zero?				NR 149.444 (4)(d)
30	Does the curve have a correlation coefficient (r) of at least 0.995 for a linear curve or a coefficient of determination (r^2) of at least 0.995 for a quadratic curve?				NR 149.444 (6)(e), (6)(f)

	Calibration Verification and Quality Control	**Y**	**N**	**Notes**	**Reference**
31	Is a second source standard (ICV) always analyzed after the initial calibration and before samples are analyzed?				NR 149.444 (7)
32	Is the ICV within 90 - 110% of the true value unless otherwise specified in the method?				NR 149.444 (7)(a)

33	Is the CCV performed first (before method blanks and samples) on non-calibration days?				NR 149.446 (1), (2)(a)
34	If more than 20 samples are analyzed in a batch, is a CCV analyzed after the 20th sample?				NR 149.446 (2)
35	Is the CCV within 90 - 110% of the true value?				NR 149.446 (4)(a)
36	If the initial calibration standards are digested, are the ICV and CCV also digested?				NR 149.444 (7), NR 149.446 (1)(b)
37	Is a method blank run with each sample batch (up to 20 samples) and processed in the same manner as the samples?				NR 149.48 (5)(a), (5)(b)
38	Is the method blank less than the highest of the LOD, 5% of the regulatory limit, or 10% of the sample concentrations?				NR 149.48 (5)(d)

	Reporting and Qualifiers	**Y**	**N**	**Notes**	**Reference**
39	Are samples results that are less than the MDL reported on the eDMR as "<" the MDL value?				NR 149.47 (1)(a); WPDES Permit
40	Are all reported MDLs adjusted for any dilution (i.e., adjust when the sample amounts used are different than those used for the MDL determination)?				NR 149.48 (2)(d)
41	Are results qualified if samples were analyzed past hold time? *(If preserved, hold time is 28 days; if unpreserved, digestion/analysis must be started within 15 minutes of collection)*				NR 219 Table F; NR 149.47 (4)(b)
42	Are results qualified if samples were not digested for 30 minutes at 148 - 152°C?				NR 149.47 (5), NR 149.50 (2)(b)
43	Are results qualified if the method blank fails?				NR 149.47 (5), NR 149.48 (5)(d)

	Documentation and Records - Are all of the following documented or recorded, if applicable?	**Y**	**N**	**Notes**	**Reference**
44	Sample collection date				NR 149.45
45	Acid preservation verification				NR 149.45
46	Sample storage temperature				NR 149.45
47	Digestion date				NR 149.45
48	Digestion analyst				NR 149.45
49	Digestion temperature				NR 149.45
50	Analyst				NR 149.45
51	Analysis date				NR 149.45
52	Unique sample IDs				NR 149.442 (1)(d); NR 149.45
53	Lot or lab ID of standards				NR 149.45
54	Lot or lab ID of reagents (TNT vials)				NR 149.45
55	Sample neutralization				NR 149.45
56	Sample volume				NR 149.45
57	No prefilled volumes				NR 149.45
58	Raw data (absorbance)				NR 149.45

59	Units (e.g., mL, abs)				NR 149.45
60	Sequence of analysis is clear				NR 149.45
61	Calibration identification (e.g., date or link to calibration data)				NR 149.45
62	Corrections made to data were done properly (crossed out with a single line; not scribbled out or overwritten)				NR 149.39 (1)(g), NR 149.45
63	Corrective actions taken (e.g., when temperatures are out of range, analyzed past hold time, QC failures, etc.)				NR 149.38 (3), NR 149.45
64	Instrument maintenance				NR 149.45

Other Observations

WI DNR Total Phosphorus Resources

DNR Website (which includes the resources below): Laboratory Certification | Wisconsin DNR
Example Hach 10210 SOP template
Example total phosphorus colorimetric benchsheets (includes standard preparation calculator)
Example thermometer annual verification log
Example digestion block annual temperature verification log
Example total phosphorus and ammonia preservation and neutralization tracking log
Example daily equipment temperature measurements log
Example auto-pipette quarterly verification log
Example equipment maintenance log
Example prepared and purchased chemical tracking logs
Example general corrective action log
Lab Accreditation Program staff - contact any staff with questions or concerns, especially if there are ongoing QC issues