

Total Phosphorus - Hach 8190 PhosVer 3™ Method Checklist * REV. 11/17/23

Based on NR 149 (2021), NR 219 (2022), and Hach 8190 [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 $\leq 6^{\circ}\text{C}$ (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 kits, 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)

	Equipment	Y	N	Notes	Reference
7	Is the temperature of the block digester verified annually?				NR 149.44 (3)(d)(1)
8	Is the block digester able to maintain $148 - 152^{\circ}\text{C}$ during digestion?				NR 149.44 (1), 149.50 (2)(b)
9	Is absorbance measured at 650 or 880 nm?				Hach 8190 (2.1)

	Digestion and Sample Measurement	Y	N	Notes	Reference
10	Are samples and standards warmed to room temperature just before digestion?				Hach 8190 (11.2); DOC316.53.01121, Sample collection and storage section
11	If acid preserved, are samples and standards neutralized (i.e., pH of 6 - 8) with NaOH before aliquoting?				Hach 8190 (11.2); DOC316.53.01121, Sample collection and storage section
12	Are 5 mL of the sample volume (or the volume specified for the vials) accurately measured into the vial with a verified mechanical pipette or class A pipette?				NR 149.44 (1)
13	Are the contents of one potassium persulfate powder pillow added to the vial?				Hach 8190 (11.2); DOC316.53.01121
14	After the vial is capped tightly and shaken, is it then placed in the pre-heated block digester at $150^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for 30 minutes?				NR 149.50 (2)(b); Hach 8190 (11.2); DOC316.53.01121
15	After the vials have cooled, is 2 mL of the 1.54 N NaOH solution added to the vials?				Hach 8190 (11.2); DOC316.53.01121

16	Are the contents of a PhosVer 3 pillow (suitable for 5 mL sample volume) added to each vial?				Hach 8190 (11.2); DOC316.53.01121
17	After the vials are shaken, are the samples read on the spec between 2 and 8 minutes? (<i>Try to be consistent for all samples and QC.</i>)				Hach 8190 (11.2); DOC316.53.01121
18	Is an instrument blank (without color reagent) used to zero the spectrophotometer before standards or samples are measured?				NR 149.444 (1)(e)
19	If the sample response is above the response of the highest calibration standard, is the sample diluted and re-analyzed?				NR 149.47 (1)(c), NR 149.50 (2)(d)
20	Are the concentration results calculated from the measured absorbance (and not from the barcode reading)?				NR 149.444 (1)(f)
21	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; Hach 8190 (11.2); DOC316.53.01121 Interferences section
22	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
23	Is the calibration curve performed by the lab (i.e., not the vendor's or built-in calibration)?				NR 149.444 (1)(f)
24	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)
25	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)
26	Is the concentration of the highest calibration standard at or below the top range of the Hach vial? (i.e., highest standard is ≤ 20 mg/L for PhosVer 3 kits)				Hach 8190 (1.4)
27	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)
28	Is the calibration curve not forced through zero?				NR 149.444 (4)(d)
29	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
30	Is a second source standard (ICV) always analyzed after the initial calibration and before samples are analyzed?				NR 149.444 (7)
31	Is the ICV within 90 - 110% of the true value unless otherwise specified in the method?				NR 149.444 (7)(a)
32	Is the CCV performed first (before method blanks and samples) on non-calibration days?				NR 149.446 (1), (2)(a)
33	If more than 20 samples are analyzed in a batch, is a CCV analyzed after the 20th sample?				NR 149.446 (2)
34	Is the CCV within 90 - 110% of the true value?				NR 149.446 (4)(a)

35	If the initial calibration standards are digested, are the ICV and CCV also digested?				NR 149.444 (7), NR 149.446 (1)(b)
36	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)
37	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
38	Are samples results that are less than the MDL reported on the eDMR as "<" the MDL value?				NR 149.47 (1)(a); WPDES Permit
39	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)
40	Are results qualified if samples were analyzed past hold time? (<i>If preserved, hold time is 28 days; if unpreserved, digestion/analysis must be started within 15 minutes of collection</i>)				NR 219 Table F; NR 149.47 (4)(b)
41	Are results qualified if samples were not digested for 30 minutes at 148 - 152°C?				NR 149.47 (5), NR 149.50 (2)(b)
42	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
43	Sample collection date				NR 149.45
44	Acid preservation verification				NR 149.45
45	Sample storage temperature				NR 149.45
46	Digestion date				NR 149.45
47	Digestion analyst				NR 149.45
48	Digestion temperature				NR 149.45
49	Analyst				NR 149.45
50	Analysis date				NR 149.45
51	Unique sample IDs				NR 149.442 (1)(d); NR 149.45
52	Lot or lab ID of standards				NR 149.45
53	Lot or lab ID of reagents (PhosVer kit)				NR 149.45
54	Sample neutralization				NR 149.45
55	Sample volume				NR 149.45
56	No prefilled volumes				NR 149.45
57	Raw data (absorbance)				NR 149.45
58	Units (e.g., mL, abs)				NR 149.45
59	Sequence of analysis is clear				NR 149.45
60	Calibration identification (e.g., date or link to calibration data)				NR 149.45

61	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
62	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
63	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 8190 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	