

Total Phosphorus - Ascorbic Acid Method Checklist *

Based on NR 219 (2004), NR 149 (2008), HACH 8190 and Standard Methods (18th, 19th and 20th Editions)

*This checklist is for the aid of the Department and the laboratory. It is only an audit guideline, it is not meant to establish regulatory standards or to dictate audit format. Laboratories must comply with all applicable rule and method requirements whether listed on this checklist or not. The general quality control and record requirements are covered on a separate QC and Records Checklist.

Sample Storage and Pretreatment		Y	N	Notes	Citation
1	Are phosphorus samples stored at $\leq 6^{\circ}\text{C}$ prior to analysis?				NR 219; Table F
2	If analysis is not started immediately (≤ 15 minutes) is the sample preserved at pH of <2 using sulfuric acid?				NR 219; Table F
3	Are samples analyzed within the hold time of 28 days (preserved at pH <2 and $\leq 6^{\circ}\text{C}$)?				NR 219; Table F
4	What is digested? ___ samples ___ Cal standards ___ cal blank ___ instr.blank ___ MB ___ LCS ___ CCV	NA	NA		NA

Equipment		Y	N	Notes	Citation
5	Are all apparatus, reagents and glassware available?				4500-P-E; 2&3.
6	Does glassware cleaning include a hydrochloric acid rinse or is clean glassware stored filled with distilled water and subjected to occasional acid rinse?				4500-P-E; 2.

Standard Persulfate Digestion		Y	N	Notes	Citation
7	Is combined reagent made fresh daily & used w/in 4 h?				4500 P-E; 3.
8	Are reagent to sample volumes proportional to those required by the method?				4500-P-B(5)c.
9	Is mixed sample measured into a flask, then phenolphthalein indicator added? If red color appears is sulfuric acid solution added until it clears?				4500-P-B(5)c.
10	Then is sulfuric acid solution and either ammonium persulfate or potassium persulfate added to the flask?				4500-P-B(5)c.
11	Are samples gently boiled on a hot plate for 30-40 minutes or until the volume is 10 mL?				4500-P-B(5)c.
12	Alternatively, are samples heated for 30 minutes in an autoclave or pressure cooker at 98 to 137 kPa?				4500-P-B(5)c.
13	Is the sample then cooled, diluted with RW, phenolphth added & then neutralized w/NaOH to a faint pink color?				4500-P-B(5)c.
14	Is digested sample pipetted into a flask, phenolphthalein indicator added, and if red 5N sulfuric acid added until the solution is clear?				4500-P-E(4)a.
15	Is combined reagent then added and mixed?				4500-P-E(4)a.

HACH Persulfate Digestion		Y	N	Notes	Citation
16	If samples are preserved prior to analysis, are they warmed to room temperature and adjusted to pH 7 before beginning digestion (record volumes of base added)?				HACH 8190
17	Is 5 mL of neutral sample added to a test vial (already contains acid) & 1 K persulfate pillow added.				HACH 8190
18	Vial is capped tightly and shaken, then placed in the pre-heated block digester at 150°C for 30 minutes?				HACH 8190
19	Is the sample cooled, 2 mL of 1.54N sodiumOH std solution added to the vial, then capped and shaken?				HACH 8190
20	Is contents of one phosver3 phosphate pillow then added to vial, capped, shaken & 2 min timer started?				HACH 8190

Calibration/Sample Measurement		Y	N	Citation
21	Is the spectrophotometer calibrated annually with at least 3 standards and a blank? Also calibrated after CCV failure or non-routine maintenance?			NR149.44(6)(d)
22	Is the calibration curve acceptable (0.995 or better), and properly used to derive sample concentrations based on absorbance data?			NR149.44(6)(g)(2)
23	Are samples above the upper end of the calibration curve diluted and re-analyzed with minimal dilution?			NR 149.44(6)(L)
24	For samples digested using autoclave/hotplate Is the sample absorbance measured between 10 and 30 minutes after the addition of the combined reagent?			4500-P-E(4)a.
25	For samples digested following HACH method is the vial wiped clean and the absorbance measured between 2 and 8 minutes after phosphor3 pillow packet was added?			HACH 8190
26	Is absorbance measured at the appropriate wavelength - (610, 660, 880, or 890 depending on the method)?			4500-P-E HACH 8190
27	Is an instrument blank (RW no CR) used to zero and a calibration blank (RW with CR) used as the 0 point in the curve?			149.44(5)(h)
28	Is a method blank run with each sample batch and processed in the same manner as the samples?			NR149.48(3)(b)
29	Is method blank below highest of: LOD, 5% regulatory limit or 10% sample concentration?			NR149.48(3)(d)
30	On days when ICAL (initial calibration) is analyzed is a 2nd source ICV analyzed right after calibration?			NR149.44(6)(i)
31	If 2nd source ICV is not run, is a QCS run three times a year?			NR149.48(5)(a)
32	Are CCVs analyzed after every 20 samples and before sample analysis if no ICAL run that day?			NR149.44(7)(a)
33	Does CCV meet the 10% recovery criteria?			NR149.44(7)(d)(1)
34	Is an LCS analyzed with each batch (unless ICAL, ICV and CCV are digested then the CCV can cover LCS)?			NR149.48(4)(a)

Other Observations				