

Ammonia - Hach 10205 (TNT 830, 831, 832) Method Checklist * REV. 11/17/23

Based on NR 149 (2021), NR 219 (2022), and Hach 10205 [equivalent to EPA 350.1, Standard Methods 4500-NH₃ F (1997 and 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 analysis is not started immediately (≤ 15 minutes), are the samples stored at $\leq 6^{\circ}\text{C}$ (but not frozen) prior to analysis?				NR 219 Table F; NR 149.442 (4)(b)
2	If 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	If wastewater samples are collected after chlorination, are samples checked for residual chlorine?				SM 4500-NH ₃ A (2); EPA 350.1 (4.2)
5	If residual chlorine is present, is it removed with sodium thiosulfate or sodium sulfite?				SM 4500-NH ₃ A (2), B (3)(d); EPA 350.1 (4.2), (7.5)
6	Are preserved samples analyzed within the hold time of 28 days?				NR 219 Table F

Reagents and Standards		Y	N	Notes	Reference
7	If the ammonia standard is prepared from ammonium chloride, is it dried first ($\sim 100 - 105^{\circ}\text{C}$)?				SM 4500-NH ₃ F (3)(f), D (3)(d); EPA 350.1 (7.11)
8	If standards are purchased, are they stored as indicated by the manufacturer?				Manufacturer's requirements
9	Are Hach kits, reagents, and standards unexpired?				NR 149.39 (3)(d)
10	Are reagents properly labeled (with chemical name, concentration, and expiration date)?				NR 149.39 (3)(a)
11	If different types of vials (e.g., 830 and 831) are used for compliance samples, are separate calibration curves used?				NR 149.444 (1), NR 149.48 (2)(b)

Equipment		Y	N	Notes	Reference
12	Is absorbance measured at $690 \pm 5 \text{ nm}$?				Hach 10205 (2.1)

Distillation - Industrial Treatment Plants Only		Y	N	Notes	Reference
13	Unless the lab has completed an approved study, are industrial (i.e., non-municipal) waste samples distilled? <i>This checklist does not include distillation procedure requirements. See SM 4500-NH₃ B for details.</i>				Hach 10205 (2.3); SM 4500-NH ₃ A (1), B, F; EPA 350.1 (11.3); NR 219 Table B Parameter 4

Sample Measurement		Y	N	Notes	Reference
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14	Are samples and standards warmed to room temperature just before analysis starts?				Hach DOC 316.53.010181 Sample collection, preservation and storage section
15	If acid preserved, are samples and standards neutralized (i.e., pH of 6 - 8) with NaOH before aliquoting?				Hach 10205 (8.2)
16	Is the sample volume accurately measured with a verified mechanical pipette or class A pipette?				NR 149.44 (1)
17	If using 830 vials, is 5 mL of sample added to the vials? If using 831 vials, is 0.5 mL of sample added to the vials? If using 832 vials, is 0.2 mL of sample added to the vials?				Hach 10205 (11.2.2)
18	Is the cap flipped over after the sample is added, is the vial shaken 2-3 times, and is it verified that the reagent is dissolved?				Hach 10205 (11.2.3 11.2.4)
19	Do you set a timer set for 15 minutes?				Hach 10205 (11.2.5)
20	After the 15 minutes, is the vial shaken again 2-3 times, and the vial absorbance read within 15 minutes of the timer going off?				Hach 10205 (11.2.6)
21	Is an instrument blank (without color reagent) used to zero the spectrophotometer before standards or samples are measured?				NR 149.444 (1)(e)
22	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)
23	Are the concentration results calculated from the measured absorbance (and not from the barcode reading)?				NR 149.444 (1)(f)
24	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 DOC316.53.01081 Sample blanks section
25	The method blank is NOT used to adjust the sample results, correct?				NR 149.48 (1)(b)

	Calibration	Y	N	Notes	Reference
26	Is the calibration curve performed by the lab (i.e., not the vendor's or built-in calibration)?				NR 149.444 (1)(f)
27	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)
28	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)
29	Is the concentration of the highest calibration standard at or below the top range of the Hach vial? (i.e., highest standard is ≤ 2 mg/L for 830 vials, ≤ 12 mg/L for 831 vials, and ≤ 47 mg/L for 832 vials)				Hach 10205 (1.2), (7.2)
30	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)
31	Is the calibration curve not forced through zero?				NR 149.444 (4)(d)

32	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)
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	Calibration Verification and Quality Control	Y	N	Notes	Reference
33	Is a second source standard (ICV) always analyzed after the initial calibration and before samples are analyzed?				NR 149.444 (7)
34	Is the ICV within 90 - 110% of the true value unless otherwise specified in the method?				NR 149.444 (7)(a)
35	Is the CCV performed first (before method blanks and samples) on non-calibration days?				NR 149.446 (1), (2)(a)
36	If more than 20 samples are analyzed in a batch, is a CCV analyzed after the 20th sample (or as required by the method)?				NR 149.446 (2)
37	Is the CCV within 90 - 110% of the true value?				NR 149.446 (4)(a)
38	If the initial calibration standards are distilled, are the ICV and CCV also distilled?				NR 149.444 (7), NR 149.446 (1)(b)
39	Is a method blank processed with each sample batch (up to 20 samples) in the same manner as the samples?				NR 149.48 (5)(a), (5)(b)
40	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
41	Are samples results that are less than the MDL reported on the eDMR as "<" the MDL value?				NR 149.47 (1)(a); WPDES Permit
42	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)
43	Are results qualified if samples were analyzed past hold time? (<i>If preserved, hold time is 28 days; if unpreserved, distillation/analysis must be started within 15 minutes of collection</i>)				NR 219 Table F; NR 149.47 (4)(b)
44	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
45	Sample collection date				NR 149.45
46	Residual chlorine checks and treatment				NR 149.45
47	Acid preservation verification				NR 149.45
48	Sample storage temperature				NR 149.45
49	Distillation records				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 analyzed past hold time, QC failures, etc.)				NR 149.38 (3), NR 149.45
64	Instrument maintenance				NR 149.45

Other Observations

WI DNR Ammonia Resources

DNR Website (which includes the resources below): Laboratory Certification Wisconsin DNR
Example Hach 10205 SOP template
Example ammonia colorimetric benchsheets (includes standard preparation calculator)
Example thermometer annual 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