Ammonia - Ion-Selective Electrode (ISE) Method Checklist * REV. 11/17/23

Based on NR 149 (2021), NR 219 (2022), and Standard Methods 4500-NH_3 D (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 ≤6ºC (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)
5	If residual chlorine is present, is it removed with sodium thiosulfate or sodium sulfite?				SM 4500-NH_3 A (2), B (3)(d)
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 any reagent (e.g., NaOH/EDTA solution) is prepared in the lab, is it made according to the instructions in the method?				SM 4500-NH_3 D (3)
8	If the ammonia standard is prepared from ammonium chloride, is it dried at 100°C?				SM 4500-NH_3 D (3)(d)
9	If standards are purchased, are they stored as indicated by the manufacturer?				Manufacturer's requirements
10	Are reagents and standards unexpired?				NR 149.39 (3)(d)
11	Are reagents properly labeled (with chemical name, concentration, and expiration date)?				NR 149.39 (3)(a)

	Equipment	Y	N	Notes	Reference
12	Is the ammonia probe maintained properly (including regular membrane changes, filling solution changes, proper storage, etc., per manufacturer)?				NR 149.44 (1); Manufacturer's Owner's Manual

	Calibration and Sample Measurement	Y	N	Notes	Reference
13	Are samples and standards at about the same temperature during testing?				SM 4500-NH_3 D (1)(b), (4)(b)
14	Is the meter calibrated each use with at least 3 standards?				NR 149.444 (2), NR 149.50 (3)
15	Does the calibration yield a slope of 59 mV (-54 to -60) or a slope required by meter/probe manufacturer?				SM 4500-NH_3 D (4)(c), (4)(d); NR 149.444 (4); Manufacturer's Owners Manual

16	Is 100 mL (or suitable volume) of sample and standard used to do the analysis?				SM 4500-NH_3 D (4)(b), (4)(e)
17	Are samples and standards slowly stirred at a consistent rate?				SM 4500-NH_3 D (4)(b), (4)(e)
18	Is the NaOH buffer solution added to samples and standards after the probe is immersed in the sample?				SM 4500-NH_3 D (4)(b), (4)(e)
19	Is enough buffer used to raise the sample pH above 11 as evidenced by color indicating buffer or by checking the pH? *(EDTA is not to be used for high performance probes)*				SM 4500-NH_3 D (4)(b), (4)(e)
20	Is the meter allowed sufficient time to stabilize (as indicated by flashing "ready" or about 3 minutes for low level standards/samples)?				SM 4500-NH_3 D (4)(b)
21	If the same volume of buffer was not added to samples as was used for the standards, is the amount noted and used in the calculation?				SM 4500-NH_3 D (4)(b), (5)
22	If the sample response is above the response of the highest calibration standard, is a new portion of the sample diluted and re-analyzed?				SM 4500-NH_3 D (4)(e)

	Calibration Verification and Quality Control	**Y**	**N**	**Notes**	**Reference**
23	Is the ammonia glassware rotated so that the QC are not always in the same container?				NR 149.48 (1)(a)
24	Is a second source standard (ICV) always analyzed after the initial calibration and before samples are analyzed?				NR 149.444 (7)
25	Is the ICV within 90 - 110% of the true value unless otherwise specified in the method?				NR 149.444 (7)(a)
26	If more than 20 samples are analyzed in a batch, is a CCV analyzed after the 20th sample?				NR 149.446 (2)
27	If a CCV is analyzed, is it within 90 - 110% of the true value?				NR 149.446 (4)(a)
28	Is a method blank analyzed with each batch (up to 20 samples) and processed in the same manner as the samples?				NR 149.48 (5)(a), (5)(b)
29	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**
30	Are samples results that are less than the MDL reported on the eDMR as "<" the MDL value?				NR 149.47 (1)(a); WPDES Permit
31	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)
32	Are results qualified if samples were analyzed past hold time? *(If preserved, hold time is 28 days; if unpreserved, distillation/analysis must be started within 15 minutes of collection)*				NR 219 Table F; NR 149.47 (4)(b)
33	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**

34	Sample collection date				NR 149.45
35	Residual chlorine checks and treatment				NR 149.45
36	Acid preservation verification				NR 149.45
37	Sample storage temperature				NR 149.45
38	Analyst				NR 149.45
39	Analysis date				NR 149.45
40	Unique sample IDs				NR 149.442 (1)(d); NR 149.45
41	Lot or lab ID of standards				NR 149.45
42	Lot or lab ID of reagents (buffer)				NR 149.45
43	Sample volume				NR 149.45
44	Buffer volume				NR 149.45
45	No prefilled volumes				NR 149.45
46	pH verification after buffer addition				NR 149.45
47	Raw data (mV readings)				NR 149.45
48	Units (e.g., mL, mV)				NR 149.45
49	Sequence of analysis is clear				NR 149.45
50	Calibration slope				NR 149.45
51	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
52	Corrective actions taken (e.g., when analyzed past hold time, QC failures, etc.)				NR 149.38 (3), NR 149.45
53	ISE meter/probe maintenance				NR 149.45

Other Observations

WI DNR Ammonia Resources

DNR Website (which includes the resources below): Laboratory Certification | Wisconsin DNR
Example ammonia ISE SOP template
Example ammonia ISE benchsheet
Example thermometer annual verification log
Example total phosphorus and ammonia preservation 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