

Ammonia Ion-Selective Electrode (ISE) Method Checklist *

Based on NR 149 (2008), NR 219 (2004), EPA Method 350.3, 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 ammonia 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 (stored at pH <2 and $\leq 6^{\circ}\text{C}$)?				NR 219; Table F
4	Are samples and standards at room temperature before analysis starts?				19 & 20:4500 NH3-D; 4b. 18:4500 NH3-F; 4b.
5	Are samples distilled unless they are wastewater samples and a copy of the SLH distillation study is on file?				Lab Cert Allowance NR 219 Table B
6	If sample concentration is above the top standard is the sample diluted and re-analyzed?				4500 NH3-B; 4.e.

Equipment		Y	N	Notes	Citation
7	Are all the necessary apparatus, reagents and glassware available?				19&20: 4500-NH3-D; 2&3. 18: 4500 NH3-F; 2 & 3 350.3 (5&6)
8	Is ammonia -free water being used for analysis?				4500-NH3-B;3.a. 350.3 (6.0)
9	Is the ammonia probe maintained properly? <i>Including regular membrane changes, filling solution changes, proper storage (in 100 or 1000 ppm std.), etc.?</i>				NR149.44(2)(a)

Calibration/Sample Measurement		Y	N	Notes	Citation
10	Is 100 mLs of sample/standard used to do the analysis?				4500 NH3-B; 4.e.
11	Are samples slowly stirred using a thermally insulated magnetic stirrer?				4500 NH3-B; 4.b.
12	Is the NaOH buffer solution added to samples and standards <u>after</u> the probe is immersed in the sample?				19&20:4500 NH3-D; 4b 18:4500 NH3-F;4b. 350.3 (7.2)
13	Is enough buffer used to raise the sample pH above 11?				19&20:4500 NH3-D; 4b. 18:4500 NH3-F;4b. 350.3 (7.2)
14	If more than 1mL of buffer is added to samples (and less is added to the calibration standards) is the amount noted and used in the calculation?				19&20:4500 NH3-D; 4b. 18:4500 NH3-F;5.
15	Is the meter allowed sufficient time to stabilize (about 3 minutes on the low level standards/samples)?				4500 NH3-B; 4.b.& e.
16	Is the meter calibrated daily with at least 2 standards?				NR 149.44(6)(d)(2)
17	Is the meter calibrated starting with the lowest standard and progressing to higher standards?				19&20:4500 NH3-D; 4b. 18:4500 NH3-F;4b. 350.3 (7.2)
18	Does the calibration yield a slope of 59 mV (54-60) or slope required by meter manufacturer? <small>Orion -54 to -60 mV, Corning -50 to -60 mV, Acumet -55 to -63 mV Hach -54 to -62 mV</small>				19&20:4500 NH3-D; 4c. 18:4500 NH3-F;4c.