

NC DEQ/DWR WASTEWATER/GROUNDWATER LABORATORY CERTIFICATION BRANCH

LABORATORY NAME:	CERT #:
PRIMARY ANALYST:	DATE:
NAME OF PERSON COMPLETING CHECKLIST (PRINT):	
SIGNATURE OF PERSON COMPLETING CHECKLIST:	

Parameter: **pH**
Method: **SW-846 9045 D (Non-Aqueous)**

Equipment:

pH meter (type):		pH buffers (S.U.)	
		Value:	Exp:
Analytical Balance capable of weighing 0.1 g		Value:	Exp:
		Value:	Exp:

PLEASE COMPLETE CHECKLIST IN INDELIBLE INK
Please mark Y, N or NA in the column labeled LAB to indicate the common lab practice and in the column labeled SOP to indicate whether it is addressed in the SOP.

	GENERAL	L A B	S O P	EXPLANATION
1	Is the SOP reviewed at least every 2 years? What is the most recent review/revision date of the SOP? [15A NCAC 02H .0805 (g) (4)] Date:			Quality assurance, quality control, and Standard Operating Procedure documentation shall indicate the effective date of the document and be reviewed every two years and updated if changes in procedures are made. Verify proper method reference. During review notate deviations from the approved method and SOP.
2	Are all review/revision dates and procedural edits tracked and documented? [15 A NCAC 02H .0805 (g) (4)]			Each laboratory shall have a formal process to track and document review dates and any revisions made in all quality assurance, quality control and SOP documents.
3	Is there North Carolina data available for review?			If not, review PT data.
4	Are the following items documented with each analysis? [15A NCAC 02H .0805 (g) (2)]			
	The method or SOP reference			
	Laboratory identification			
	Instrument identification			
	Sample collector			
	Signature or initial of the analyst			
	Sample identification			
	Proper units of measure			
	Quality control assessments			
	Value from the measurement system			
	Final value to be reported			
	PRESERVATION and STORAGE	L A B	S O P	EXPLANATION
5	Are samples analyzed as soon as possible, within the same day of collection? [SW-846 9045 D (6.0)] [EPA Guidance]			https://www.epa.gov/hw-sw846/holding-time-preservation#question13

				SW-846 Q and A page - no definitive guidance regarding the interpretation of "as soon as possible" Method states samples should be analyzed as soon as possible. 40 CFR 136 Table II defines the maximum holding time for pH as 15 minutes. However, that does not apply to SW-846 methods and 9045D is a Method Defined Parameter that must be followed exactly as written with no modifications. The actual holding time for pH analysis should be specified in the QA plan. It is recommended performing the analysis within a few hours or receipt and not the next day.
6	Are date and time of sample collection documented? [15A NCAC 02H .0805 (g) (2) (F)]			
7	Are date and time of sample analysis documented? [15A NCAC 02H .0805 (g) (2) (G) and (H)]			One time may be documented for sample collection and analysis if there is documentation
	PROCEDURE – Meter Calibration	L A B	S O P	EXPLANATION
8	Is the meter calibrated daily before sample analysis with at least 2 buffers? [SW-846 9045 D (7.1.2)] [NC WW/GW LCB SW-846 pH Calibration Verification Policy] List Buffers:			Instruments are to be calibrated according to the manufacturer's calibration procedure prior to analysis of samples each day compliance monitoring is performed. Calibration must include at least two buffers. The meter calibration must be verified with a third standard buffer solution (i.e., calibration check buffer) prior to sample analysis. The calibration and check standard buffers must bracket the range of the samples being analyzed.
9	Is the meter calibration documented? [15A NCAC 02H .0805 (g) (3)]			
10	Is the time of meter calibration documented? [15A NCAC 02H .0805 (g) (3)]			
	PROCEDURE – Sample Analysis	L A B	S O P	EXPLANATION
11	Are samples stirred gently at a constant rate during measurement? [SW-846 9045 D (7.2)]			
12	If measuring pH values over 10 S.U. is a "low sodium error" electrode used? [SW-846 9045 D (3.1)]			Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10 S.U., the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solution, with a true pH of <1 S.U., may give incorrectly high pH measurements.
13	Is the electrode in good condition (i.e., no accumulation of debris, chloride precipitate, oily material, etc.)? [SW-846 9045 D (3.3)]			Errors will occur when the electrode becomes coated.
	Soil Sample Analysis			
14	Is 20 mL of reagent water added to 20 g of sample in a 50-mL beaker, covered, and the suspension allowed to stir continuously for 5 minutes? [SW-846 9045 D (7.2.1)]			Additional dilutions are allowed if working with hygroscopic soils and salts or other problematic matrices.
15	Is the aqueous phase collected for pH measurement after the suspension is centrifuged, filtered or allowed to stand for approximately 1 hr. to allow most of the suspended clay to settle out? [SW-846 9045 D (7.2.2)]			Let the soil suspension stand for 1 hr. to allow of the suspended clay to settle out from the suspension or filter or centrifuge off the aqueous phase for pH measurement.
16	Is the pH sensing portion and reference junction completely immersed? [SW-846 9045 D (7.2.3)]			Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant solution to establish a good electrical contact through the ground-glass joint or the fiber-capillary hole. Insert the electrodes into the sample solution in this manner. For combination electrodes, immerse just below the suspension.
17	If the sample temperature differs by more than 2 °C from the buffer solution are the measured pH values corrected? [SW-846 9045 D (Section 7.2.4)]			Report the results as "soil pH measured in water at ___ °C" where "___ °C" is the temperature at which the test is conducted.
	Waste Sample Analysis			
18	Is 20 mL of reagent water added to 20 g of sample in a 50-mL beaker, covered, and the suspension allowed to stir continuously for 5 minutes? [SW-846 9045 D (7.3.1)]			Additional dilutions are allowed if working with hygroscopic soils and salts or other problematic matrices.

19	Is the aqueous phase collected for pH measurement after the suspension is centrifuged, filtered or allowed to stand for about 15 min. to allow most of the suspended waste to settle out? [SW 846 9045 D (7.3.2)]			Let the waste suspension stand for about 15 min. to allow most of the suspended waste to settle out from the suspension or filter or centrifuge off the aqueous phase for pH measurement.
20	If the waste is hygroscopic, is 20g of sample and 40 mL of reagent water used? [SW-846 9045 D (7.3.2)]			If the waste is hygroscopic and absorbs all the reagent water, begin again using 20g of waste and 40 mL of reagent water.
21	Is the oily phase decanted and aqueous phase pH measured if the supernatant multiphasic? [SW-846 9045 D (7.3.2)]			If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase. The electrode may need to be cleaned if it becomes coated with an oily material.
22	Is the pH sensing portion and reference junction completely immersed? [SW-846 9045 D (7.3.3)]			Adjust the electrodes in the clamps of the electrode holder so that, upon lowering the electrodes into the beaker, the glass electrode will be immersed just deep enough into the clear supernatant solution to establish a good electrical contact through the ground-glass joint or the fiber-capillary hole. Insert the electrodes into the sample solution in this manner. For combination electrodes, immerse just below the suspension.
23	If the sample temperature differs by more than 2 °C from the buffer solution is the measured pH values corrected? [SW 846 9045 D (Section 7.3.4)]			Report the results as "waste pH measured in water at ___ °C" where "___ °C" is the temperature at which the test is conducted.
	QUALITY ASSURANCE	L A B	S O P	EXPLANATION
24	Is a check standard buffer analyzed after meter calibration, before sample analysis? [NC WW/GW LCB SW-846 pH Calibration Verification Policy]			The meter calibration must be verified with a third standard buffer solution (i.e., calibration check buffer) prior to sample analysis
25	What is the true value of the check buffer standard? Answer:			
26	Is the acceptance criterion for the check standard buffer ± 0.1 S.U. of true value? [NC WW/GW LCB SW-846 pH Calibration Verification Policy]			All calibration check standard buffers must read within ± 0.1 S.U. of its true value to be acceptable.
27	What corrective action is taken if the check buffer does not meet the acceptance criterion? [NC WW/GW LCB SW-846 pH Calibration Verification Policy] Answer:			Check again with a freshly poured buffer. If the buffer still does not meet the criterion, recalibrate the instrument.
28	Is a post-analysis check buffer analyzed at the end of the run any time the meter is transported by vehicle to another location after calibration? [NC WW/GW LCB SW-846 pH Calibration Verification Policy]			When performing analyses at multiple sample sites, a post-analysis calibration verification using the check standard buffer must be analyzed at the end of the run.
29	Is the acceptance criterion for the post-analysis check standard buffer ± 0.1 S.U. of true value? [NC WW/GW LCB SW-846 pH Calibration Verification Policy]			The post-analysis check standard buffer(s) must read within ± 0.1 S.U. of its true value or corrective actions must be taken.
30	What corrective action is taken if the post-analysis check buffer does not meet the acceptance criterion? [NC WW/GW LCB SW-846 pH Calibration Verification Policy] Answer:			If recalibration is necessary, all samples analyzed since the last acceptable calibration verification must be reanalyzed, if possible. If samples cannot be reanalyzed, the data must be qualified.
31	Are results qualified to indicate quality control failures or sample anomalies when reporting results? [15A NCAC 02H .0805 (e) (5)]			Reported data associated with Quality Control failures, improper sample collection, holding time exceedances, or improper preservation shall be qualified as such.

Additional Comments:

Inspector: _____ Date: _____