

# NCMA Annual WQ Workshop February 18, 2025 Michael Cumbus NC Wastewater/ Groundwater Certification Branch



### **Presentation Overview**

- How to get certified
- Maintaining Certification
  - Time-bound requirements
  - Policies (with a few examples that apply to all)
- Field Parameter requirements
- Updates on Certification Rules and EPA (PFAS, TN, Pharmaceutical Pollutants)



Department of Environmental Quality

### Initial Certification

- Complete Application (Field or Non-field as appropriate)
  - Obtain EPA lab code
  - Include the methods you are requesting
- Non-field labs will be invoiced a \$300 application fee
- Perform and submit Proficiency Testing Samples (where required)
- Submit Standard Operating Procedures (SOPs) for each certified method; some labs have separate Quality Assurance Manuals (QAM) for other laboratory processes outside of method specific
- Implement training program, document the training of associated analysts, submit the documented training
  - The training program elements must be included in SOPs or QAM



## Maintaining Certification

- Requirements for Maintaining Certification
  - PT samples our office must receive graded study from PT Vendor no later than September 30<sup>th</sup> or it will be considered an unacceptable result
  - Pay annual fee usually invoiced on July 1 and due September 1. However, annual fees are now set through the <u>NC State Budget(pg. 388)</u>, if budget is not approved by July 1 there could be a delay. Due 60 days after issued
  - SOP REVIEW every two years at minimum; REVISE in real time if there are changes to procedure
  - Perform and document training for any new analysts
  - Update our office with any Supervisor or Manager changes
  - <u>Policies</u> up to date Policies on our website, recommend joining listserv where we will notify when a policy updates or is generated
  - Go to <u>https://lists.deq.nc.gov/</u>, include your name, email address and choose "DWR.Lab\_Cert (Laboratory Certification Updates)" as the List to join.



### Recurring Time-bound Requirements for Laboratory Equipment

- Temperature-measuring devices in equipment such as sample refrigerators, residue ovens, incubators – See <u>Policy</u>
- Automatic pipettes field labs must check every 12 months; non-field labs must check every 6 months [15A NCAC 02H .0805 (a) (7) (O); 15A NCAC 02H .0805 (g) (10)]
- Analytical Balance professional maintenance every 12 months [15A NCAC 02H .0805 (a) (7) (J)]
- Weight calibration every 5 years [15A NCAC 02H .0805 (a) (7) (J)]

(not all-inclusive list)



### Sample Collection and Receipt

- Our Rules do not require a specific Chain of Custody (COC) form, however, the Rule and Policy require certain documentation, so it is an easy way to organize
- Basic documentation for <u>sample collectors/submitting laboratory</u>
  - 1. Facility identification (name or permit number);
  - 2. Sample collector (printed name or signature required);
  - 3. Date and time of each sample collection;
  - 4. The parameter and/or analytical method to be performed (as stated in the permit where applicable);
  - 5. Sample type (e.g., composite, grab, water, soil or sludge);
  - 6. Sample identification (effluent, influent, upstream, downstream, monitoring well, pretreatment, etc.);
  - 7. Sample (or temperature blank) temperature at time of collection when required (i.e., to show a downward trend in temperature if transport time is too short to reach required preservation temperature);
    8. Chemical and/or physical preservation/treatment(s) used where required (e.g., name of preservative, pH<2 S.U., pH>9 S.U., field filtration, TRC neutralization, etc.);
  - 9. Sample storage refrigerator temperature for each day samples are placed into or removed from the refrigerator. Required documentation includes the date, temperature and name or initials of the responsible party.



### Sample Collection and Receipt

- Documentation requirements for receiving lab
  - 1. Sample receiver (printed name and signature where required);
  - 2. Date and time of receipt;
  - 3. Preservation/treatment status where required (e.g., temperature, pH, absence of residual chlorine, etc.);
  - 4. Sample storage refrigerator temperature for each day samples are placed into or removed from the refrigerator. Required documentation includes the date, temperature and name or initials of the responsible party.

Field Lab Policy non-Field Lab Policy



### Traceability Documentation

- Rule [.0805 (a) (7) (K)/.0805 (g) (7)] and Policy
- Chemical containers must be dated when received and when opened
- All chemicals (reagents/standards) and consumables such as bacteriological filters, must be traceable from their origin to their use during a specific analysis
- Easiest way to adhere to traceability requirements is to maintain a receipt log and a standard/reagent prep log



#### Lab Supply Receipt Log

Standard	and	Reagent	Prep	Log
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Item Name	Vendor	Lot #	Date Received	Expiration Date	Date Opened/In Use	Lab ID	Prepared Solution Name
Ammonia 100 mg <b>/L sto</b> ck	ABC Supplies	01234	12/13/2024	12/31/2025	1/25/2025	01252025_ 01_NH3	Ammonia CCV
						01252025_ 02_NH3	Ammonia LLS
						8	
						8	
						I	1

Final Conc. Procedure Followed Date Expiration me CV Prepared Date 10 mg/L 1/25/2025 This column includes the volume or weight of 2/25/2025 standard(s) used, the solvent and final volume of the solution 1 mL of 100 mg/L Ammonia stock (Lot #01234), brought to volume with DI water in 100 mL volumetric flask. 1/25/2025 10 mL of 10 mg/L Ammonia CCV (01252025\_01\_NH3) 2/25/2025 LS 1 mg/L brought to volume with DI water in 100 mL volumetric flask.

Examples of a reagent receipt log and a preparation log for standards/reagents



### Training Program

- Rule references: non-Field 15A NCAC 02H .0805 (a) (7) (P);
   Field 15A NCAC 02H .0805 (g) (5)
- The Rule includes the minimum training program elements
- Each element needs to be expanded and specific to your lab
- Make sure that each element is documented as performed, and retained
- Even if there is currently only one operator/analyst at a lab, a program must be developed for ongoing and future adherence to Certification requirements



## Simple Example of Training Documentation

Paran	neter and Method:			
1.	Education, Training, or Experi	ence		
	List your specific requirements	for the employ	yee.	These requireme
	Employees Initials:	Date:		would also be in t
2.	Demonstration of Proficiency	DOP)		SOP/QAM
	List your specific requirements Proficiency Testing sample wi		nployee has successfully analyzed a sult of Acceptable.	
	Employees Initials:	Date:		
	each time the SOP is updated		SOP Revision Date:	
			SOP Revision Date:	
			<ul> <li>March 200 and 11 and 12 and 10 and  10 and 10</li></ul>	
	Employees Initials:	Date:	SOP Revision Date:	
	Employees Initials:	Date:	SOP Revision Date:	
	Employees Initials:	Date:	SOP Revision Date:	
(Train	ner) Print Name:		Date:	
(Train	ner) Signature :		Date:	
(Train	nee) Print Name :		Date:	
(Train	nee) Signature :		Date :	



### Field Parameter Requirements

**Approved Procedures** 

**SOP** Templates

Checklists

Temperature

**Dissolved Oxygen** 

Conductivity

рΗ

**Total Residual Chlorine** 

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### Field Laboratories

- <u>Approved Procedure Documents</u>
  - Requirements for the method and documentation requirements, you do not have to purchase the source material (i.e., Standard Methods)
- <u>SOPs</u> templates available for most but not all field parameter methods
  - Not required to use but it includes all necessary elements.
  - There is still work that must be done by your lab- customize
  - Reminder that you must review the SOP at least every 2 years and document the date
  - Any changes to your procedure must be updated real-time in the SOP document what changed and the date
- <u>Checklists</u> methods and 'walk-through'



### Temperature

- No PT
- Every 12 months, must check the temperature measuring device against a Reference thermometer at two temperatures that bracket the compliance sample temperature range
  - Must read within 0.5 °C of the Reference Thermometer at both points
  - Reference Thermometer must have an accuracy of 0.5 °C
- Analyze in stream or within 15 minutes
- Report Temperature in whole degrees



### рН

- Analyze in stream or within 15 minutes
- Calibration
  - Calibrate with at least 2 buffers
  - Analyze a calibration check buffer, must read within ±0.1 SU
  - If the meter is transported by vehicle after calibration, a post-analysis check buffer must be analyzed (within ±0.1 SU)
- Document in hundredths on benchsheet but report in tenths



### Total Residual Chlorine (TRC)

- If you have a permit limit, must use a meter that measures to that level. For example, must be able to display 17 μg/L, not 0.01 or 0.02 μg/L (10 or 20 μg/L), as this does not demonstrate compliance with a limit of 17 μg/L
- Calibration- most use the factory-set curves and those must be verified INITIALLY and every 12 months
  - 5 standard concentrations that bracket your permit limit up to Proficiency Testing Sample concentration
  - Must include a Method Blank (water used to make the standards with DPD powder added) and must read ≤ ½ the lowest standard concentration
  - Curve verification spreadsheet on our <u>website</u>
  - Reporting limit the lowest concentration that you verify in your annual curve verification is the reporting limit. If the meter displays a measured value less than that, report on DMR as < RL. For example, with a curve consisting of 15, 25, 50, 200, 400 µg/L standards, and your instrument reads 12 µg/L, you will report as <15 µg/L</li>



### Total Residual Chlorine

- Zero meter with blank gel-type standard or lab water without DPD
- Daily Check Standard
  - Most use a gel-type standard that comes in a pack, but only one standard that is within the calibration range needs to be measured
  - The gel standard must be assigned a value every 12 months by measuring 3 times and averaging (even initially, do not use the value that is printed on the box)
  - If multiple meters or curves are used, a value must be assigned to the standard for each meter/curve
  - Daily check standard must read within 90-110% of the assigned value
  - If the meter is transported by vehicle after daily calibration check, perform a postanalysis verification with same standard at the end of analysis; same acceptance criterion
- Analyze sample within 15 minutes of collection
  - Analysis time is when the DPD (color) reagent is added to the sample



## Dissolved Oxygen (DO)

- No PT
- Analyze in stream or within 15 minutes
- Calibration
  - Prior to analysis, calibrate the meter per manufacturer's instructions
  - · Document the temperature, barometric pressure OR altitude, and salinity
    - Note- salinity is often set at a default value of 0 ppt. This still needs to be documented on your benchsheet
  - When calibration is complete, return to measurement mode and document the DO value or percent saturation that the meter is reading inside the calibration chamber
  - When the meter is transported by vehicle after calibration, recalibrate at each site or perform a post-analysis calibration verification at the end of the day
  - Post-analysis calibration verification compares the reading on the meter when probe placed back in the calibration chamber to a calculated theoretical value (See the Approved Procedure)
    - The values must agree within ± 0.5 mg/L



### Conductivity

- Calibrate with one standard and analyze a daily check standard at another concentration
- Do not dilute conductivity samples or standards, they do not have a linear relationship when diluting
- Every 12 months, the Automatic Temperature Compensation must be checked
  - Check at exactly 25 °C and another temperature that brackets the low range of the samples encountered during the year. If sample temperatures sometimes reach above 25 °C, check at a third higher temperature.
  - Verify the acceptance criteria are met (See the Approved Procedure)



### Certification Rule Updates

Certification Rules determine what Parameters can be Certified by the program

- Effective 2024, PFAS was added as a Certifiable Parameter in 15A NCAC 02H .0804
  - Note: Even though we can certify the Parameter, specific Parameter Methods must still be found in the sources referenced in 15A NCAC 02H .0805. (e.g.; 40 CFR Part 136)
  - EPA 1633A cannot be certified until it is published in 40 CFR Part 136.
  - The PROPOSED <u>Method Update Rule 22</u> was printed in the federal register on January 21, 2025 and this includes adding EPA 1633A to Part 136
  - 30-day comment period ends February 20, 2025
  - Complete timing unknown, likely to be a delay due to executive order announced after proposal
- We are currently in the process of revising .0804 again to add Total Nitrogen and Pharmaceutical Pollutants (found in Table IF 40 CFR Part 136.3)
  - Multiple steps in the approval process; first meeting with Water Quality Committee, March 12
  - If all runs on time, we should go before the EMC in September or November for adoption



# Thank You!



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