Ohio EPA Drinking Water Laboratory Certification Updates and Tips

OTCO Compliance Workshop

October 9, 2024

Ohio Environmental Protection Agency

OVERVIEW

- Program Changes
- Lab Replacement/Remodeling Requirements
- Invoicing and Fee Payments
- 5-Year Rule Review
- 2025 Manual Edits
- Survey Tips
- HABs/qPCR Certification
- PFAS Certification

PROGRAM CHANGES

- Data request prior to survey
- 100% Electronic
- Remote surveys for IAs, HAB/qPCR
- Staffing

LAB REPLACEMENT/REMODELING REQUIREMENTS

- New labs, remodeled labs, and temporary labs
- See Chapter 2 of the Micro and Chem manuals.
- Contact Lab Certification prior to construction
- Plan approval **and** Construction approval
- Methods needed?

  -Check plan approval or other information provided by DDAGW.

INVOICING AND FEE PAYMENTS

- Ensure payments are made by the deadline
- Reminder emails
- Electronic payments

FIVE-YEAR RULE REVIEW

- Updating references
- Updating reporting limit for Microcystin
- Adding reporting limits for regulated PFAS compounds
- Rule Package Status:
  - -Early stakeholder outreach completed August 11, 2024
  - -Internal review completed September 30, 2024
- Ensure you are signed up for Ohio EPA's electronic mailing list
  - -https://public.govdelivery.com/accounts/OHEPA/subscriber/new

2025 MICRO MANUAL EDITS

a. Reagent Grade Water: Only satisfactory reagent water from deionization units may be used to prepare media, reagents and dilution/rinse water for performing microbial analyses.

1. If a resistivity indicator light is used, sensitivity of the light must be set at 0.5m. > 0.5 megohms resistance or < 2 micromhos/cm.

Prior to use, the quality of the reagent water should be tested and meet the criteria as listed in Table 1.

Table 1: Required Reagent Grade Water Criteria

Parameter	Limits	Frequency[1]
Conductivity	> 0.5 megohms resistance or < 2 micromhos/cm (microsiemens/cm) at 25°C	Monthly[2,3]
Total Chlorine Residual	< 0.1 mg/L	Monthly[2,3]
Pb, Cd, Cr, Cu, Ni, Zn	Per Contaminant < 0.05 mg/L Collectively < 0.1 mg/L	Annually[4]

[1] If the laboratory purchases bottled reagent grade water, Table 1 does not apply.

[2] If the meter has a resistivity indicator light (i.e., green/red light), record color of light on Microbiological Laboratory Reagent Grade Water Record. The water should only be used if the light is green at the time of use. No conductivity or chlorine residuals are necessary.

[3] If no resistivity light, must analyze with each new batch of reagent water.

[4] Must be analyzed by an Ohio EPA Drinking Water certified or accepted laboratory.

Microbiological Laboratory Reagent Grade Water Record

See Table 1 for criteria.

Laboratory _____ Date of Annual Trace Metals Analysis _____

Analyst	Date	Resistivity Indicator Light (Green or Red)	Conductivity (microsiemens/cm)	Total Chlorine Residual (mg/L)	Comments/Corrective Actions Taken

2025 MICRO MANUAL EDITS

MMO-MUG Reagent Quality Control Record

To be recorded for each new lot or annually. If performed by a different laboratory, results must be retained.

Laboratory _____

Analyst	Reagent Type	Brand/Lot Number	Incubation Start Date/Time	Incubation End Date/Time	Test Results					
					E. coli		*Klebsiella/Variicola*		*Pseudomonas*	
					Color Change	UV	Color Change	UV	Color Change	UV

Sample Bottle Sterility/Fluorescence Record

To be recorded for each lot received or batch sterilized

Laboratory _____

Analyst	Date Received/ Sterilized	Brand/Lot Number	Number of Bottles Received/ Sterilized	Growth Incubation Start Date/Time	Growth Incubation End Date/Time	Growth Results Interpretation		UV Results		Comments
						Number Positive	Number Negative	Number Positive	Number Negative	

Media Quality Control Record

To be checked and recorded for each new prepared batch and annually

Laboratory _____

Analyst	Media	Brand/Lot Number	pH	Growth Incubation Start Date/Time	Growth Incubation End Date/Time	Growth Results Interpretation		Comments
						Positive	Negative	

2025 CHEMISTRY MANUAL EDITS

Monthly Fluoride QC Sample Record

Laboratory_____

Analyst	Date	Results (mg/L)	Certified Value Range per PT Provider (mg/L)	Results Within Range (Y/N)	QC Sample Provider Name	Sample Lot #

2025 CHEMISTRY MANUAL EDITS

Monthly Alkalinity Titrant Standardization Record

Laboratory_____

Standard Concentration_____

Analyst	Date	Reagent Water Volume (mL)	Blank Verification Result * (mL/drops)	Standard Volume (mL)	Titration #1	Titration #2	Titrant Lot Number/Date Prepared	Corrective Action Taken If Out of Range

*Blank verification must be <0.2 mL or 4 drops.

2025 CHEMISTRY EDITS

Inorganic Analytical Methods

Analysis of inorganic constituents in drinking water must be performed following Ohio EPA accepted analytical methods referenced in rule 3745-81-27(A) of the OAC. Unless otherwise specified below, quality control (QC) acceptance limits listed in the individual method must be followed. In addition to individual method's QC requirements, the Laboratory Certification Section requires that, **at minimum**, the following program specific inorganic analysis QC be met. See Chapter 2, Section B of this manual for Quality Assurance Plan requirements.

- Laboratory analyte reporting limits must meet reporting limit concentrations referenced in the appendix to rule 3745-89-03 of the OAC.
- An Initial Demonstration of Capability (IDC) study, (i.e., a blank and 4 LFBs), must be completed and documented for each analyst certified for drinking water method analysis.
- For methods not included in this manual, certified analysts must generate a curve at least once annually for all analytical methods which they are certified.
- Curve generation is limited to 1st or 2nd order. Calibration curves must result in a Correlation Coefficient (R) greater than 0.995 or a Coefficient of Determination (R^2) greater than 0.990 to be acceptable for drinking water analysis. It is recommended that curves not be forced through zero. (Calibration curves must be at least 3 standards and a blank, unless otherwise specified in the method.)
- Any concentrations above the highest standard in the calibration curve must be diluted to fall within the calibration range.
- At least once every three months, a drinking water sample must be analyzed using the inorganic analytical methods for which the laboratory is certified.
- An annual Method Detection Limit (MDL) study must be performed using the most recent version of USEPA's "Definition and Procedure for the Determination of the Method Detection Limit" in accordance with the 40 Code of Federal Regulations (C.F.R.).
- A Reporting Limit Verification (RLV) sample must be analyzed with each analytical run. The RLV concentration is equal to the reporting limit concentration for each analyte of interest. If there is no regulatory reporting limit, use the lowest calibration concentration point as the RLV. The acceptance

- Initial Demonstration of Capability study must be documented for each analyst certified for drinking water methods (for methods not included in the manual).
- Method Detection Limit study must be performed annually for each lab.

INITIAL DEMONSTRATION OF CAPABILITY (IDC)

- Used to determine the analyst's ability to perform the method with acceptable precision and recovery
- Method specific
- Often is a variation of a blank and 4 LFBs within a recovery of ±10%
- Ensure all lab SOPs are updated to reflect this requirement

METHOD DETECTION LIMITS (MDLS)

EPA 821-R-16-006 – Definition and Procedure for the Determination of the Method Detection Limit, Revision 2, December 2016

- Applies to all drinking water MDLs **except HABs and Hach TNT methods**
- Ensure all lab standard operating procedures are updated to reflect this revision.

Annual MDLs should be submitted to dwlabcert@epa.ohio.gov for review.

METHOD DETECTION LIMITS (MDLS)

- Initial MDL: at least seven spikes/blank prepared and analyzed on three different days
- Once an initial MDL is established, the MDL is re-calculated annually including all spike/blank values over the last 24 months
- If seven points are not achieved over a 24-month period, a new initial MDL must be established
- The correct Student-t value must be used corresponding to the number of spikes in calculation (n-1)

METHOD DETECTION LIMITS (MDLS)

MDL Procedure: epa.gov/sites/default/files/2016-12/documents/mdl-procedure_rev2_12-13-2016.pdf

MDL Frequently Asked Questions: epa.gov/cwa-methods/method-detection-limit-frequent-questions

Expanded Student t Value Table: itl.nist.gov/div898/handbook/eda/section3/eda3672.htm

2025 CHEMISTRY MANUAL ADDITIONS

- Fluoride by SPADNS 2 (Arsenic-Free) Method 10225
- Nitrate by Hach TNT plus 835/836 Method 10206
- Orthophosphate by Hach Method 8048 (EPA 365.1)

Fluoride Analysis by SPADNS 2 (Arsenic-Free) Method 10225

Quick Reference	**Standard/Reagent/Equipment**	**Requirements**
Standard/Reagent Storage	SPADNS 2 Reagent	Manufacturer's Recommendations
	0.5/1.0/1.5 mg/L Standards	Manufacturer's Recommendations
	100 mg/L Stock Standard	Manufacturer's Recommendations
Standard/Reagent Expiration	**Standard/Reagent**	**Expiration**
	SPADNS 2 Reagent	1 Year After Opening/ Manufacturer's Expiration Date
	0.5/1.0/1.5 mg/L Standards	1 Year After Opening/ Manufacturer's Expiration Date
	100 mg/L Stock Standard	1 Year After Opening/ Manufacturer's Expiration Date
Required Quality Control	**QC Procedure**	**Frequency**
	Meter Calibration Verification	Once Every Three Months
	Blank, QCS	Once Per Batch
	QC Sample Analysis	Once Per Month
Sample Collection	**Preservation**	**Maximum Hold Time**
	None	48 Hours [See OAC rule 3745-83-01(F)(4)(b)] **or** 1 Month [See OAC rule 3745-81-23(J)]

INITIAL DEMONSTRATION OF CAPABILITY FOR ADDED METHODS

Initial Method Detection Limit (MDL) study
Initial Precision and Recovery (IPR) study

Method Detection Limit (MDL) Study

*To be performed in a single run

Laboratory:		**Analyst:**	
Instrument:			
			(number) (unit)
Date:		**True Value:**	

Replicate	Value (mg/L)
1	
2	
3	
4	
5	
6	
7	
Average	#DIV/0!
Std Deviation	#DIV/0!
MDL Result	#DIV/0!
MDL Acceptable	**#DIV/0!**

Initial Precision and Recovery Study

*To be performed in a single run

Laboratory:		**Analyst:**	
Insrument:			
			(number) (unit)
Date:		**True Value:**	

Replicate	Value (mg/L)	% Recovery
1		#DIV/0!
2		#DIV/0!
3		#DIV/0!
4		#DIV/0!
Average	#DIV/0!	#DIV/0!
Std Deviation	#DIV/0!	
%RSD	#DIV/0!	

Accuracy (% Recovery) Passing?	**#DIV/0!**
Precision (%RSD) Passing?	**#DIV/0!**

SURVEY TIPS - MICRO

- Reagent water quality (indicator light) - verify prior to use
- Incubator temperatures must be recorded on weekends if samples are being incubated
- Autoclave timer must be checked only at times used (e.g., 15, 30, 45); use proper procedure
- Balance verification must be done prior to use
- Sampling instructions for micro samples requires analyzing for chlorine residual after disinfection of sample tap

MMO-MUG Analysis for Total Coliform and *E. coli* by Colilert and Colisure

Quick Reference	**Standard/Reagent/Equipment**	**Requirements**
Standard/Reagent/Equipment Storage	MMO-MUG Reagent	Colilert – Dark Environment and Manufacturer's Recommendations Colisure – Refrigerated and Manufacturer's Recommendations
	Chemical Reagents	Manufacturer's Recommendations
	Dehydrated Media	Manufacturer's Recommendations
	Media Performance Check Cultures	Manufacturer's Storage Requirements
	Prepared Media	Refrigerated/Room Temperature
	pH Electrodes	pH 7 Buffer/Manufacturer's Storage Solution
	pH Buffers	Room Temperature
Standard/Reagent Expiration	**Standard/Reagent**	**Maximum Storage Time**
	MMO-MUG Reagent	Manufacturer's Expiration Date
	Chemical Reagents	Manufacturer's Expiration Date
	Dehydrated Media	6 Months After Opening **or** 1 Year After Opening if Stored in Desiccator
	10% Sodium Thiosulfate	1 Year After Preparation/ Manufacturer's Expiration Date
	Media Performance Check Cultures	Manufacturer's Expiration Date
	Prepared Media	3 Months Refrigerated (screw-capped tubes/flasks/vessels) **or** 1 Week Room Temperature (sealed/covered)
	pH Buffers	6 Months After Opening/ Manufacturer's Expiration Date
Required Quality Control	**QC Procedure**	**Frequency**
	Total Coliform/*E. coli* positive	Once Per Month Per Analyst
	Sample/Test Bottle Sterility Check	One Per Batch Prepared **or** 1% Per Lot Received *(maximum of 4 per lot)*
	Sample/Test Bottle Fluorescence Check	Every Sample/Test Bottle Prepared **or** 1% Per Lot Received *(maximum of 4 per lot)*
	Media Performance Check	Once Per Batch
	MMO-MUG Reagent Check	Once Per Lot and Annually
	Glass/Electronic Thermometer/ Data Logger Calibration	Annually
	Dial Thermometer Calibration	Once Every Three Months
	Equipment Timers	Once Every Three Months
	pH Meter Calibration	Prior to Use
	pH Linearity/Slope/pH 4 Buffer	Prior to Use
	Balance Calibration Check	Prior to Use
	Refrigerator Record	Daily
	Incubator Record	Twice Daily
Sample Collection	**Preservation**	**Maximum Holding Time**
	10% Sodium Thiosulfate	30 Hours

SURVEY TIPS - MICRO

Autoclave Sterility Check

- Required once every three months, per autoclave
- May use biological indicator ampules following manufacturer's instructions
- May use TSB or BHI, inoculated with a known coliform culture
- Ensure recorded on Autoclave Sterilization Record

Thermometer Calibration Record

- Must first include the NIST thermometer's temperature at ice point
- Recommend including each thermometers serial number
- MRTs are not calibrated with NIST
- Autoclave Dial (Display) Thermometers are not required to be calibrated unless fast exhaust is used.

SURVEY TIPS - MICRO

Media Preparation(e.g., TSB, BHI)

- Balance Calibration Record
- pH Meter Slope/Linearity Verification
- Media Quality Control Record
- Autoclave Sterilization Record
  - TSB or BHI at temperature 12-15 min
  - Autoclave door must be opened no later than 45 min after closing

Pre-Made Purchased TSB

- Use manufacturer's expiration date prior to opening.
- Keep all paperwork.

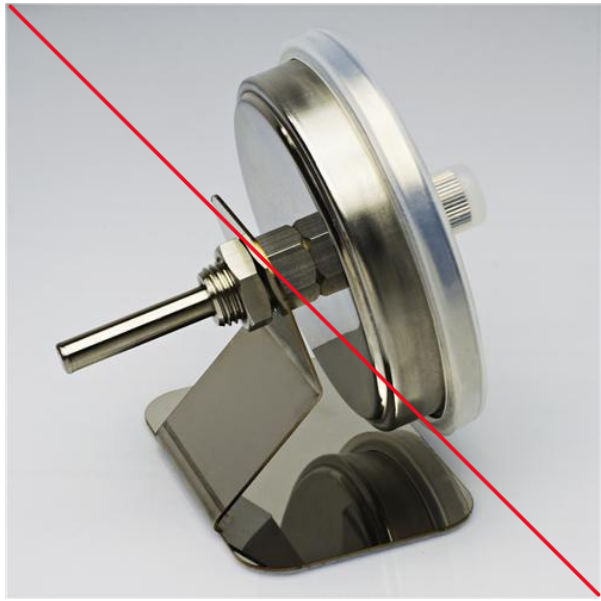
Microbiological Test Data Sheets

- All data from our bench sheets must be recorded to avoid invalidation of sample results.

SURVEY TIPS - MICRO

Maximum Registering Thermometers (MRTs)

- Calibrated by Lab Certification staff at the renewal survey
- Ohio Revised Code 3734.63, Sale of mercury-containing thermometer for promotional purposes.
    - If required to comply with federal law, these can be sold and distributed.
- Dial autoclave thermometers are not permitted.



SURVEY TIPS - CHEMISTRY

- QC requirements on first page of each method in the manual
- Never pipette directly out of a standard bottle
- Stability by saturation is to be filtered using a fine porosity fast-flow glass fiber filter paper
- Dry secondary chlorine standards with lint-free wipes
- Verification of alkalinity endpoint by pH 4.5
- Hach TU 5200 has a different Method Number

You may NOT perform analysis unless IA is granted, or a survey is successfully completed.

Alkalinity Analysis by Sulfuric Acid Titration Method

Quick Reference	**Standard/Reagent**	**Requirements**
Standard/Reagent Storage	0.020 N Sulfuric Acid (H_2SO_4)	Manufacturer's Recommendations
	Indicator (Bromcresol Green/ Methyl Red)	Manufacturer's Recommendations
	Sodium Thiosulfate	Manufacturer's Recommendations
	0.020 N Sodium Carbonate (Na_2CO_3) Standard	Manufacturer's Recommendations
Standard/Reagent Expiration	**Standard/Reagent**	**Expiration**
	0.020 N Sulfuric Acid (H_2SO_4)	1 Year After Opening/ Manufacturer's Expiration Date
	Indicator (Bromcresol Green/ Methyl Red)	1 Year After Opening/ Manufacturer's Expiration Date
	Sodium Thiosulfate	1 Year After Opening/ Manufacturer's Expiration Date
	0.020 N Sodium Carbonate (Na_2CO_3) Standard	1 Year After Opening/ Manufacturer's Expiration Date
Required Quality Control	**QC Procedure**	**Frequency**
	Standardize Titrant	Once Per Month
	pH 4.5 Endpoint Verification	Once Per Month
Sample Collection	**Preservation**	**Maximum Hold Time**
	4°C	14 Days

Method Reference

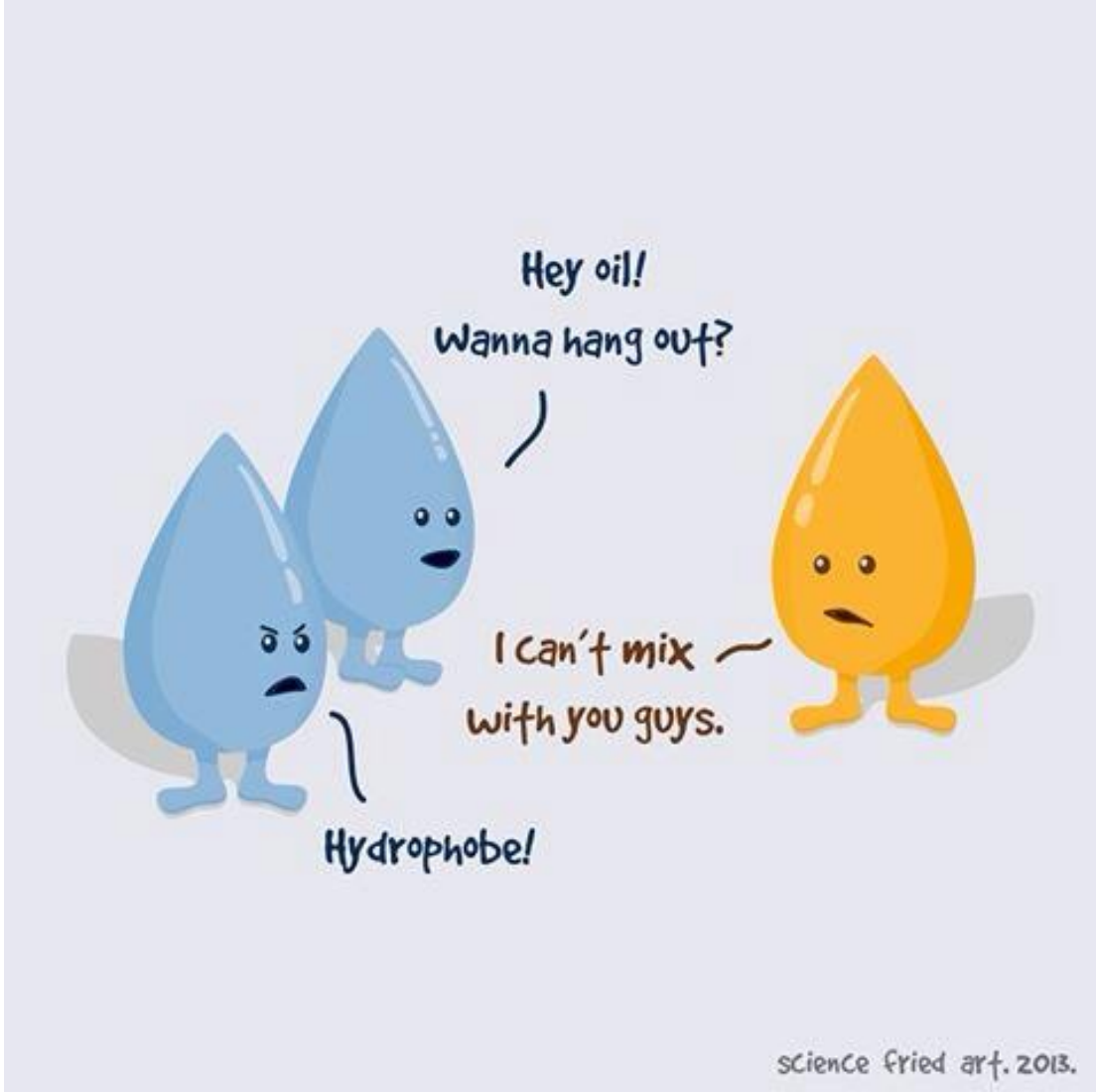
Standard Methods 22nd Edition (2320)

On-Site Survey Requirements

- Each certified analyst must be able to perform the alkalinity titrant standardization described in Section 7.0 of this method.
- Operationally certified analysts will be required to analyze a plant tap sample and may be required to analyze a performance sample.
- Procedural technique will be observed.
- All reagents, standards and solutions used for this method will be audited for correct labeling and dating.
- All records will be audited.

SURVEY TIPS - GENERAL

- Update bench sheets to most recent version available.
- Ensure all laboratory records are recorded **<u>using ink</u>** and are printed legibly.
- Scribbling/ writing over is unacceptable.
- Errors? Cross out with 1 line, initial, add correct information. **No White Out!!**
- **Include results to the 10th**
  (e.g., 121 is 121.0)
- Avoid eating or drinking in the lab.
- **<u>Annual review of manual(s) - REQUIRED</u>**



SURVEY TIPS- GENERAL

Annual Laboratory Manual Review Record

Laboratory _____ Methods Reviewed _____

Analyst No.	Analyst Signature	Date of Review	Analyst No.	Analyst Signature	Date of Review

CYANOTOXIN & CYANOBACTERIA CERTIFICATION

- Annual HAB MDLs and biannual qPCR curves as well as associated test data are to be sent to the dwlabcert@epa.ohio.gov email.
- Adding new analysts between renewal periods:

  -HABs (MDL study, associated test data and calibration curve)

  -qPCR (application, calibration curve, associated data and sample results), survey
- SOPs for microcystin and qPCR are available on our Lab Certification website.
- Review MDLs and curves prior to submitting; don’t send if clearly failed.

PFAS CERTIFICATION

- We have begun certifying laboratories for EPA Methods 533 and/or 537.1, rev 2.0.

  -Applications are posted.
- Reporting limit for each of the six regulated compounds will be 2.0 ppt
- The six regulated compounds are PFOA, PFOS, GenX, PFBS, PFHxS, PFNA.
- Lab Certification has issued acceptance for 16 laboratories to analyze for PFAS.

Thank You

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