Ohio EPA
Laboratory Certification Update
2018

Laboratory Certification Update

- Chlorine kit calibration verification
- Hach TU 5200 Calibration and use
- Iron & Manganese test methods
- Autoclave Sterility Check
- Personnel Changes
- Cyanotoxin & Cyanobacteria Program update
- 2019 Laboratory Manual update

Chlorine kit calibration verification

- Acceptable methods of verification
- $KMnO_4$
- Free Chlorine Ampules
- Manufacturer produced Secondary Standards
- Frequency Requirement is same
  - At least once every 3 months

Chlorine kit calibration verification

Ohio Environmental Protection Agency

Division of Environmental Services
Laboratory Certification Section
September 2016

Calibration Verification of Chlorine Meters (an alternate procedure)

The calibration verification procedure required for chlorine meters has been one of the more difficult and technique dependent procedures performed in many of Ohio's certified laboratories. Most of this difficulty is due to the use of a micro-pipette to create a series of chlorine standards. The calibration verification is needed to ensure meters are determining the chlorine concentration properly. The procedure is not, however, intended to test the analysts' ability to use a micro-pipette. The procedure outlined in the "Ohio EPA Laboratory Manual for Chemical Analyses of Public Drinking Water 2014" is a verification of the meter's calibration; not a calibration of the meter itself. Since laboratories are not establishing the meter's calibration, the Ohio EPA's Laboratory Certification Section is now permitting an alternate procedure to verify the meter is reading within acceptable limits. The use of secondary standards for calibration verification is an acceptable alternative to the current method of making up various standards and reading them in the chlorine meters.

What are Secondary Standards?

At this time only a few chlorine kit manufacturers provide secondary standards (i.e., Hach and LaMotte). Typically, the secondary standard sets contain four vials (a blank and three standards) filled with gel or liquid that simulates the chlorine at various concentrations.

Each set of standards is specific to the manufacturer and the meter's analytical range; low-range (LR), mid-range (MR) and high-range (HR). Verification must be performed per manufacturer's instructions. Secondary standards are good until the manufacturer's expiration date and must be stored per manufacturer's instructions.

Calibration Verification Requirements for Secondary Standards

Meters must be verified for all tested chlorine ranges. If chlorine concentrations are observed greater than the limits of the LR setting, then a meter with a MR setting or the meter in the HR setting must be verified using the appropriate secondary standards. However, if chlorine concentrations do not exceed the limits of the meter's LR setting (2.20 mg/L (Hach); 4.00mg/L (LaMotte)), then only the LR setting must be verified.

1. All chlorine meters must be checked at least once every three months.
2. Observed readings must be within 10% of the manufacturer's assigned value. If the observed readings are outside the acceptable range, the meter must be serviced or replaced.
3. A minimum of three secondary standards must be verified.
4. Verification must be recorded and records must be maintained with the laboratory's QC records.
5. The blank standard provided with a set of standards, must only be used with the same set of standards (i.e., LR blank with LR standards of the same lot number).
6. All analytical ranges used for reporting chlorine concentrations must be verified (i.e., LR|setting with LR standards, MR setting with MR standards and HR setting with HR standards).

Calibration Verification of Chlorine Meters (an alternate procedure)

Breakdown of Calibration Verification Requirements

	Verification Using Current Lab Cert Manual	Verification Using Secondary Standards
Verification Frequency	Once Every Three Months	Once Every Three Months
Range of Acceptance	Prepared standards must be within 10% of calculated value	Meter readings must be within 10% of the standard's certified value
Records	Recorded on calibration verification record and stored with lab QC records	Recorded on calibration verification record and stored with lab QC records
Standard Storage Requirement	Chlorine free ampules and prepared potassium permanganate stored in refrigerator or per manufacturer's recommendations	Secondary standards stored per manufacturer requirements, in original box
Standard Maximum Storage Time	Chlorine free ampules: manufacturer's expiration date Or Potassium permanganate: 1 year after opening or manufacturer's expiration date, whichever occurs first	Manufacturer's expiration date
DI Blank Check	Prior to calibration verification with a total chlorine reagent	None, but the meter must be zeroed with the blank standard provided with the secondary standard kit

Daily Use Requirements for a Meter Verified by Secondary Standards

Samples must be analyzed using a verified meter in the appropriate setting (LR, MR or HR). During sample analysis, meters must be zeroed with the sample to be tested, without DPD reagent added. A meter cannot be zeroed with the blank standard from the secondary standard kit.

Timeframe for Implementation

Laboratories may continue to use the verification procedure described in the "Ohio EPA Laboratory Manual for Chemical Analyses of Public Drinking Water 2014". However, effective immediately, secondary standards may be used as an alternative to this procedure. Laboratories must use the correct bench sheet for the appropriate verification procedure. **Click Here** for a copy of the required bench sheets.

Contact

If any questions, please contact a member of the Ohio EPA's Laboratory Certification Section or email DWLabCert@epa.ohio.gov. Contact information can be found at the following link: Laboratory Certification Contacts.

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Hach TU 5200 Calibration and use

Ohio Environmental Protection Agency

Division of Environmental Services
Laboratory Certification Section
September 2017

Hach TU 5200 Meter

Turbidity Analysis by 360° Nephelometry

Quick Reference	Standard/Reagent	Requirements
Standard/Reagent Storage	Formazin 4000 NTU	Refrigerated
	AMCO Standards	Room Temperature
	StablCal Standards	Room Temperature
	Secondary Standards	Room Temperature
Standard/Reagent Expiration	**Standard/Reagent**	**Maximum Storage Time**
	Formazin 4000 NTU	1 Year After Opening/Manufacturer's Expiration Date
	AMCO Standards	1 Year After Opening/Manufacturer's Expiration Date
	StablCal Standards	1 Year After Opening/Manufacturer's Expiration Date
	Secondary Standards	Manufacturer's Expiration Date
	Diluted formazin	Discard after use
Required Quality Control	**QC Procedure**	**Frequency**
	Record Secondary Standard Verification	Once Per Shift
	Turbidimeter Calibration	Once Per Three Months
	Secondary Standard Value Assignment	Once Per Three Months, Following Calibration Procedure
Sample Collection	**Preservation**	**Maximum Hold Time**
	4°C	48 Hours

Method Reference

Hach Method 10258

Quarterly Turbidimeter Calibration Record

Date _____ Analyst(s)_____

	Result (NTU)
LTW Result (NTU)	
1.0 Result (NTU)	
1.0 NTU Acceptance Range (0.9 to 1.1)	Circle One: **Yes** or **No**
Corrected 1.0 NTU (1.0 NTU minus LTW NTU)[1]	

[1]Not applicable for AMCO Clear or Hach StablCal

Secondary Standard Assigned Values

Secondary Standards	0 – 2.0 (NTU)	0 – 20 (NTU)
Assigned Value		
Acceptance Range (± 10%)	To	To

Quarterly Turbidimeter Calibration Record

Date _____ Analyst(s)_____

	Result (NTU)
LTW Result (NTU)	
1.0 Result (NTU)	
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Secondary Standard Assigned Values

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Acceptance Range (±10%)	To	To

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Hach TU 5200 Calibration and use

- Method number is different
- Certificates must be updated
- Follow manufacturers calibration
- Vials Must be stored with Laboratory pure Water
- Do Not set Vials on counter
- Bottom of vial Must be Clean
- First reading is not always reliable
- Stable-Cal must be used for 1.0 NTU daily verification

Iron & Manganese test methods

- Approved Methods for Manganese
  - 200.5 ICP-AES
  - 200.7 ICP-AES
  - 200.8 ICP-MS
  - 200.9 Furnace AA
  - SM 3111B Flame AA
  - SM 3113 Furnace AA
  - SM 3120 ICP-AES

Iron & Manganese test methods

- Recommended Approach
- OAC 3745-83-01
  - (6) Manganese.

A community public water system that provides treatment to reduce manganese shall monitor for manganese at least at each entry point to the distribution system.

(a) Community systems serving up to and including two hundred and fifty persons shall monitor either of the following:

(i) Weekly with an in-house test kit in accordance with paragraph (F)(6)(c) of this rule and one split sample monthly by a certified laboratory.

(ii) Weekly by a state certified laboratory.

(b) Community systems serving greater than two hundred fifty persons shall monitor either of the following:

(i) A minimum of five days per week with an in-house test kit in accordance with paragraph (F)(6)(c) of this rule and one split sample monthly by a certified laboratory.

(ii) Weekly by a state certified laboratory.

(c) A manganese test kit shall have a minimum detection level of 0.02 milligrams per liter. Furthermore, the deviation of the split sample shall be no greater than 0.04 milligrams per liter. If it is greater, then the public water system shall cease monitoring with the test kit and substitute with weekly monitoring at a state certified laboratory. A public water system may resume monitoring with their test kit once a deviation of the split sample is no greater than 0.04 milligrams per liter. The director may accept an alternate collection frequency and deviation from in-house test kits for split sampling requirements.

Autoclave Sterility Check

- Autoclave sterility checks are required **once every three months**, per autoclave.
- • If using a biological indicator ampule, follow manufacturer's instructions. **Note:** After sterilization, remove and allow ampules to cool for 10 minutes prior to incubation. Incubate at 55 - 60°C for 24 hours. Growth is evident by a color change per manufacturer's instructions. If color change occurs, corrective action for the autoclave is required.
- • Alternatively, fill an Erlenmeyer flask with 25 to 50 mL of TSB/BHI, inoculate with a known coliform culture, cover flask opening with aluminum foil and incubate at 35 ± 0.5°C for 24 hours. After incubation, when TSB/BHI shows growth, autoclave at 119 - 121°C for 12 to 15 minutes on slow exhaust. Allow to cool to room temperature. Fill a test vessel with approximately 25 mL of TSB/BHI and inoculate the TSB/BHI with the "sterilized" culture from the Erlenmeyer flask. Incubate test vessel at 35 ± 0.5°C for 24 hours. After the 24 hour incubation period, remove the test vessel from the incubator. The inoculated test vessel must not show growth. If growth is present in the inoculated test vessel, corrective action for the autoclave is required.

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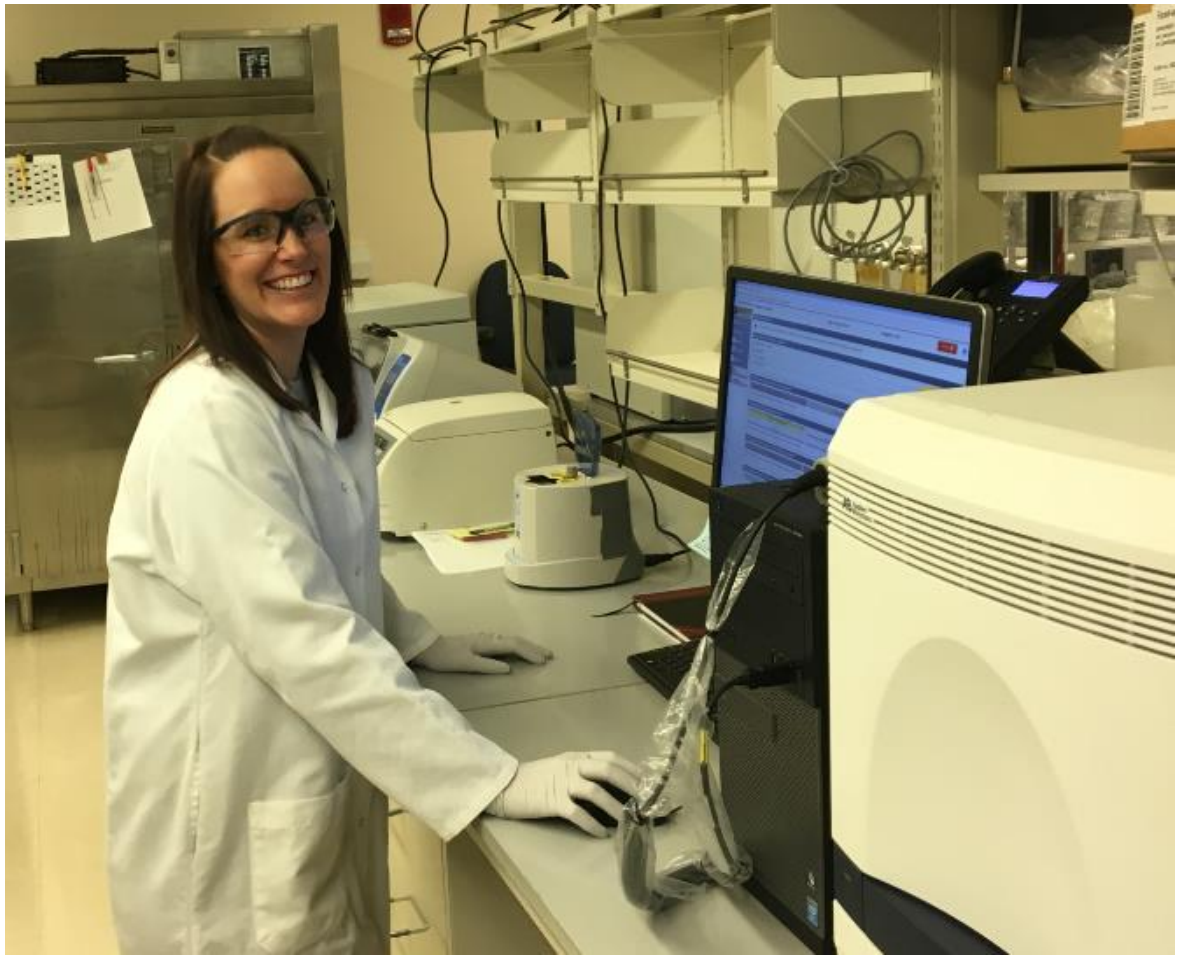
Pop Quiz

- When must Laboratories begin to meet the new reporting requirements?

- October 1, 2018

Personnel Changes

Personnel Changes



Cyanotoxin & Cyanobacteria Program update

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Cyanotoxin & Cyanobacteria Program update

- Surveys are currently being scheduled
  - Most surveys will be scheduled on Mondays
  - Certification will be extended as long as application is received by 5/31/2018
- Qualifier Records
  - Record qualifiers on laboratory records if used when reported
- Pipette Verification
  - Acceptance criteria is 5.0%

Cyanotoxin & Cyanobacteria Program update

- New Qualifier

  We have added a new qualifier to our Quality Control section of our Ohio EPA Total (Extracellular and Intracellular) Microcystins-ADDA by ELISA Analytical Methodology standard operating procedure 701.0. The following is a description of the new qualifier (UJ) and how it is to be used.

- UJ The analyte was not detected above the sample quantitation limit (QL). However, the reported QL is estimated.

- This qualifier is applied only if the sample result is below the reporting limit and any of the following conditions apply:
  - sample is collected in improper sample container
  - received warm (>4^{o}C)
  - low LCRC recovery

- This qualifier is to be used **effective immediately.**

Pop Quiz

- New Requirement is
- a requirement to complete analysis, including quality control, no later than ????? business days after receipt of samples

2019 Laboratory Manual update

Pop Quiz

- True or False

- results are required to be reported to Ohio EPA and to public water systems by the tenth day following the date the analyses are completed.

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