



**Collecting Surface Water Samples for Cyanobacteria
and Cyanotoxin Analysis**

S-003-OWQ-WAP-TGM-23-T-R1

Technical Standard Operating Procedure (TSOP)

Office: Water Quality

Branch: Watershed Assessment and Planning

Section: Targeted Monitoring

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Purpose

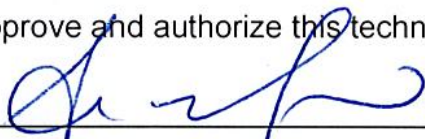
This technical standard operating procedure (TSOP) outlines the process for collecting surface water samples for cyanobacteria and cyanotoxin analysis.

Scope

Office of Water Quality staff collecting surface water samples for cyanobacteria and cyanotoxin analysis.


Authorizing Signatures

I approve and authorize this technical standard operating procedure:




Ali Meils, Section Chief
Targeted Monitoring Section, Watershed Assessment and Planning Branch

2/21/23
Date



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Kristen Arnold, Branch Chief
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Date

This technical standard operating procedure is consistent with agency requirements.



Quality Assurance Staff
Office of Program Support

2/23/23
Date

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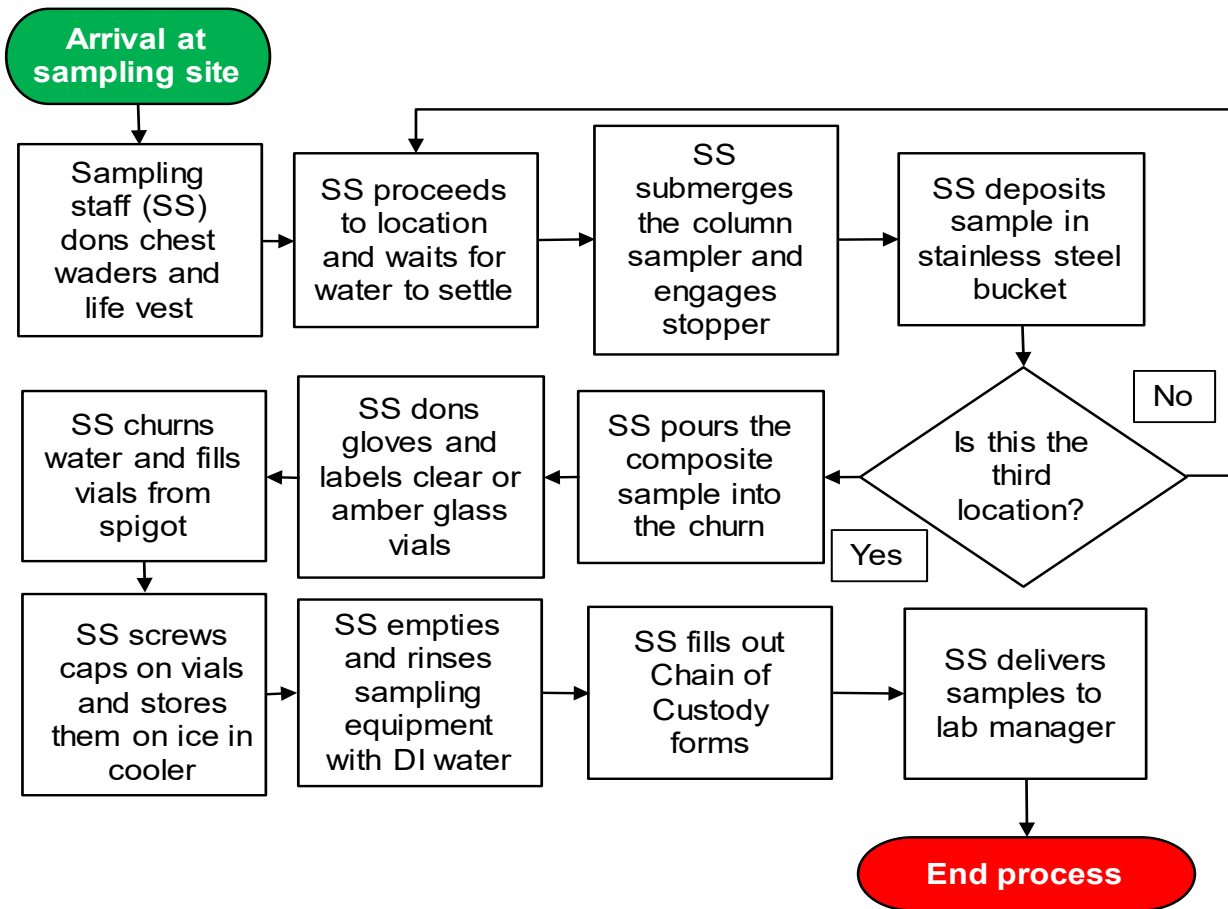
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1.0. Overview Flowchart

Not Applicable (N/A)

2.0. Procedure

2.1. Procedural Flowchart



2.2. Procedural Steps

Note: At each beach swimming area, one composite sample will be collected from three locations, one from each side and one from the middle portion. At the Fort Harrison Dog Park Lake, grab samples are collected near the shore where dogs are most likely to contact and consume lake water.

- Step 1. Sampling staff (SS) puts on chest waders and a life vest before proceeding into the water with the integrated water column sampler and stainless steel bucket.
- Step 2. SS rinses the integrated water column sampler and stainless steel bucket before walking slowly out into the water to the first location. SS wades out until the water is approximately chest high, and then stands still to let the water and sediment settle.
- Step 3. SS holds the depth integrated water column sampler and slowly submerges it vertically until it just touches the wader boot. This is to avoid getting sediment in the sample. SS moves the sampler above and away from the boot before engaging the stopper, which is attached to the sampler via a cable, to secure the sample in the tube.
- Step 4. SS deposits the sample in a stainless steel bucket by holding the bottom of the tube over the center of the bucket and slowly releasing the stopper. Note: If this is a site where field duplicate or matrix spike and matrix spike duplicate (MS/MSD) samples is collected, SS collects two samples with the integrated water column sampler at each of the three locations.
- Step 5. Repeat steps 2 through 4 at the other two locations in the swimming area.
- Step 6. SS pours the composite sample from the stainless-steel bucket into the churn.
- Step 7. SS dons nitrile gloves.
- Step 8. SS labels three 40 milliliter (ml) amber glass vials., one of which will be analyzed for cyanobacterial cell counts and the remaining two will be analyzed for cyanotoxins. Of the cyanotoxin samples, one is unpreserved and the other is preloaded with a buffer solution in the lab before the sampling event. See Figure 1 for an example of labels.

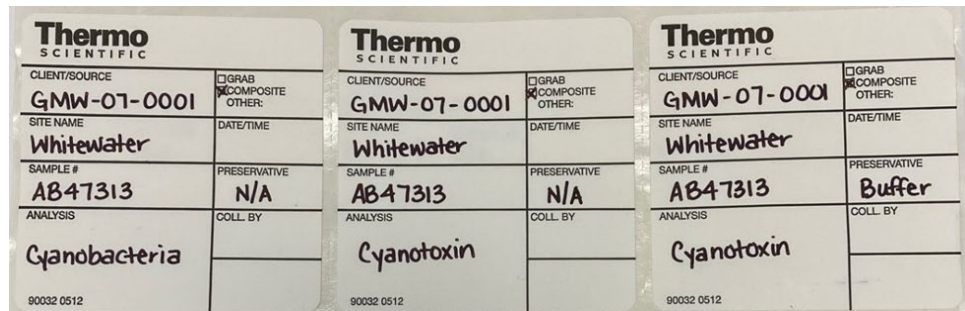


Figure 1. An example of labels for cyanobacteria and cyanotoxin sample vials.

- Step 9. SS begins to churn the water and fill each vial from the churn spigot. Fill the vials labelled as “Cyanobacteria” three-quarters of the way to allow headspace for mixing the sample in the lab. Fill the vials labelled as “Cyanotoxin - unpreserved” to the base of the collar. SS then pours half of the contents of the “Cyanotoxin - unpreserved” vial into the “Cyanotoxin – Buffer” vial. Screw the caps on to close each vial.
- Step 10. SS checks the pH of the buffered cyanotoxin vial to make sure it is between 6 to 8 due to sample storage requirements for anatoxin-a. Record the result in the notes section on the field data sheet.
- Step 11. SS stores all samples on ice in the cooler for transport to the lab.
- Step 12. SS dumps the bucket contents on the ground. Gloves may be removed and disposed of at this point.
- Step 13. SS rinses all sampling equipment with Millipore water.
- Step 14. SS fills out Chain of Custody forms.
- Step 15. SS delivers samples to the on-duty WAPB laboratory manager.

2.3. Related Technical Issues

A. Health and Safety Warnings

- 1. Avoid dermal contact with blooming cyanobacteria which can produce cyanotoxins in concentrations which can be toxic or lethal to animals and humans.

B. Cautions

- 1. Do not pull up on stopper too firmly or it will break.
- 2. Slowly release the stopper when emptying the composite sampler to avoid splashing.

C. Interferences

- 1. When wading out to sampling locations, allow lake water sufficient time to settle before sampling to minimize sediment in the composite sample.

D. Calibration

N/A

E. Troubleshooting

N/A

3.0. Roles

3.1. Responsibilities

A. Sampling Staff (SS)

- 1. Responsible for following this TSOP.

- B. Crew Chief
 - 1. Responsible for training the SS on this TSOP.
- 3.2. Training requirements
 - A. Using the integrated water column sampler and churn
 - 1. SS
 - B. Water sampling and Chain of Custody
 - 1. SS
 - C. Annual branch safety training
 - 1. SS
 - 2. Crew chief

4.0. Required Forms, Equipment, and Software List

- 4.1. Forms
 - A. Field Data Sheet
 - B. Chain of Custody
- 4.2. Equipment
 - A. Integrated water column sampler
 - B. pH meter
 - C. Stainless steel bucket
 - D. Churn
 - E. 40 ml amber glass vials
 - F. Labels
 - G. Cooler with ice
 - H. Millipore water
 - I. Nitrile gloves
 - J. Chest waders
 - K. Life vest
- 4.3. Software
 - A. N/A

5.0. Records Management

- 5.1. Records management is covered in the WAPB TSOPs “Determination of Cyanobacteria Toxins in Ambient and Drinking Water by ELISA”, S-001-OWQ-WAP-TGM-21-T-R4, and “Cyanobacteria Identification and Enumeration”, B-002-WAP-XX-15-T-R0.

6.0. Definitions

- 6.1. “Composite Sample” – A representative water sample made up of individual smaller samples taken at periodic intervals and composited into one representative sample for analysis.
- 6.2. Cyanobacteria – A division of microorganisms that are related to the bacteria but are capable of photosynthesis. They are prokaryotic and represent the earliest known form of life on the earth.
- 6.3. Cyanotoxins – Toxins produced by cyanobacteria.

7.0. Quality Assurance and Quality Control

- 7.1. SS collects field duplicates and MS/MSD samples at the rate of one per sample analysis set or one per every 20 samples, whichever is greater. SS also collects field blank samples, using ASTM D1193-91 Type I water, at a rate of one per sample analysis set or one per every 20 samples, whichever is greater. For each sampling event, SS fills out a Chain of Custody form which accompanies the samples from sample collection until delivery to the WAPB lab.
- 7.2. All sample labels are accurately and thoroughly completed, including AIMS sample numbers, waterbody name, sample type, preservative, date, time, and sample collector initials. After sampling completion at a given site, all equipment in contact with the sample is rinsed with ASTM D1193-91 Type I water.
- 7.3. SS undergoes periodic field audits by the section chief of the Targeted Monitoring Section to verify procedures are followed.

8.0. References

- [Determination of Cyanobacteria Toxins in Ambient and Drinking Water by ELISA](#), S-001-OWQ-WAP-TGM-21-T-R4
- [Cyanobacteria Identification and Enumeration](#), B-002-WAP-XX-15-T-R0.

9.0. Appendices

- 9.1. Field Data Sheet
- 9.2. Chain of Custody

Appendix 9.1: Field Data Sheet

IDEM Stream Sampling Field Data Sheet

Analysis Set #	EPA Site ID	Rank
22LAW004		1

Sample #	Site #	Sample Medium	Sample Type	Duplicate Sample #
AB51329	WLV-12-0002	Water + Igae	Normal	
Stream Name:	Cecil M Harden Lake	River Mile:	County:	Parke
Site Description:	Raccoon Lake DNR State Recreational Area Beach			
Survey Crew Chief	Sample Collectors	Sample Collected	Hydrolab #	Water Depth/Gage Ht (ft)
TED	ARS	Date: 5/31/22 Time: 9:30	E1	
Water Flow (cf/sec)	Flow Estimated?	Algae?	Aquatic Life?	
	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	
Sample Taken?	Aliquots	Water Flow Type	Water Appearance	Canopy Closed: %
<input checked="" type="checkbox"/> Yes <input type="checkbox"/> No; Stream Dry <input type="checkbox"/> No; Owner refused Access	<input type="checkbox"/> 1 <input type="checkbox"/> 2 <input type="checkbox"/> 3 <input type="checkbox"/> 4 <input type="checkbox"/> 6 <input type="checkbox"/> 8 <input type="checkbox"/> 12 <input type="checkbox"/> 24 <input type="checkbox"/> 48 <input type="checkbox"/> 72 <input type="checkbox"/> AS-Flow	<input type="checkbox"/> Riffle <input type="checkbox"/> Dry <input type="checkbox"/> Pool <input type="checkbox"/> Run <input type="checkbox"/> Glide <input type="checkbox"/> Eddy	<input type="checkbox"/> Stagnant <input type="checkbox"/> Flood <input checked="" type="checkbox"/> Other <input type="checkbox"/> Clear <input type="checkbox"/> Murky <input type="checkbox"/> Black <input type="checkbox"/> Brown <input type="checkbox"/> Green <input type="checkbox"/> Sheen <input type="checkbox"/> Black <input type="checkbox"/> Gray (Septic/Sewage)	<input checked="" type="checkbox"/> 0-20% <input type="checkbox"/> 20-40% <input type="checkbox"/> 40-60% <input type="checkbox"/> 60-80% <input type="checkbox"/> 80-100%
Special Notes:	Cyanotoxin buffer vial pH = 7.46			

Field Data:

Date (m/d/yy)	24-hr Time (hh:mm)	D.O. (mg/l)	pH	Water Temp (°C)	Spec Cond (µmhos/cm)	Turbidity (NTU)	% Sat.	Chlorine (mg/l)	Chloride (mg/l)	Chlorophyll (mg/l)	Weather Codes: SC WD WS AT			
5/31/22	09:30	9.27	8.49	22.985	367.4	27.1	108.2				1	18	1	5
Comments:														
Comments:														
Comments:														
Comments:														
Comments:														
Comments:														

Measurement Flags	< v m e R	< Min. Meter Measurement > Max. Meter Measurement Estimated (See Comments) Rejected (See Comments)	Weather Code Definitions			
			SC Sky Conditions	WD Wind Direction	WS Wind Strength	AT Air Temp

Field Calibrations:

Date (m/d/yy)	Time (hh:mm)	Calibrator Initials	Type	Meter #	Value	Units
5/31/22	09:30	ARS	Temp	54	22.9	°C
5/31/22	09:30	ARS	pH	54	8.56	SU
5/31/22	09:30	ARS	DO	51	9.20	mg/L
5/31/22	09:30	ARS	Turbidity	Q3	64.6	= NTU
Calibration Type						pH DO Turbidity
						70.8

Preservatives/Bottle Lots:

Group: Preservative	Preservative Lot #	Bottle Type	Bottle Lot #	GC	Groups: Preservatives	Bottle Types
1 Nx: H2SO4	070621	1000P	0114801E	Nx	General Chemistry: Ice	2000P 2000mL Plastic, Narrow Mouth
2 Cyano: Ice	N/A	406VAB	8D12D10	Metals	Nutrients: H2SO4	1000P 1000mL Plastic, Narrow Mouth
1 Cyano: buffer	20635016	406VAB	9140620	Metals	Metals: HNO3	500P 500mL Plastic, Narrow Mouth
				Metals	Cyanide: NaOH	250P 250mL Plastic, Narrow Mouth
				O&G	Oil & Grease: H2SO4	1000G 1000mL Glass, Narrow Mouth
				Toxics	Toxics: Ice	500G 500mL Glass, Wide Mouth
				EcolI	Bacteriology: Ice	250G 250mL Glass, Wide Mouth
				VOA	Volatile Organics: HCl & Thiosulfate	125G 125mL Glass, Wide Mouth
				Pest	Pesticides: Ice	40GV 40mL Glass Vial
				Phen	Phenols: H2SO4	120PB 120mL Plastic (Bacteria Only)
				Sed	Sediment: Ice	1000PF 1000mL Plastic, Coming Filter
				Gly	Glyphosate: Thiosulfate	500PF 500mL Plastic, Coming Filter
				Hg	Mercury(1631): HCl	60P 60mL Plastic
				Cr6	ChromiumVI(1636): NaOH	250T 250mL Teflon
				MeHg	Methyl Mercury(1630): HCl	500T 500mL Teflon
						125T 125mL Teflon

Data Entered By: TEA 6/1/22 qc1: ARS 6/2/22
 QC2: MR 6/20/22

