

# Total Phosphorus Sampling Methods

## Water Action Volunteers Manual 2017<sup>1</sup>

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### I. Project Contacts

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## II. Project Background and Goal

Phosphorus is the most visible, widespread water pollutant in Wisconsin. In more than 80% of our state's lakes, high levels of phosphorus can trigger excess algae and plant growth. When these excess plants die and decompose, oxygen levels drop dramatically and can lead to fish kills.

Streams act like conveyor belts, delivering phosphorus directly to lakes. Additionally, phosphorus is associated with excess sediments covering stream bottoms, the most common biological impairment in streams. Phosphorus in streams and lakes originates naturally from rocks, but its major sources today are usually associated with human activities: soil erosion, human and animal wastes, septic systems, detergents, and runoff from farmland or lawns.

An analysis of phosphorus often includes both *total phosphorus* and *soluble reactive phosphorus*. Volunteers will only sample for total phosphorus, which is considered a better indicator of nutrient status because its levels remain more stable than soluble reactive phosphorus. Total phosphorus includes particulate phosphorus—which is attached to bottom sediments and contained in plant and animal fragments suspended in water—and soluble phosphorus. Soluble reactive phosphorus dissolves in water and readily aids plant growth, but its concentration varies widely over short time periods as plants take it up and release it.

The goal of this monitoring is to characterize the total phosphorus concentrations most commonly occurring in the streams during the primary algae and aquatic plant “growing season” of May through October. Data will be reviewed during impaired waters listing assessments made by Department of Natural Resources staff.

## III. Volunteer Time Commitment

Volunteers must commit to attending a one to two hour training in its entirety prior to beginning participation in this monitoring project. Once trained, most volunteers will monitor one site monthly from May to October. Once at the monitoring site, it takes about five minutes to conduct the sampling. Volunteers must commit to sending in up to six water samples to the Wisconsin State Lab of Hygiene (WSLH) as soon as possible after sampling. All postage will be paid by the project sponsors, but the volunteer will need to package the sample on ice in a cooler that will be

provided, contact a WAV staff person to obtain a printable FedEx shipping label via email, print and affix that mailing label to the cooler, and arrange for the package to be picked up by FedEx and/or delivered to a local drop off point for FedEx delivery. In all, we estimate the time commitment per volunteer to be approximately 6 to 8 hours per year (including travel time to/from their monitoring site) if one site is monitored.

#### IV. Volunteer Duties

Volunteers will take grab samples in streams to monitor total phosphorus as a potential cause of biological impairment. **Phosphorus water samples should be collected once per month, for six months from May through October. Each sample should be collected approximately 30 days apart, with no samples collected within 15 days of one another.** Some volunteers may be asked to sample less frequently if some total phosphorus data are already available.

All grab samples should be shipped via FedEx to the Wisconsin State Lab of Hygiene in Madison for total phosphorus analysis.

#### Monitoring Schedule

	2016											
	Nov	Dec	Jan	Feb	Mar	April	May	June	July	Aug	Sept	Oct
Total Phosphorus Grab Samples	No total phosphorus samples are collected during this time period						One sample per site collected per month (approximately 30 days apart, with no samples collected within 15 days of one another)					

#### V. Safety

Safety precautions of a general nature should be recognized. Sampling should be done from shore whenever possible using an extension pole sampler to aid in water collection. Collecting samples in extremely hot and humid weather carries the risk of dehydration and heat stroke. Preserving nutrient samples requires the use of small amounts of acid. Caution should be used to avoid contact with skin or eyes when acidifying the sample. A first aid kit should always be carried for general safety considerations.

## VI. Field Sampling and Preservation Methods

### A. Overview

The following water sampling requirements are taken from the Wisconsin State Lab of Hygiene protocols. All water samples collected for analysis need to comply with the equipment and procedures that follow.

### B. Equipment

- 250 mL polyethylene bottle(s) (one per site)
- Extension pole (PVC pipe or stick) with rubber band to use to fasten bottle
- 1.0 mL vial of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>)
- Nitrile or latex gloves
- Safety glasses
- Waterproof pen or marker
- Lab slips (also called “Test Request – Inorganic Surface Water & Microbiology” form) (one lab slip for each sampling site per visit; each lab slip is unique and should *never* be photocopied)
- Cooler(s) (one for every 2-3 samples)
- 2 gallon Ziploc bags filled with ice cubes (not ice packs)
- Waders or shoes that can get wet (not needed if sampling is done from shore)

### C. Considerations/ Precautions

To avoid bias, the monitoring should be conducted at a sampling location as follows:

- **Avoid disturbing the sample.** If the sample is collected by wading in the stream, walk upstream to the sample location and take the sample facing upstream. Also avoid surface water and your hands from touching the rim of the bottle or inside of the cap.
- **Collect water sample 3 to 6 inches below surface, rinse sample bottle three times and fill it to its neck the fourth time.** Surface samples tend to have debris and other things floating on the surface and should be avoided.
- **Do not collect sample immediately downstream of a wastewater or storm sewer outfall pipe.**
- **Ensure sample is representative of the upstream conditions.** Stream reaches with major springs or major sediment deposits, such as former millpond beds, may create much localized conditions that aren’t reflective of the upstream conditions and should be avoided. Also avoid reaches immediately downstream of where cattle are in the stream.

- **Ensure sample is collected in an area with thorough mixing of stream water.** Stream reaches immediately downstream from tributaries or major springs may not have complete mixing and should be avoided.
- **Collect sample in portion of stream with greatest or strongest flow<sup>2</sup>.** This may or may not be in the middle of the stream. In general, relatively straight reaches of the stream are preferred. However, if a meander section of the stream is selected for sampling, the sample should be collected in the portion with greatest flow at the outside of the meander. Slow flow areas along the banks, in eddies or immediately downstream of islands should be avoided. Note: If sampling with an extension pole, reaching out from shore to an area of flow with some movement (and not necessarily to an area of strongest flow) is sufficient. Your safety is most important!
- **Don't trespass on private lands to collect sample.** Use a public access point, such as a road right of way, or seek permission from the landowner or operator to cross their land for the purpose of collecting the samples.

#### D. Collection method

1. **On a 250 ml sample bottle circle the word “nutrients,” check the H<sub>2</sub>SO<sub>4</sub> box, and write the field number and sample location on the bottle** (the latter two are listed on your lab slip as the “Field Number” and the “Sample Address or Location Description”) (Figure 1).
2. Locate a sampling location that is at least 10 to 20 feet away from a bridge crossing, in the middle of the stream channel, and is at least knee deep\*.  
**Enter the stream and walk upstream** towards the sampling location. This ensures the sample is not contaminated by sediment that has been dislodged from the substrate.
3. Facing upstream, **rinse the 250 mL polyethylene bottle three times with water 3 to 6 inches below the water's surface. The fourth time, fill the bottle to its shoulder and cap.** Whenever possible, and especially when stream flow is swift or water levels are

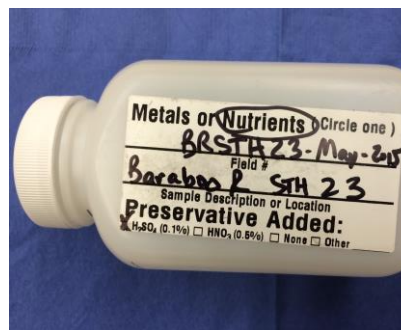


Figure 1. 250 mL polyethylene sample bottle.

<sup>2</sup> In some instances, water depth may be very shallow. When sampling during low flow conditions, take extreme care to ensure that the sample is not contaminated with sediment dislodged from the substrate. Also, at such times, it is more important to collect the sample in the area of strongest flow than the deepest location. Contact WAV staff if the water levels are low and you require a syringe to collect the water sample.

high, fasten the bottle to an extension pole and use that to collect stream water that is well mixed.

4. Avoid touching or allowing water at the surface / scum on the surface to touch the rim of the bottle or inside of the cap. One way to prevent this is to uncap and recap the bottle underwater. If you uncap the bottle above the water's surface, always place the cap top side down to avoid contamination (Figure 2).
5. Wearing gloves and safety glasses, preserve the sample by **adding a vial of sulfuric acid to the 250 mL bottle** (Figure 2). (Back at home, triple rinse the empty vial with water and dispose in the garbage.)
6. **Securely cap the bottle and mix the sample by inverting the bottle several times.**
7. Transport the sample on ice and prepare it to be shipped to the State Lab of Hygiene



**Figure 2.** Add a vial of sulfuric acid to sample. Be sure to place cap topside down to avoid contamination.

## E. Site Photographs

During each visit, please **take a photograph of the monitoring site** and submit it to the monitoring coordinator to upload to SWIMS.

## F. Documentation

**A separate lab slip must be submitted with each sample.** Lab slips will be provided to you by WAV staff and should *never* be photocopied. Most of the required fields on the lab slip are automatically filled out for you, but volunteers still need to fill the following fields<sup>3</sup>:

- Time and Date of Sample Collection, including:
  - Date (mm/dd/yyyy)
  - Time (24-hr clock)
- Who Collected the Sample, including:
  - Your name
  - Your phone number
  - Your email



**Figure 3.** Complete one lab slip for each sample.

<sup>3</sup> Without the above information on the lab slip, the lab will be unable to process the water sample. This could result in the sample not being tested in a timely manner and therefore be flagged in SWIMS as exceeding the holding time.

## VII. Delivery of Samples to the Wisconsin State Lab of Hygiene

### A. Overview

Madison area volunteers should deliver their samples directly to the Wisconsin State Lab of Hygiene (2601 Agriculture Drive Madison, WI 53718) whenever possible. Volunteers outside of the Madison area will be sent a prepaid FedEx shipping label to send samples to the State Lab of Hygiene at the beginning of each month so that the sample can be sent out as soon as possible after collection (see instructions below). Please contact Ilana Haimes if you have not received an email with the label by the date you are sampling.

If you would like WAV staff to schedule a FedEx pick up for your sample(s), please contact Ilana Haimes in advance to let her know the day and time your sample(s) will be ready and if there are any specific instructions for pickup (back door, on porch, etc). Contact Ilana if you have any questions about this process.

### B. Equipment

- 250 mL polyethylene bottle(s) (one per site)
- Lab slips (one per site)
- Shipping cooler(s)
- Quart Ziploc baggies (one per sample)
- 3 gallon Ziploc bags
- 1 large plastic liner bag
- Ice cubes (not ice packs)
- 1 plastic zip tie
- Shipping tape

### C. Packing and Shipping Instructions

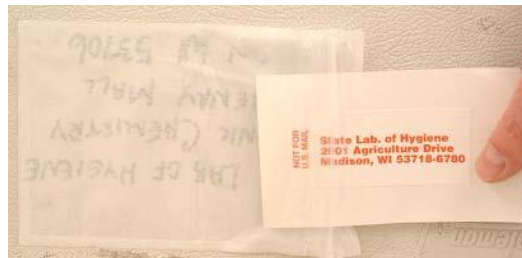
1. Place each sample in **separate quart-size Ziploc bag** and securely close. DO NOT place multiple 250 mL bottles in the same bag.
2. Line cooler with the large plastic bag provided.
3. Fill two gallon size Ziploc bags with ice cubes (generally you want at least equal parts of ice and water sample in the cooler, more if it's very warm outside).
3. Insert one Ziploc bag of ice into the cooler inside the liner bag.
4. Add the quart Ziploc bag(s) containing the sample(s) on top of that.
5. Add the second Ziploc bag of ice on top of the samples.

6. Twist the liner bag closed.
7. Securely seal the bag with a plastic zip tie to prevent leakage during shipping.
8. Double check that the lab slip(s) is/are completed, and then **place lab slip(s) in a gallon Ziploc bag and place on top of the bag, or on top of the Styrofoam cooler but within the shipping box**, being cautious not to tape the Ziploc if the box isn't fully closed when you add the packaging tape.
9. Close the box lid and wrap with reinforced shipping tape completely around the box.



**Figure 4.** Shipping container with FedEx label on the side.

10. Remove the mailing label card from the plastic envelope, and flip over so the Wisconsin State Laboratory of Hygiene address is exposed and reinsert into envelope.



**Figure 5.** Mailing label card with WSLOH address facing out.

11. Ship the samples to the Wisconsin State Laboratory of Hygiene using FedEx. Contact Ilana Haimes to obtain the prepaid shipping label via email if you have not received one. Any time WAV staff request a FedEx shipping label on your behalf, you will receive an email with a link that when you click on it, will display a printable shipping label. Print it out and affix it to your cooler. Then bring the package to the nearest FedEx shipping location or call (1-800-463-3339) or contact WAV staff to have a FedEx pickup scheduled for you.
12. **Report the date you sampled in the Google Drive document** (the link for this will be shared with you via email following your training). As it is imperative all six monthly samples are collected, a WAV staff person will contact you if you have not entered a sampling date to this document by about the 20<sup>th</sup> of each month.



## D. Considerations/ Precautions

**Samples should be shipped as soon as possible, ideally within 1-2 days after being collected.** Holding times are longer than one week, but the clock begins immediately after sampling and the lab needs them quickly in order to process samples on time. **However, do not mail samples on Fridays or Saturdays because lab staff is usually not present on weekends. If the ice melts completely, sample data will be flagged and unusable.** If the weather is extremely warm, if you collect a sample from more than one site, and/or if you collect QA/QC samples in addition to your regular sample you should add extra ice or use multiple shipping coolers to submit your samples to the lab. **Refrigerate the samples or keep them on ice until they are shipped.**

## E. Questions?

Volunteers should feel free to contact Ilana Haimes (608-266-3599) or Peggy Compton (608-342-1633) with questions or concerns about any aspect of the project.

## VIII. Field QC Duplicate Samples

### a. Overview

To document the accuracy and precision of the field data collected by volunteers, ten percent of the samples that are monitored for total phosphorus (TP) are chosen each year to participate in collection of additional quality assurance/quality control (QA/QC) samples. The samples are randomly selected from the list of stations that are monitored. These QA/QC tests document the accuracy and precision of the data collected and look at natural variability and sampling error.

Two types of QA/QC samples will be collected by volunteers: field blank and field replicate (duplicate) samples for total phosphorus. If your site(s) have been selected at random for QA/QC sampling, you will be notified. To carry out sampling, please read the following instructions.

The result of this additional sampling is that you will be mailing a total of three water samples to the lab following one of your site visits: one regular sample, one duplicate, and one blank. Each sample should be placed in a separate Ziploc baggie. Refrigerate or ice the samples until you are ready to ship, ideally within 1-2 days, like with your regular sample.

## B. Equipment

- 250 mL polyethylene bottle
- Extension pole (PVC pipe or stick) with rubber band to use to fasten bottle
- 1.0 mL vial of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>)
- Nitrile or latex gloves and safety glasses
- Waterproof pen or marker
- Lab slip marked as “duplicate”
- Cooler with ice (from regular sampling)
- Waders or shoes that can get wet (not needed if sampling is done from shore)

## C. Collection Method

Please prepare this sample at the same time as your regular samples, as follows:

**Collect, preserve, and ship a second sample from the same location** following the directions on pages 4-8 of this manual, **but write “DUPLICATE” on the bottle after the field number.** Find the lab slip that is pre-marked “DUPLICATE” near middle of the page, fill in the required fields, and place it in the Ziploc bag containing lab slips.

Collected By Name		Telephone	Email
<b>Where the sample was collected</b>			
Station ID (STORET #)	Sample Address or Location Description		
573076	Baraboo River at STH 23-Bridge in Reedsburg		
County	Waterbody ID (WBIC)	Point / Outfall (or SWIMS Fieldwork Seq No)	
57 - Sauk	1271100	81878578	
<b>Sample Details</b>			
Sample Description / Device Description			
Enforcement? <input type="radio"/> Yes <input type="radio"/> No	If Field QC Sample (select one):		Depth of Sample: _____ <input type="radio"/> ft <input type="radio"/> m <input type="radio"/> in <input type="radio"/> cm
If yes, include chain of custody form.	<input checked="" type="radio"/> Duplicate <input type="radio"/> Blank <input type="radio"/> _____		Or Top and Bottom of Sample Interval:
Is Sample Disinfected? <input type="radio"/> Yes <input type="radio"/> No	Grant or Project Number		_____ - _____ <input type="radio"/> ft <input type="radio"/> m <input type="radio"/> in <input type="radio"/> cm
If yes, how? _____			

**Figure 6.** Lab slip for the duplicate sample.

## IX. Field QC Blank Samples

### A. Equipment

- Distilled water
- 250 mL polyethylene bottle
- 1 mL vial of sulfuric acid (H<sub>2</sub>SO<sub>4</sub>)
- Waterproof pen or marker
- Lab slip marked as “blank”

## B. Collection Method

Please prepare this sample after the collection of your regular samples, as follows:

**With a waterproof marker, circle the word “nutrients” on a 250 ml bottle, check the H<sub>2</sub>SO<sub>4</sub> box, and write the field number and sample location on the bottle** (these are listed on your lab slip as “Field Number” and “Sample Address or Location Description”). **To the right of the field number, write “BLANK”.**

**To prepare the sample, rinse the bottle three times with distilled water, then fill it to its neck with distilled water. Preserve the sample with acid and then securely cap it. Mix the sample by inverting the bottle several times.** Ship the sample along with the regular and duplicate samples following the directions in Section VII of this manual.

**To complete the lab slip,** find the lab slip that is pre-marked with the words “BLANK” near the middle of the page, fill the required fields, and place it in the Ziploc bag containing the regular and duplicate sample lab slips.

Collected By Name	Telephone	Email
<b>Where the sample was collected</b>		
Station ID (STORET #) 573076	Sample Address or Location Description Baraboo River at STH 23-Bridge in Reedsburg	
County 57 - Sauk	Waterbody ID (WBIC) 1271100	Point / Outfall (or SWIMS Fieldwork Seq No) 81878578
<b>Sample Details</b>		
Sample Description / Device Description		
Enforcement? <input type="radio"/> Yes <input type="radio"/> No If yes, include chain of custody form.	<b>If Field QC Sample (select one):</b> <input type="radio"/> Duplicate <input checked="" type="radio"/> Blank <input type="radio"/>	Depth of Sample: _____ <input type="radio"/> ft <input type="radio"/> m <input type="radio"/> in <input type="radio"/> cm
Is Sample Disinfected? <input type="radio"/> Yes <input type="radio"/> No If yes, how? _____	Grant or Project Number	Or Top and Bottom of Sample Interval: _____ - _____ <input type="radio"/> ft <input type="radio"/> m <input type="radio"/> in <input type="radio"/> cm

**Figure 7.** Lab slip for the blank sample (filled with distilled water).

## X. Equipment Cleaning Procedures

*Anytime you monitor, even if just at one stream site:*

BEFORE leaving the stream:

- INSPECT equipment and REMOVE sediments, plants and animals
- SCRUB equipment with a stiff brush (including crevices) to remove sediment and debris
- RINSE equipment with tap water (using a spray bottle is useful for this)
- DRAIN all water from equipment

