OLD WOMAN CREEK
NATIONAL ESTUARINE RESEARCH RESERVE

Technical Reports

Determination of Wetland Total Phosphorus Retention Capacity

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OLD WOMAN CREEK NATIONAL ESTUARINE RESEARCH RESERVE

Old Woman Creek National Estuarine Research Reserve (OWCNERR) is managed as a cooperative partnership between the Ohio Department of Natural Resources and the National Oceanic and Atmospheric Administration (NOAA). OWCNERR is one of 29 coastal reserves connected nationally through NOAA to address state and regional coastal management needs through research, education, and stewardship. The National Estuarine Research Reserve System uses its network of living laboratories to help understand and find solutions to crucial issues facing America's coastal communities.

The mission of OWCNERR is to improve the understanding, stewardship, and appreciation of Great Lakes estuaries and coastal wetland ecosystems. Integrated Reserve research, education, and stewardship programs address threats to Great Lakes coastal wetland ecosystems including nonpoint source pollution, aquatic invasive species, habitat loss, and climate change. OWCNERR provides laboratories for ecological research and education and training to support decisions and actions that benefit Lake Erie ecosystems.

OWCNERR publishes the OWCNERR Technical Reports series to provide constituents with information derived from Great Lakes coastal research, management, education, and outreach projects conducted in partnership with and/or at OWCNERR.

Janice Kerns, *Manager*

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The Old Woman Creek National Estuarine Research Reserve is part of the National Estuarine Research Reserve System, (NERRS), established by Section 315 of the Coastal Zone Management Act, as amended. Additional information about the system can be obtained from the National Ocean Service Office for Coastal Management, National Oceanic and Atmospheric Administration, US Department of Commerce, 1305 East West Highway – N/ORM5, Silver Spring, MD 20910.

Determination of Wetland Total Phosphorus Retention Capacity

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Monitoring Goal and Conceptual Framework

This protocol is intended to enable wetland managers, conservationists, and other practitioners to monitor and estimate a wetland's long-term Total Phosphorus (TP) retention capacity threshold. This method can be applied to flow-through wetlands; i.e., wetlands that have a distinct point of inflow (e.g., stream or pump) and point of outflow (e.g., stream or water control structure).

Total Phosphorus retention in a wetland is a measure of the storage of TP via either short-term or long-term processes. Each of these compartments has a **retention capacity**, which is the maximum amount of TP it can store. Short-term storage compartments include plants, algae, microbes, and the soil (via adsorption and precipitation). Long-term phosphorus storage occurs when TP is buried and retained in the sediment (Figure 1). This long-term storage is what determines a wetland's **long-term retention capacity** and is a function of the rate of sediment accretion in the wetland (Richardson et al. 1997).

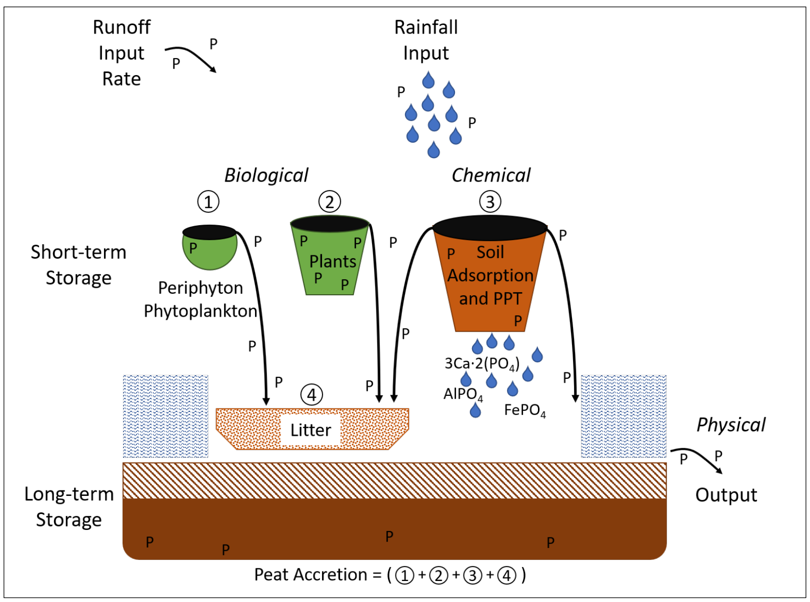

Figure 1. Conceptual model of wetland phosphorus retention. P=total phosphorus; PPT=precipitation. Modified from Richardson et al. (1997).

A wetland's **TP retention capacity threshold** is its maximum storage threshold, i.e., the point at which the wetland can no longer store additional TP. The threshold can be determined by comparing the rate of TP flowing into the wetland (i.e., TP loading in ton/y) to the concentration of TP (mg/L) leaving the wetland. As TP loading into a wetland increases from low to high, the concentration of TP flowing out of the wetland remains low until TP loading in reaches a certain threshold level. Above that level, outflowing concentrations become higher and more variable. We refer to this relationship as a "hockey-stick" relationship, which can be quantified using a change-point model (Figure 2). The TP retention threshold is the point at which the relationship shifts from a mostly flat line to an increasing, diagonal line. Beyond this point, the wetland's long-term retention capacity is exceeded, and the excess TP will flow out of the wetland sooner or later (when various short-term storage pools are filled). In other words, the wetland is being overloaded with TP. The retention threshold for wetlands will differ based on their size, age, and rate of sediment accumulation.

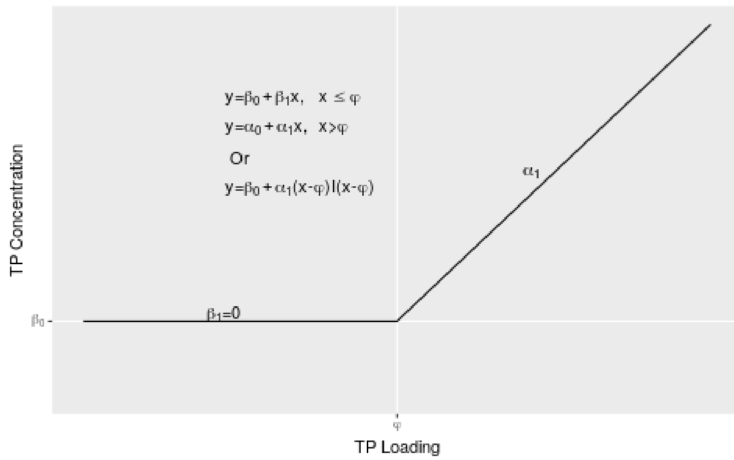

Figure 2. Changepoint model showing the relationship between wetland inflow TP loading and outflow TP concentration. The point of inflection (φ) is an estimate the TP retention threshold. Modified from Qian and Richardson (1997).

The long-term TP retention threshold value can be used to quantify the relative contribution of a wetland in retaining TP before water flows into a recipient water body. The threshold can also be used to evaluate TP loading into a wetland relative to its retention threshold, to determine if a wetland is being overloaded or if it could retain additional loading. Monitoring a wetland's retention threshold over time could be used to identify when the wetland is becoming saturated and might need a management intervention such as sediment removal. Additionally, the fact that this is a Bayesian model means that threshold estimates will become more accurate as more wetland data are included.

Sample Collection

A. *Inflow sampling requirements*

Parameters: TP concentration (mg/L) in water column

Water discharge (rate of flow, feet^3/second or m^3/sec)

Location: Both should be sampled at the same location, at or immediately upstream of the inflow to the wetland

B. *Outflow sampling requirements*

Parameters: TP concentration (mg/L)

Location: At the or immediately upstream of the outflow from the wetland

Note: avoid or flag the collection of water samples when water from the receiving water body is flowing into the wetland via the outflow (e.g., seiche or micro-tidal induced inflow of Lake Erie water into a coastal wetland)

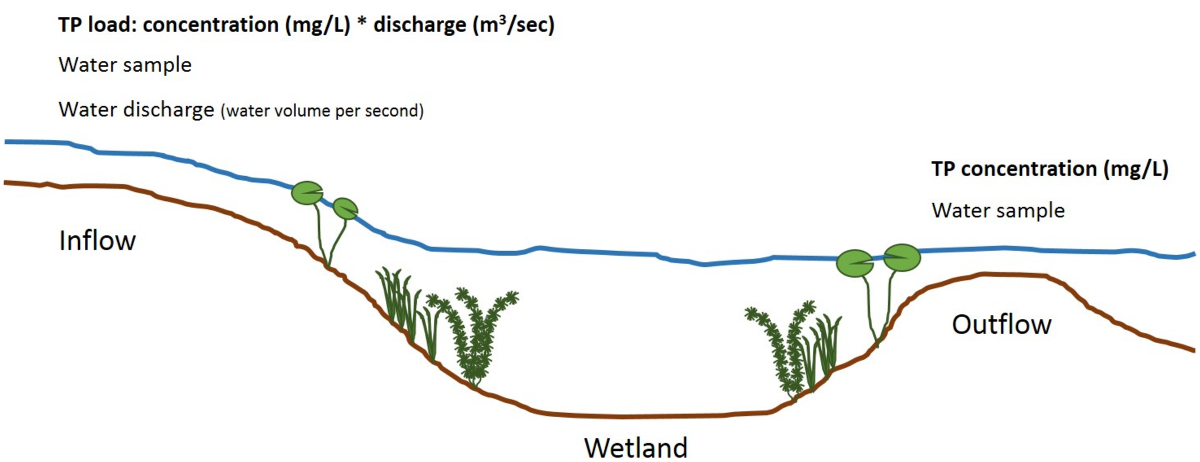

Figure 3. Schematic showing inflow and outflow sampling needed to estimate a wetland's TP retention capacity.

C. *Equipment and supplies needed*

See Appendix A for example companies, models, and approximate pricing

<u>Water Samples</u>

Autosampler:	Autonomous sampling of water quality samples at set intervals (e.g., daily, weekly)
	Ice packs, sample bottles, batteries or power source

Grab samples:	1 L, amber high-density polyethylene bottles, wide mouth

Cooler:	With ice packs, for sample transport
Freezer:	If needed, for sample storage before analysis

<u>Water discharge</u>

USGS or river current profiler

- A map of USGS gauge stations in Ohio can be found at waterdata.usgs.gov/oh/nwis/rt
- River or stream current profilers are expensive and require technical skills to set up and maintain;

<u>or</u>

Pump rate (volume/time), if all inflowing water is being pumped;

<u>or</u>

Deployable water level sensor (requires a stage-discharge rating curve; see below).

D. Sampling Procedure

TP concentration

Water samples should be collected weekly or bi-monthly at both sites and on the same dates. Samples should be collected throughout the year and wet and dry conditions to include a range of low to high concentrations. Sampling should span at least two year; the specific minimum sample size requirement depends on site conditions.

Samples can be collected automatically using an autosampler programmed to collect samples at a set interval or collected by hand. Samples must be kept cool, either with a refrigerated sampler or by putting ice packs in the sampler. Samples should be transported in a cooler with ice or ice packs.

Procedure

1. All sample bottles and caps should be acid washed with 10% Hydrochloric acid (36.5-38%; CAS: 7647-01-0);
    i. For 1 L of acid wash, add 10 mL of hydrochloric acid to 90 mL of deionized water;
        - *Always add the acid to the water, wear safety goggles, and do this in a lab hood or well-ventilated area!*

    ii. Use a squirt bottle to spray the entire inside surface of each bottle *or* pour enough acid wash in each bottle that swirling and shaking the bottle with the cap to expose the entire inside surface;

    iii. Caps can be washed like the bottles or they can all be submerged in acid wash in a plastic or glass container.

2. Following acid wash, rinse each bottle and cap 6 times with deionized water and let dry;

3. If using an autosampler:
    i. Bottles should be placed in carousel and caps removed immediately before programming and starting the sampler per directions;

    ii. Program the autosampler to rinse the intake tubing 3 times before filling bottle;

    iii. Change out the peristaltic pump tubing as needed to maintain autosampler performance (default programming is ~ every 10,000 pumps);

    iv. Change out the intake tubing as needed to reduce development of detritus and biological "film" inside tubing.

If collecting grab samples by hand:

i. Rinse the bottle(s) and cap(s) three times with water from the sample site, slightly away from where the sample will be collected;

ii. Submerge bottle, mouth down; while submerged, turn bottle such that mouth faces up for air bubbles escape;

iii. Lift bottle from water, leaving ~ ½" of air at the top;

iv. Cap the bottle.

4. Place bottles in a cooler with ice or ice packs for transportation;

5. Preserve, store, and/or ship samples following the analytical lab's instructions (see below for recommended sample preservation and hold times).

Water discharge

Measured at least as frequently as sampling occurs.

1. If a USGS gauge station is located at or immediately upstream of the inflow sample site:

   i. Download data from the USGS website;

   ii. Calculate daily or weekly means.

2. If all water entering the wetland is being pumped:

   i. Use the known pump rate; if pump rate varies, calculate daily or weekly means.

3. For non-gauged, non-pumped sites:

   i. A rating curve is required to convert water level to flow (see Cornell 2013 in "Related Documents" for instructions on how to develop a rating curve);

   ii. Record water levels using a water level datalogger programmed to collect readings at set intervals;

   iii. Download the data from the logger per manufacturer's instructions and calculate daily or weekly means.

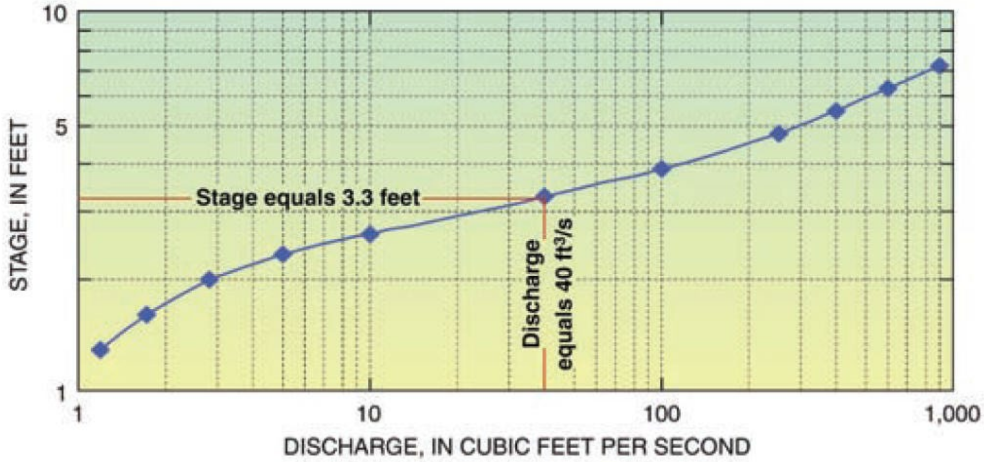

Figure 4. Example rating curve showing the relationship between stage and discharge. Source: usgs.gov.

Sample Analysis

A. *Analytes*

Total Phosphorus

Note: future model development could include the ability to analyze soluble reactive phosphorus (i.e., orthophosphate) and/or nitrate

B. *Sample preservation and hold times*

Standard Methods for Examination of Water and Wastewater

1. Store at 4˚C, up to 24 h or
2. Add sulfuric acid (H2SO4) until sample is pH < 2 and refrigerate up to 28 d

Alternate methods

1. NOAA National Estuarine Research Reserve System: freeze at < -20˚C up to 28 d
2. Recommended or required protocol of contract lab

C. *Analytical method*

4500-P Persulfate Digestion Method, 880 nm spec or autoanalyzer (Standard Methods for Examination of Water and Wastewater)

D. *Laboratory options*

Samples can be sent to a state, university, or private laboratory for analysis.

Appendix B lists some laboratories in Northern Ohio that can be contacted to inquire about sample analysis services and pricing.

Data Management

Data management, quality control and assurance (QAQC), and documentation are important for maintaining a robust and meaningful dataset.

A. Metadata recommendations

Detailed metadata are important for documenting and maintaining data sets. Metadata include general information about monitoring objectives, sampling locations, procedures, equipment, and data ownership and management. They also include detailed information about sample and data collection, sample analysis, data calculations, and data validation (i.e., quality assurance and control measures).

See Appendix C for an example metadata template.

B. Data Management

Most likely, data will be received for individual sample runs in digital format as a .csv or .txt file or a file produced by spreadsheet software (e.g., .xlsx from Excel)

<u>Master spreadsheet</u>
A master spreadsheet should be created into which data from other sources (e.g., USGS or contract lab) can be copied and pasted

1. The following columns are needed at a minimum: (include QAQC columns)
    i. Date of sample collection
    ii. Measured inflow (m3/sec)
    iii. Measured TP input concentration (mg/L)
    iv. Measured TP output concentration (mg/L)
2. Units should be included in the column headings for each analyte or parameter
    i. Make sure units reported by the contract lab match the units in the master spreadsheet or that data conversions are completed correctly
3. Include conversion columns in the master spreadsheet, so that those can be reviewed if needed
    i. To convert from cfs to m^3/s
        1. 1 cfs = 0.02832 m^3/s
        2. Flow in m^3/s = X cfs 8 0.02832 m^3/s

3. Example calculation, if flow is 12.4 cfs

   12.4 cfs * 0.02832 m^3/s = 0.35117 m^3/s

ii. To convert from microMolar (µM, or micromoles/L) to mg/L

1. The atomic weight for P = 30.97 g/mol
2. microMolar units are micromoles/L
3. 30.97 g/mol = 30970.0 mg/mol = 0.03097 mg/micromole
4. 0.03097 mg/micromole * X micromoles/L → mg/L as P
5. Example calculation, if lab reports a P value of 2.061 µM/L:

   2.061 µM/L * 0.03097 mg/ µM = 0.0638 mg/L

C. *Data Validation*

Data validation or quality assurance and control (QAQC) can be conducted on individual sample run data sets and/or over larger time periods that cover multiple runs (e.g., quarterly or annual).

Validation provides a way to evaluate the quality of the data and document any concerns about the validity of the data, including rejecting erroneous data.

Validation should include:

- A coded system for flagging data as rejected or suspect, including the reason;
- Reviewing any metadata or correspondence from the contract lab about the quality of individual sample runs
  - Use this information to flag any samples as rejected or suspect, as needed; and
- Plotting the data as a time series and reviewing the plots for outliers.

Below is an example coding system that includes general flags and specific codes reported as *generalflag_specificcode* (e.g., -2_CM):

General flags:

-2	*Data rejected during validation*
-1	*Missing data*
0	*Data passed validation*
1	*Suspect data*
2	*Corrected data*

These general flags can be combined with more specific codes to explain the flagging or factors.

CM	*Calculated value could not be determined due to missing data*
CR	*Calculated value could not be determined due to rejected data*
DM	*Data missing or sample never collected*
RD	*Replicate values differ substantially*
BL	*Value below minimum limit of method detection*
CB	*Calculated value could not be determined due to a below MDL component*
UL	*Value above upper limit of method detection*
OR	*Value outside of sensor detection range*
HB	*Sample held beyond specified holding time*
US	*Lab analysis from unpreserved sample*
AP	*Algae or scum present in sample vicinity*
IP	*Ice present in sample vicinity*
RE	*Significant rain event*
SE	*Significant seiche event*
WE	*Significant weather event*
SM	*See metadata notes*

Data analysis and interpretation

A. *Github Repository*

A model user guide, example data set, statistical code, and other supporting information are housed in a Github repository. The user guide includes instructions for preparing and uploading data to run the model. It also includes instructions and a dataset for working through an example. https://github.com/songsqian/owc/tree/master/threshold.

References and Resources

A. References

Qian, S.S., and C.J. Richardson. 1997. Estimating the long-term phosphorus accretion rate in the Everglades: A Bayesian approach with risk assessment. Water Resources Research 33(7): 1681-1688.

Richardson, C.J., S. Qian, C.B. Craft, and R.G. Qualls. 1997. Predictive models for phosphorus retention in wetlands. Wetlands Ecology and Management 4: 159-175.

B. Model Interface

https://github.com/songsqian/owc/tree/master/threshold

C. Story Map

A story map, describing the application of this modeling approach to three Ohio wetlands can be viewed at: www.nerrssciencecollaborative.org/working-wetlands

D. Science Collaborative project page

Information about the project and links to final products can be accessed at: http://nerrssciencecollaborative.org/project/Arend17

Acknowledgements
This work was sponsored by the National Estuarine Research Reserve System Science Collaborative, which supports collaborative research that addresses coastal management problems important to the reserves. The Science Collaborative is funded by the National Oceanic and Atmospheric Administration and managed by the University of Michigan Water Center (NA14NOS4190145).

Appendix A. Equipment and Supplies Specifications

1. *Autosamplers (note: these come with sampler-specific sample bottles)*

   a. Hach brand AS950 Peristaltic Samplers
      i. Portable (~ $5.5-6 K): www.hach.com/as950-peristaltic-samplers/portable-samplers/family?productCategoryId=35547137070
      ii. All-weather refrigerated (likely ≥ $6 K): www.hach.com/as950-peristaltic-samplers/all-weather-samplers/family?productCategoryId=35547137072
   b. Xylem/YSI
      i. WS705 Composite-Discrete Water Sampler (~$2 K): www.ysi.com/WS705
      ii. ProSample portable samplers (~$3-4 K): www.ysi.com/prosample
   c. Teledyne ISCO
      i. Portable Samplers (sequential and composite only) (likely < $5 K): www.teledyneisco.com/en-us/water-and-wastewater/portable-samplers
      ii. Portable Refrigerated Samplers (sequential and composite only) (likely < $5K): https://www.teledyneisco.com/en-us/waterandwastewater/Pages/Avalanche.aspx
      iii. 5800 Refrigerated Sequential/Composite Sampler (~$6-7 K): www.teledyneisco.com/en-us/waterandwastewater/Pages/5800-Sampler.aspx
   d. Note: some accessories, such as replacement peristaltic pump and intake tubing can be purchased through secondary vendors at cheaper prices.

2. *Grab Sample Collection Bottles: 1 L, amber, high-density polyethylene for collection samples by hand; smaller volume bottles can be used to store frozen samples for analysis*

Vendor	Brand	Catalog Number	Cost / quantity
VWR	VWR	89221-680	$255/case of 24
VWR	Nalgene	16059-900	$580/case of 50
Fisher Scientific	Wheaton	02-911-273	$425/case of 48
Fisher Scientific	Nalgene	05-719-345	$260/case of 24

Note: additional vendors exist

3. *Water Level Dataloggers (note: software usually required for data acquisition)*

   a. Solinst Levelogger Junior Edge (~ $400): www.solinst.com/products/dataloggers-and-telemetry/3001-levelogger-series/levelogger-junior-edge/

   b. Solinst Levelogger Edge (~$600 Cost): www.solinst.com/products/dataloggers-and-telemetry/3001-levelogger-series/levelogger-edge/

   c. HOBO U20 Series ($300 - $500; starter kit, includes software and base station: $600): www.onsetcomp.com/products/data-loggers/u20-series-water-level-data-loggers

   d. In-Situ Rugged TROLL 100 Data Logger ($420; docking station: $225): in-situ.com/products/water-level-monitoring/rugged-troll-100/

4. *Vitalli Sheremet at University of Rhode Island has developed low-cost flow meters that could be used in place of water level dataloggers and a rating curve:* https://www.eurekalert.org/pub_releases/2010-09/uori-ncm091410.php

Appendix B. Laboratories in NE Ohio for sample analysis

Laboratory	Location	Website	Contact
National Center for Water Quality Research, Heidelberg University	Tiffin, OH	ncwqr.org/water-testing/surface-water-testing/	419.448.2940; 800.925.9250 Ext. 2940
Jones and Henry Laboratories	Northwood, OH	www.jhlaboratories.com/services.html	419-666-0411
Alloway Laboratories	Lima, Mansfield, and Marion, OH	www.alloway.com/sampling-analysis	800-436-1243; Mansfield: 419-525-1644
Northeast Ohio Regional Sewage District	Cleveland, OH	www.neorsd.org/business-home/environmental-services-and-laboratory/	216-881-6600
Old Woman Creek National Estuarine Research Reserve, ODNR	Huron, OH	http://coastal.ohiodnr.gov/oldwomancreek	419-433-4601

Appendix C. Example Spreadsheet

Date_ Collected	F_Record	InflowRate _cfs	InflowRate _m3s	F_InflowRate	TPin_*labunits*	F_Tpin_ *labunits*	TPin_mgL	F_Tpin_mgL	TPout_*labunits*	F_Tpout_*labunits*	TPout_mgL	F_TPout_mgL
4/4/2020		250	7.08		13.56		0.4200		6.46		0.2001	
4/5/2020		12.4	0.351168		7.51		0.2326		3.24		0.1003	

Note: above example is for lab units originally reported as µm/L; the column headings would therefore be: "TPin_umolL" and "TPout_umolL"

Appendix D. Example Metadata Template

Template is a modification of the Nutrient Metadata Template (2018) developed by the National Estuarine Research Reserve System Centralized Data Management Office.

Organization Name Wetland Name **Water Quality Monitoring Metadata**

Months and year the documentation covers

Latest Update: *Date that the last edits were made*

<u>Research Objectives, Study Sites, and Methods</u>

A. Principal investigator(s) and contact persons

List the staff member(s) responsible for the implementation and collection of the data. Include phone and email contact information.

List the Laboratory responsible for sample analysis. Include phone and email contact information.

B. Research objectives

Briefly describe the purpose of the monitoring program.

C. Site location and character

Describe the wetland in general and the sampling sites associated with the monitoring program. For each site include the following: latitude and longitude; mean water depth or depth range; dominant surrounding land use draining into the wetland (e.g., agriculture; urban; forested).

D. Research methods

Provide detailed information for: water sample and discharge data collection protocol(s), including the source of discharge measurements; water sample collection date/times; name of laboratory that analyzed the water samples; model numbers and maintenance of any equipment used; and calculations performed on data (e.g., equations for loading calculations).

E. Laboratory methods

List the laboratory and reference method, the method reference, a brief description of method and a brief description of the sample preservation method used for each parameter that was analyzed. For example:

a) Parameter: Total Phosphorus

Old Woman Creek NERR Laboratory Method and Reference: Total Phosphorus Test, OWC Standard Operating Procedures and Quality Control

EPA or other Reference Method: 4500-P Persulfate Digestion Method, 880 nm spec
Method Reference: Standard Methods for the Examination of Water and Wastewater
Preservation Method: Samples were stored at 4 ℃ up to 24 hours.

F. Data collection period

List the range of dates and sampling frequency for discharge measurements. Include a table that lists the date and time each water quality sample was collected at each station.

G. Data Ownership and Distribution

Specify who or what entity owns the data, if/how it can be used by others, how the data can be obtained, and how the data source should be cited.

Data Acquisition, Entry, and Verification

A. Data Acquisition and Entry

Explain how data are acquired: how water sample data are provided by the analytical laboratory (e.g., digital or hard copies); how discharge data are downloaded from website/sensor and format (e.g., .txt file, .csv file). Explain how data are entered and collated into one or more spreadsheets (e.g., entered manually or copied and pasted into Microsoft Excel). Specify if and how missing values are coded/identified.

B. Parameters Included

List any measured and calculated parameters reported and used as part of the monitoring program in table format. For example:

Parameter	***Parameter Name***	***Units***	***Measured/Calculated***
Total Phosphorus	*TP*	*mg/L*	*M*
Phosphorus loading	*TPload*	*Ton/yr*	*C*

"Parameter Name" refers to how the parameter is identified in the data set (i.e., spreadsheet).

C. Field and Laboratory QAQC programs

Describe if and how field and laboratory variability are evaluated. For example: collection of field replicates at one or more sample sites; use of a field blank (deionized water transported into the field or placed in the autosampler, then preserved, stored, and analyzed with other samples); laboratory replicates, sample spikes and standard reference material analysis (if known).

D. Limits of detection

Obtain the Method Detection Limit(s) (MDLs) for the parameters analyzed by the laboratory. List the MDL values and analysis dates for which they were in use. If possible, *include the date that each MDL was revisited by the lab for appropriateness (laboratories should do this at least annually).*

For example: Method Detection Limits (MDLs), the lowest concentration of a parameter that an analytical procedure can reliably detect, have been established by the Old Woman Creek NERR Analytical Laboratory. The MDL is determined as 3 times the standard deviation of a minimum of 7 replicates of a single low concentration sample. These values are reviewed and revised annually.

Parameter	***Start Date***	***End Date***	***MDL***	***Revisited***
TP (mg/L)	*02/07/18*	*12/13/2018*	*0.006*	*02/16/2019*

E. Data Verification

Explain how data verification (Quality Assurance / Quality Control) is performed to identify and flag suspicious data or reject erroneous data. For example, are data plotted to identify suspicious data? How values below the laboratory's reports Method Detection Limit (MDL) are treated, if the laboratory reports them (e.g., replaced with the MDL). Explain if and how any values are converted to different units of measurement. List who was responsible for these tasks.

F. QAQC flag definitions

Describe any flagging/coding included in the data set (i.e., spreadsheets) that document the quality and any factors affecting the quality of the data. Identify column headings that contain this information for each parameter (e.g., V_TP).

General flags:

-4	*Outside sensor or detection range*
-3	*Data rejected during validation*
-2	*Missing data*
0	*Data passed validation*
1	*Suspect data*
5	*Corrected data*

These general flags can be combined with more specific codes to explain the flagging or factors.

For example:

CM	*Calculated value could not be determined due to missing data*
CR	*Calculated value could not be determined due to rejected data*
DM	*Data missing or sample never collected*
RD	*Replicate values differ substantially*
BL	*Value below minimum limit of method detection*
CB	*Calculated value could not be determined due to a below MDL component*
UL	*Value above upper limit of method detection*
HB	*Sample held beyond specified holding time*
US	*Lab analysis from unpreserved sample*
AP	*Algae or scum present in sample vicinity*

IP	*Ice present in sample vicinity*
RE	*Significant rain event*
SE	*Significant seiche event*
WE	*Significant weather event*
SM	*See metadata notes*

G. Other remarks/notes

Use this section for further documentation of the data set. Include any additional notes regarding the data set in general, circumstances not covered by the flags and comment codes, or specific data that were coded with the SM "See metadata notes" comment code. Include information on major weather, precipitation, or seiche events that could have affected the data. Include any information on sample collection, data collection, or analytical equipment malfunctions or problems that did or could have compromised the quality of the data. Explain any missing data.