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State of Washington

Quality Assurance Project Plan

Prevalence and Persistence of Cyanotoxins in Lakes of the Puget Sound Basin

September 2018

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Each study conducted by the Washington State Department of Ecology (Ecology) must have an approved Quality Assurance Project Plan (QAPP). The plan describes the objectives of the study and the procedures to be followed to achieve those objectives. After completing the study, Ecology will post the final report of the study to the Internet.

This Quality Assurance Project Plan is available on Ecology's website at https://fortress.wa.gov/ecy/publications/SummaryPages/1803115.html.

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Quality Assurance Project Plan

Prevalence and Persistence of Cyanotoxins in Lakes of the Puget Sound Basin

September 2018

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Signatures are not available on the Internet version.
EAP: Environmental Assessment Program

1.0 Table of Contents

List of Figures and Tables

Page

Figures

Tables

2.0 Abstract

Cyanobacteria (blue-green algae) are a diverse group of algae with a variety of life histories and habitat niches. The most widely recognized are the planktonic (open water) species. Some species are capable of producing toxins (collectively called cyanotoxins) that are harmful to humans and wildlife. Studies by Ecology and others have shown that cyanotoxins can accumulate in the deeper sediments of lakes, as well as in the organs and tissues of fish.

Previous researchers have not examined shorelines of lakes (littoral zones) as possible locations of human exposure to cyanotoxins. In this study, we will investigate the presence and persistence of cyanotoxins in littoral sediments during and following a cyanobacterial bloom. We will also collect continuous monitoring data of a cyanobacterial pigment (phycocyanin) at one lake over the course of the summer and fall growing season.

The pigments present in cyanobacteria also accumulate in the deeper sediments of lakes. An additional goal of this project is to collect and date a sediment core from Anderson Lake in Jefferson County, which experiences annual cyanobacterial blooms. Analysis of the sediment core is an effective way to show how cyanobacterial communities have changed over time (approximately 150 years). This historical context is useful in the interpretation of the current prevalence of cyanobacterial blooms observed at this lake.

3.0 Background

3.1 Introduction and problem statement

Cyanobacteria (blue-green algae) are common in many inland waters worldwide. This diverse group of algae has a variety of life histories and habitat niches; however, the most widely recognized algae in Washington lakes are the planktonic (open water) species. Some species are capable of producing toxins (collectively called cyanotoxins) that are harmful to humans and wildlife. While it is understood which species of cyanobacteria are responsible for some specific toxins, the field of cyanotoxin research is still very active (Carmichael, 1992; Paerl et al., 2018). The drivers of cyanobacterial growth and toxin production continue to be explored in the literature; however, it is clear that temperature and nutrient enrichment play key roles (Davis et al., 2009; Van de Waal et al., 2014; Jacoby et al., 2015).

Monitoring lakes for toxic algal blooms in Washington State has relied on a collaborative and opportunistic approach.[1] The Washington State Department of Ecology (Ecology) and Washington State Department of Health (DOH) oversee and advise local partners in the sampling of suspect blooms for cyanotoxins. Ecology and DOH have also conducted studies to measure the accumulation of cyanotoxins in fish tissues (Johnson et al., 2010, 2013; Hardy et al., 2015). Findings from these studies have shown that the common toxins, microcystins, accumulate mainly in the organs (liver) and tissue (fillets) of the fish in Washington State and can be safely consumed in limited amounts.

Studies by Ecology and others have also shown that cyanotoxins can accumulate in the sediments of lakes (Babica et al., 2006; Johnson et al., 2013). This is mainly due to the deposition and accumulation of algal cells on the bottom of lakes after blooms. The shoreline areas of lakes (littoral zones) are possible locations of human and wildlife exposure to cyanotoxins. In this study, we will investigate the presence and persistence of cyanotoxins in littoral sediments during and following a cyanobacterial bloom at multiple Washington lakes.

The analysis of lake sediment cores for cyanotoxins has been used to decipher the historical prevalence of cyanobacteria through time (about the last 100 years) (Efting et al., 2011). An additional proxy of the prevalence of cyanobacteria are the pigments and carotenoids in the algal cells that are deposited on the lake bottom over time (Pal et al., 2015). This is an effective way to show how algal communities have changed over time and if there are coincident external drivers (Taranu et al., 2015). Such a historical perspective from a sediment core is not available for any of Washington's lakes that experience routine toxic algal blooms. In this study, we will collect and analyze a sediment core from Anderson Lake in Jefferson County, which experiences annual cyanobacterial blooms; this will allow us to document the history of cyanobacteria in the lake over the last 150 years.

[1] https://www.nwtoxicalgae.org

3.2 Study area and surroundings

The study area is the Puget Sound region, focusing on Jefferson, King, Kitsap, Pierce, and Thurston Counties.

3.2.1 History of study area

Algal blooms in the study region have been sampled and tested under Ecology's Freshwater Algae Control Program (FACP) since its inception in 2008. Figure 1 shows each of the lakes in the study counties where samples of suspected cyanobacterial blooms have been taken. For this study, lakes will be selected for shoreline sampling based on the presence of a bloom of toxic algae during the summer of 2018. Shoreline (littoral) sediments will be analyzed for cyanotoxins. The King County Environmental Lab (KCEL), which analyzes all samples submitted under FACP for cyanotoxins, will notify the project team when a toxic algal bloom is taking place.

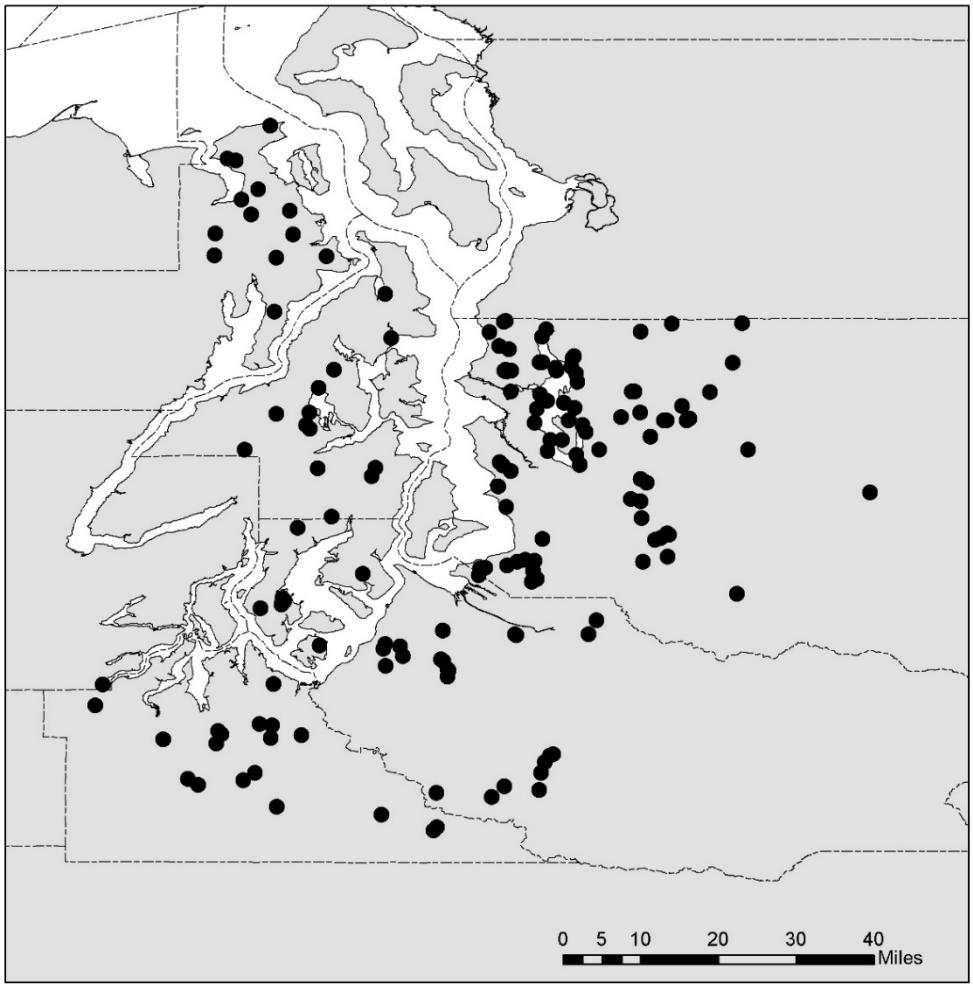

Figure 1. Map of Puget Sound region showing locations where algal blooms have been sampled.
All lakes sampled under Ecology's Freshwater Algae Control Program since 2008 are shown; the study region includes Jefferson, King, Kitsap, Pierce, and Thurston Counties.

Most of the lakes in the study region are in a lowland, urbanized setting with developed shorelines and public access. There is considerable variability in the volume of the lakes, and water quality sampling and data are sparse. Generally, counties oversee any public response to water quality concerns. Ecology has not had a statewide lake monitoring program to assist with water quality issues since the late 1990s.

Anderson Lake in Jefferson County is where we will collect a sediment core to detail the historical presence of cyanobacteria using algal pigments. Anderson Lake is situated in a state park surrounded by cedar, fir, and alder. The park was established in 1966 through land acquisition. The lake is 25 ha (60 acres) in area and has maximum and mean depth of 7.6 m (25 ft) and 3.7 m (12 ft) respectively (Figure 2). There is a large marsh at the south end of the lake. The lake's submerged plant community was assessed in 1996 and 2017 by Ecology. Common waterweed and pond-lilies dominated the communities in 1996, while in 2017 plants were dominated by bulrush, bur-reed, pondweeds, and the noxious weed - reed canary grass.

Anderson Lake is stocked by the Washington Department of Fish and Wildlife with rainbow trout; however, since 2006 toxic algal blooms have closed the lake to all recreational activity for part of each year, usually from May through September. The blooms are dominated by the cyanobacteria genus *Dolichospermum*. However, the particular species responsible for the cyanotoxin has a genetic structure that allows it to produce much higher toxin concentrations than more common *Dolichospermum* species (e.g., *Dolichospermum flos-aquae*) (Brown et al., 2016).

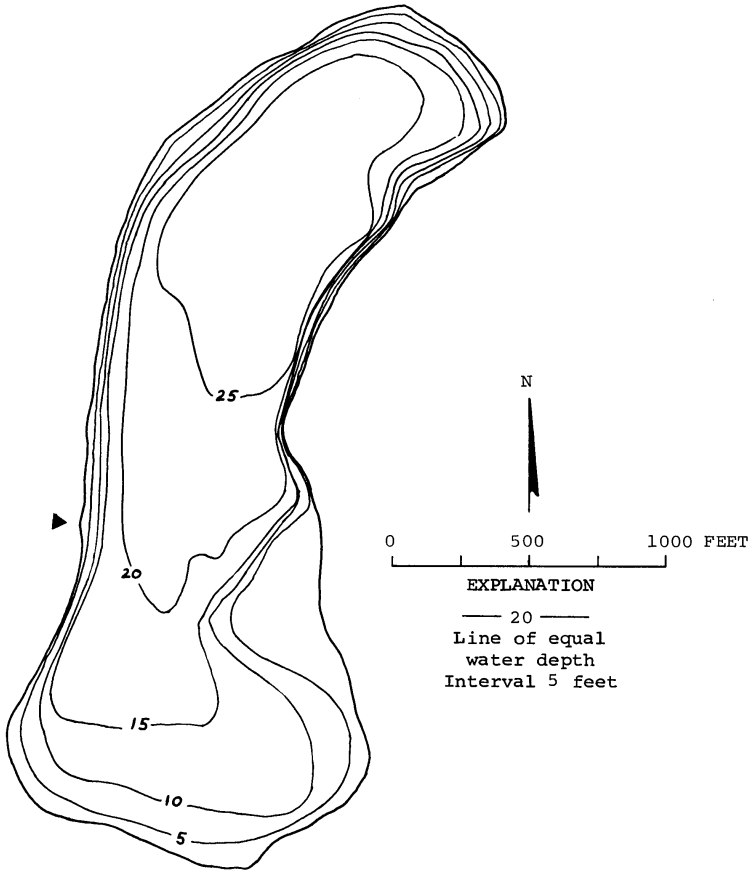

Figure 2**.** Bathymetric map of Anderson Lake.
The triangle indicates the boat launch and public access.

3.2.2 Parameters of interest and potential sources

There are numerous cyanotoxins in freshwaters, produced by a variety of algae species. Cyanotoxins are produced by the algae as secondary metabolites, meaning they are produced for a secondary function, such as protection of the organism (Carmichael, 1992). They are released from the cell into the water following cell senescence or death. In this project we are interested in the most common freshwater cyanotoxins, microcystins. Microcystins (MCs) are a hepatotoxin, affecting liver function. Acute and chronic MC exposures can cause death or permanent liver damage (Nishiwaki-Matsushima et al., 1992) and have been implicated in hepatocarcinoma (Grosse et al., 2006). Signs of toxin poisoning through ingestion include vomiting, abdominal pain, weakness, severe thirst, and death. Dogs and wildlife are particularly susceptible to cyanotoxin poisoning because of direct ingestion of lake water and the volume ingested relative to body mass (Backer et al., 2013). In addition to ingestion of contaminated water, dermal exposure or inhalation of cyanotoxins can cause skin rashes, allergic reactions, and respiratory complications (Drobac et al., 2013).

Microcystin refers to a group of compounds or variants of the general structure of MCs (Figure 3). There are approximately 150 known structural variations of the MC compound, and the variation is attributable to the positions of amino acids in the compound (Foss and Aubel, 2015). A number of the MC variants have been resolved using liquid chromatography - tandem mass spectrometry (LC MS/MS) (Mekebri et al., 2009). The most dominant variants can make up the majority of the total MC in a sample (Foss and Aubel, 2015). The MC variants also vary in toxicity and stability. Based on their stability, occurrence, and our ability to detect the variants reliably, we are interested in analyzing for those listed in Table 1.

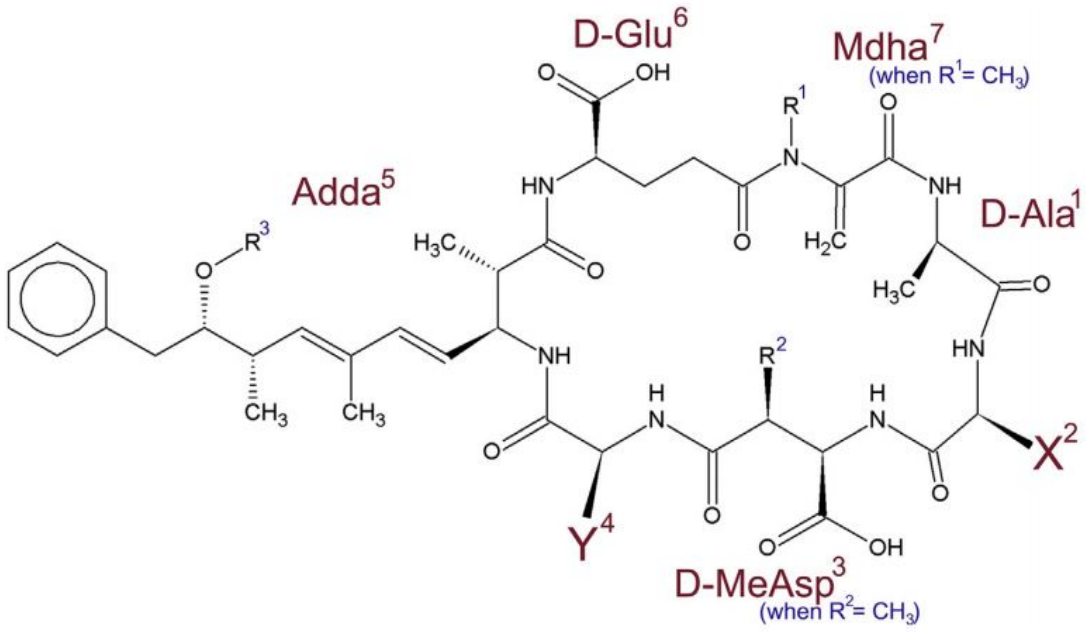

Figure 3. General structure of microcystin (Foss and Aubel, 2015).
Amino acids: D-Glu[6] (Glutamic acid); Mdha[7] (methyl-dehydroalanine); D-Ala[1] (Alanine); D-MeAsp3 (D-erythro-β-methylaspartic acid); X and Y are variable amino acids; Adda[5] [(2S,3S,4E,6E,8S,9S)-3-amino-9-methoxy-2,6,8trimethyl-10-phenyldeca-4,6-dienoic acid].

Table 1. List of microcystin variants of interest.

Microcystin congener	Amino acid in X	Amino acid in Y
Microcystin-LR	leucine	arginine
Microcystin-RR	arginine	arginine
Microcystin-YR	tyrosine	arginine
Microcystin-LA	leucine	alanine
Microcystin-LY	leucine	tyrosine
Microcystin-LF	leucine	phenylalanine
Microcystin-LW	leucine	tryptophan

In addition to MC, the cyanotoxin known as anatoxin-a is of interest in Anderson Lake, Jefferson County. Anatoxin-a is a potent neurotoxin, and responses to it can include seizures, gasping, muscle fasciculation (twitches), staggering, backward arching of neck in birds, and death (Carmichael, 1992). In Anderson Lake, anatoxin-a is produced by a *Dolichospermum* species, and also by *Aphanizomenon* species. In order to infer relative success of these cyanobacteria genera over time, we will be identifying the algal pigments and carotenoids specific to each of them. Like cyanotoxins, algal pigments and carotenoids are also a secondary metabolite produced by the organism (Leavitt and Hodgson, 2001). The use of algal pigments as biomarkers in lake sediment cores is well established and can be used to identify the presence and semi quantitative abundance of many different algal groups (Table 2). We will not be analyzing the sediment core directly for anatoxin-a because this compound is very labile and breaks down in water, unlike microcystins, which are very stable (Rapala et al., 1994).

In addition to measuring algal pigments in lake sediments, we are interested in taking continuous measurements from a single lake for the pigments chlorophyll *a* and phycocyanin. Chlorophyll *a* is the photosynthetic pigment in algae; the concentration of this pigment in the water can be used as a measurement of algal production. Phycocyanin is the main group of pigments in cyanobacteria, and their concentration in the water is indicative of cyanobacterial production. Continuous measurements will be taken over the growing season from June to October at a lake known to experience annual cyanobacterial blooms.

Table 2. Summary of algal pigments found in lake sediments and their taxonomic affinities (from Leavitt and Hodgson, 2001).

Pigment	Source[†]	Stability[‡]	Affinity
β,β-carotene	P, L, t	1	*Plantae, Algae*, some phototrophic bacteria
β,α-carotene	P	2	*Cryptophyta, Chrysophyta, Dinophyta*, some *Chlorophyta*
β-isorenieratene	P	1	*Chlorobiaceae* (green sulfur bacteria)
isorenieratene	P	1	*Chlorobiaceae* (brown varieties)
alloxanthin	P	1	*Cryptophyta*
fucoxanthin	P, L	2	*Dinophyta, Bacillariophyta, Chrysophyta*
diatoxanthin	P, L, s	2	*Bacillariophyta, Dinophyta, Chrysophyta*
diadinoxanthin	P, L, s	3	*Dinophyta, Bacillariophyta, Chrysophyta, Cryptophyta*
dinoxanthin	P	unkn.	*Dinophyta*
peridinin	P	4	*Dinophyta*
echinenone	P	1	*Cyanobacteria*
zeaxanthin	P	1	*Cyanobacteria*
canthaxanthin	P	1	colonial *Cyanobacteria*, herbivore tissues
myxoxanthophyll	P	2	colonial *Cyanobacteria*
scytonemin	p, L	unkn.	colonial *Cyanobacteria*
oscillaxanthin	P	2	*Cyanobacteria (Oscillatoriaceae)*
aphanizophyll	P	2	nitrogen-fixing *Cyanobacteria* (Nostocales)
lutein	P, L, t	1	*Chlorophyta, Euglenophyta, Plantae*
neoxanthin	P, L, t	4	*Chlorophyta, Euglenophyta, Plantae*
violaxanthin	P, L, t	4	*Chlorophyta, Euglenophyta, Plantae*
okenone	P	1	purple sulfur bacteria
astaxanthin	P	4	invertebrates, nitrogen-limited *Chlorophyta*
chlorophyll *a*	P, L	3	*Plantae, Algae*
chlorophyll *b*	P, L	2	*Plantae, Chlorophyta, Euglenophyta*
pheophytin *a*	P, L, t, s	1	chlorophyll *a* derivative (general)
pheophytin *b*	P, L, t, s	2	chlorophyll *b* derivative (general)
pheophorbide *a*	P, s	3	chlorophyll *a* derivative (grazing, senescent diatoms)
pyro-pheo (pigments)	L, S	2	derivatives of *a*- and *b*-phorbins
chlorophyll *c*	P	4	*Dinophyta, Bacillariophyta, Chrysophyta*

[†] P = planktonic, L = littoral, T = terrestrial, S = sedimentary or post-depositional derivative; upper case = more important source.
[‡] Ranked 1 (most) to 4 (least) for chemical stability and preservation in sediments.

In addition to the cyanotoxins and algal pigments, we are interested in assessing nutrient concentrations of the water at the time of sampling. Nutrients are one of the main drivers of algal blooms, with taxa requirements and nutrient availability in the water influencing the types of cyanobacteria present (Dedmer et al., 2014). We are interested in total phosphorus (TP) and total nitrogen (TN) concentrations as well as the dissolved inorganic nitrogen (nitrite-nitrate [NO_2–NO_3] and ammonia [NH_3]). The ratios of nutrients can be used to infer growth limitations (Bergström, 2010; Jacoby et al., 2015).

3.2.3 Summary of previous studies and existing data

We will select lakes for littoral sediment sampling based on the opportunistic water sampling of blooms in 2018 by Ecology's FACP. In 2017, 788 samples of suspected cyanobacterial blooms were submitted under the FACP (Table 3). The samples in 2017 came from 269 locations with a suspected toxic algal bloom. We are interested in lakes where the concentration of MC in the water is at least ten times the DOH recreational guideline (guideline = 6 µg/L). In 2017, 11 samples were above 60 µg/L total MC (Figure 4), with seven of these from lakes in King, Pierce, or Thurston counties.

Table 3. Number of water samples taken for microcystin analysis in Washington State (2007– 2017) and the number of samples above the Washington State Department of Health recreational guideline for human health (6 µg/L).

Year	Number of samples	Number of samples above guideline	Percentage of samples above guideline
2007	75	19	25%
2008	285	88	31%
2009	1042	115	11%
2010	1014	84	8%
2011	1121	96	9%
2012	891	77	9%
2013	811	93	11%
2014	823	101	12%
2015	995	153	15%
2016	776	44	6%
2017	788	36	5%

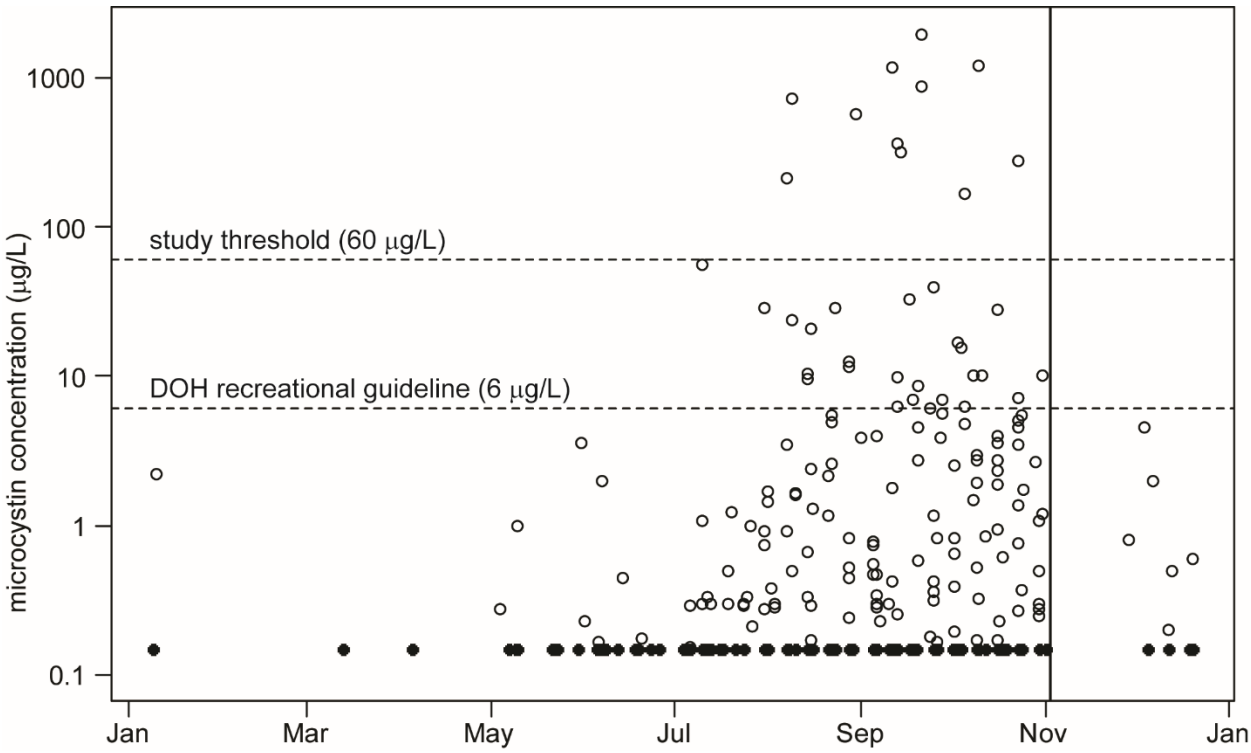

Figure 4. Microcystin results for 2017 for Washington State.
Open circles represent detected microcystin concentrations of lake water samples. Black circles are samples with concentrations below the method detection limit; horizontal dashed lines are the threshold for Washington State Department of Health (DOH) recreational advisories and the threshold of interest in this study; the vertical solid line represents the end of the 2017 budget for the Freshwater Algae Control Program, not the end of cyanobacterial blooms for 2017.

Ecology has previously conducted sampling of lake sediments at locations where blooms have been common in the Puget Sound region. Detectable concentrations of some microcystin variants were measured in all the samples (Table 4; Johnson et al., 2013). The focus of the Johnson et al. (2013) study was the persistence of MC variants in the deeper sediments of the lake, months after fall cyanobacterial blooms. The focus was not on direct human exposure of cyanotoxins from the sediments. Nevertheless, the work of Johnson et al. (2013) suggests that the variants MC-LR and MC-LA might be the dominant variants in sediments. Other studies have also found that these variants and MC-RR are dominant in the sediments that accumulate at the bottom of lakes, with some diffusion of these variants from sediment porewaters back into the bottom waters of the lake (Zastepa et al., 2015; 2017).

Table 4: Previous results of microcystin variants[a] in sediments (Johnson et al., 2013).

Lake	Date	Lab ID	Depth (m)	Desmethyl-LR	Desmethyl-RR	LA	LF	LR	LW	LY	RR	YR
Bay	3/23/2011	L-136-11-5	3.7	*nd*	*nd*	**51.7**	*nd*	*nd*	*nd*	*nd*	*nd*	*nd*
	3/23/2011	L-136-11-6	1.5	*nd*	*nd*	**4.6**	*nd*	**2.98**	*nd*	*nd*	*nd*	*nd*
Waughop	3/23/2011	L-136-11-9	4.6	*nd*	*nd*	**17.8**	*nd*	**9.59**	*nd*	*nd*	*nd*	*nd*
	3/23/2011	L-136-11-10	4.0	*nd*	*nd*	**14.6**	*nd*	**6.01**	*nd*	*nd*	*nd*	*nd*
Spanaway	3/23/2011	L-136-11-11	8.5	*nd*	*nd*	**10.6**	*nd*	*nd*	*nd*	*nd*	*nd*	*nd*
	3/23/2011	L-136-11-12	6.4	*nd*	*nd*	*nd*	*nd*	*nd*	*nd*	*nd*	*nd*	*nd*
Anderson	3/25/2011	L-136-11-7	7.3	*nd*	*nd*	**10.9**	*nd*	**30.4**	*nd*	*nd*	*nd*	*nd*
	3/25/2011	L-136-11-8	3.0	*nd*	*nd*	*nd*	*nd*	**2.91**	*nd*	*nd*	*nd*	*nd*
Ketchum	3/29/2011	L-136-11-1	6.4	*nd*	*nd*	*nd*	*nd*	**42**	*nd*	*nd*	*nd*	*nd*
	3/29/2011	L-136-11-2	1.5	*nd*	*nd*	*nd*	*nd*	*nd*	*nd*	*nd*	*nd*	*nd*
Cassidy	3/29/2011	L-136-11-3	6.7	*nd*	*nd*	**30.5**	*nd*	**15.4**	*nd*	*nd*	*nd*	*nd*
	3/29/2011	L-136-11-4	2.1	*nd*	*nd*	**28.5**	*nd*	*nd*	*nd*	*nd*	*nd*	*nd*

[a] See Table 1 for microcystin variant abbreviations
nd = non-detect
Desmethyl is a derivative of the original microcystin variant.

3.2.4 Regulatory criteria or standards

Ecology does not have surface water criteria for cyanotoxins. In the absence of federal recreational guidance values, DOH derived interim recreational guidelines in surface waters for total microcystins (Table 5). A number of other states and the United States Environmental Protection Agency (EPA) also have recommended guidelines for recreational activity. Washington's guideline is based on a tolerable daily intake of 0.04 µg/kg/day for a child of 15 kg at a rate of intake of 0.05 L/hr and assuming 2 hr/day exposure (Hardy, 2008). When national recreational guidance values are adopted, Washington will likely adopt the federal approach. Generally, recreational guidelines are used to trigger a plan or framework for managing public exposure to potentially harmful algal blooms.

Table 5: Recreational guidelines for microcystin in freshwater.

	Washington (Hardy, 2008)	Oregon (OPHD, 2016)	EPA Draft (2016)
Total microcystin (µg/L)	6	4	4

There are numeric benchmarks for nutrients in Washington surface waters that are used to trigger a lake-specific study for nutrients (WAC 173-201A). The action value for Puget Sound lowland lakes is 20 µg/L of total phosphorus, as measured in the epilimnetic (surface) waters of a lake over the summer (average of at least four samples from June through September in one or more consecutive years).

4.0 Project Description

4.1 Project goals

The goals of this project are as follows:

1. Determine the prevalence and persistence of cyanotoxins (MC variants) in littoral (shoreline) sediments and porewaters of lakes in the Puget Sound region.
2. Establish the trends of chlorophyll *a* and phycocyanin over the summer and early fall at a lake in the Puget Sound Region.
3. Determine the historic prevalence of cyanobacteria in Anderson Lake, Jefferson County, using a sediment core representing approximately the last 150 years.

4.2 Project objectives

The objectives of this study include the following:

- Assess the concentrations of MC variants and nutrients in the nearshore waters of seven lakes in the Puget Sound region.
- Assess the prevalence of MC variants in the littoral sediments at seven lakes in the Puget Sound region.
- Assess the persistence and attenuation of MC variants in littoral sediments at two lakes.
- Continuously assess the prevalence of cyanobacterial pigments at one lake over the summer.
- Assess the historic prevalence of cyanobacterial pigments in sediments of Anderson Lake, Jefferson County.

4.3 Information needed and sources

This project is being conducted to generate new environmental data sets.

4.4 Tasks required

Specific tasks under this project include the following:

- Coordinate with KCEL for notification of lakes in the Puget Sound region experiencing a cyanobacterial bloom with concentrations of microcystin over 60 µg/L.
- Collect surface water, sediment, and porewaters from seven lakes in Puget Sound while a cyanobacterial bloom is occurring.
- At two of the lakes return and sample the same shoreline sediments at two, four, and seven days following the bloom.
- Deploy a multiprobe sonde at a lake known to experience annual cyanobacterial blooms and take continuous measurements of chlorophyll *a*, phycocyanin, dissolved oxygen (DO), pH, temperature, and conductivity over the summer.
- Collect a sediment core from Anderson Lake in Jefferson County.
- Subsample the core at Ecology for dating, geochemical analysis, and sedimentary algal pigments.
- Construct an age–depth model for the sediment core.

- Review and assess data quality and laboratory results.
- Work with project scientists to write a draft manuscript on the prevalence and persistence of MC variants in lake littoral sediments.
- Write a report documenting the historical prevalence of cyanobacteria in Anderson Lake.
- Give presentations on the findings to Ecology's Water Quality Program and EPA.

4.5 Systematic planning process used

This QAPP represents the systematic planning for the project.

5.0 Organization and Schedule

5.1 Key individuals and their responsibilities

Table 6. Organization of project staff and responsibilities.[a]

Staff	Title	Responsibilities
Jessica Archer SCS, EAP Phone: 360-407-6698	EAP Client and Section Manager for the Project Manager	Clarifies scope of the project. Provides internal review of the QAPP and approves the final QAPP.
William Hobbs, PhD TSU, SCS Phone: 360-407-7512	Project Manager	Writes the QAPP. Oversees field sampling and transportation of samples to the laboratory. Conducts QA review of data, analyzes and interprets data, and enters data into EIM. Manages IAA and contracts with and receives analytical results from labs other than MEL (see Section 9.4). Writes the draft report and final report.
Siana Wong TSU, SCS Phone: 360-407-6432	Project Scientist	Helps collect samples and records field information. Analyzes data. Enters data into EIM.
Debby Sargeant TSU, SCS Phone: 360-407-6775	Unit Supervisor for the Project Manager	Provides internal review of the QAPP, approves the budget, and approves the final QAPP.
Joan Hardy, PhD Phone: 206-947-7400	Project Scientist	Provides regional expertise on cyanobacteria. Collaborator on study design, fieldwork, data analysis, and reporting.
Ellen Preece, PhD Robertson-Bryan Inc. Phone: 916-405-8919	Project Scientist	Provides regional expertise on cyanobacteria. Collaborator on study design, fieldwork, data analysis, and reporting.
Alan Rue Manchester Environmental Laboratory Phone: 360-871-8801	Director	Reviews and approves the final QAPP. Analyzes water samples for supplemental nutrient parameters.
Francis Sweeney King County Environmental Lab Phone: 206-477-7117	Director, Aquatic Toxicology	Reviews draft QAPP, coordinates with Project Manager. Analyzes water and sediment samples for microcystins.
Tom Gries Phone: 360-407-6327	Acting Ecology Quality Assurance Officer	Reviews the draft QAPP and approves the final QAPP. May comment on the final report.

[a] EAP: Environmental Assessment Program; EIM: Environmental Information Management database; QAPP: Quality Assurance Project Plan; SCS: Statewide Coordination Section; TSU: Toxic Studies Unit

5.2 Special training and certifications

Knowledge of calibrating and programming the YSI Exo3 sonde will be required for the project. Siana Wong, Project Scientist, has worked with this sonde during an extensive lake monitoring survey in 2017.

5.3 Organization chart

Not Applicable—See Table 6

5.4 Proposed project schedule

Table 7. Proposed schedule for completing field and laboratory work, data entry into the Environmental Information Management (EIM) database, and reports.

Field and laboratory work	Due date	Lead staff
Field work completed	November 2018	William Hobbs
Laboratory analyses completed	February 2019	
Environmental Information Management (EIM) database		
EIM Study ID	WHOB008	
Product	Due date	Lead staff
EIM data loaded	April 2019	Siana Wong
EIM data entry review	May 2019	William Hobbs
EIM complete	June 2019	Siana Wong
Final report		
Author lead / support staff	William Hobbs / Siana Wong	
Schedule		
Draft due to supervisor	June 2019	
Draft due to client/peer reviewer	July 2019	
Final (all reviews done) due to publications coordinator	August 2019	
Final report due on web	September 2019	

5.5 Budget and funding

The source of funding for this project is from EPA Agreement I-01J18701 and the State Toxics Control Fund. The detailed budget for the laboratory expenses (contractual) is outlined in Table 8, and the total project budget is summarized in Table 9.

Table 8. Detailed project budget and funding.

	Number of samples	Number of QA samples	Cost per sample ($)	In-house cost per sample ($)	Contract ($)	Subtotal ($)
Sediment core						
C:N & isotopes	20	5	15	–	375	375
pigments	20	–	125	–	2,500	2,500
radioisotopes	18	2	150	–	3,000	3,000
			Total	0	$5,875	$5,875
MC variants sampling event						
grain size	3	–	100	–	300	300
C:N & isotopes	3	6	15	–	135	135
Chlorophyll *a*	1	–	50	50	–	50
Total nitrogen	1	–	20	20	–	20
Nitrite-nitrate as N and ammonia as N	1	–	30	30	–	30
Total phosphorus	1	–	25	25	–	25
MC in water	3	–	175	–	525	525
MC in sediments	3	–	235	–	705	705
MC in porewaters	3	–	175	–	525	525
		Totals per sampling event		$125	$2,190	$2,315
MC variants attenuation sampling event						
C:N & isotopes	3	3	15	–	90	90
MC in water	3	–	175	–	525	525
MC in sediments	3	–	235	–	705	705
MC in porewaters	3	–	175	–	525	525
		Totals per sampling event		0	$1,845	$1,845

Table 9: Project budget summary.

Budget category	EPA grant ($)	State Toxics Control Fund ($)	Totals ($)
Salary	13,942	0	13,942
Benefits	5,254	0	5,254
Travel	983	0	983
Equipment	0	0	0
Supplies	5,800	0	5,800
Contractual	15,000	22,852	37,852
Construction	0	0	0
Other	0	0	0
Subtotals (total direct charges)	40,980	22,852	63,832
Indirect charges (29.35% of salary and benefits)	5,634	0	5,634
Totals	$46,614	$22,852	$69,466

6.0 Quality Objectives

6.1 Data quality objectives

All sampling will be carried out according to established standardized operating procedures (SOPs), and we do not foresee needing any data quality objectives (DQOs).

6.2 Measurement quality objectives

The measurement quality objectives (MQOs) for the analytical data in this study are detailed in Table 10. The MQOs for the field parameters (chlorophyll *a*, phycocyanin, pH, DO, temperature, and conductivity) are in Table 11.

6.2.1 Targets for precision, bias, and sensitivity

6.2.1.1 Precision

Precision is a measure of the variability in the results of replicate measurements due to random error. Precision for two replicate samples is measured as the relative percent difference (RPD) between the two results. If there are more than two replicate samples, then precision is measured as the relative standard deviation (RSD).

MQOs for the precision of laboratory duplicate samples and matrix spike duplicate samples are shown in Table 10.

6.2.1.2 Bias

Bias is the difference between the population mean and the true value. For this project, bias is measured as acceptable percent recovery. Acceptance limits for laboratory verification standards, matrix spikes, and surrogate standards are shown in Table 10.

6.2.1.3 Sensitivity

Sensitivity is a measure of the capability of a method to detect a substance above the background noise of the analytical system. The laboratory reporting limits for the project are described in Section 9.2.

Table 10: Measurement quality objectives.

Parameter[a]	Verification standards (LCS, CRM, CCV[b]) (% recovery limits)	Spiked blank (% recovery limits)	Duplicate samples (RPD[c])	Matrix spikes (% recovery limits)	Matrix spike duplicates (RPD[c])	Lowest concentrations of interest
MC-LR, MC-LA, MC-LF, MC-LY MC-YR, NOD in water and sediment; MC-RR and MC-LW in water	CCV low: 50–150 CCV mid: 70–130 CCV high:70–130	70–130	40	70–130	40	0.2 µg/L
MC-RR, MC-LW in sediment	Same as above	50–130	50	50–130	50	0.8 ng/g
Chlorophyll *a*	CCV 90–110	NA	20	NA	NA	0.004 mg/L
TP	CCV 90–110	80–120	20	75–125	20	0.0024 mg/L
NO_2–NO_3	CCV 90–110	80–120	20	75–125	20	0.01 mg/L
NH_3	CCV 90–110	80–120	20	75–125	20	0.01 mg/L
Total persulfate N	CCV 90–110	80–120	20	75–125	20	0.025 mg/L
Grain size	NA	NA	≤10	NA	NA	0.1%
Loss-on-ignition	NA	NA	≤20	NA	NA	1.0%
TOC:TN	80–120	NA	≤20	NA	NA	1%
^{210}Pb radioisotopes	80–120	NA	≤30	NA	NA	0.45 pCi/g
algal pigments	80–120	NA	≤30	80–120	NA	0.1 nmole

[a] See Table 1 for microcystin variant abbreviations; see Appendix A for remaining parameters; NOD= Nodularin
[b] LCS = laboratory control sample; CRM = certified reference materials; CCV = continuing calibration verification standard.
[c] RPD = relative percent difference.

Table 11. Measurement quality objectives for YSI Exo3 sonde calibration checks.

Parameter	Units	Accept	Qualify	Reject
Chlorophyll *a*	RFU[a]	< or = ± 1.0	> ± 1.0 and < or = ± 2.0	> ± 2.0
Phycocyanin	RFU[a]	< or = ± 1.0	> ± 1.0 and < or = ± 2.0	> ± 2.0
pH	std. units	< or = ± 0.2	> ± 0.2 and < or = ± 0.8	> ± 0.8
Conductivity*	µS/cm	< or = ± 5	> ± 5 and < or = ± 15	> ± 15
Temperature	°C	< or = ± 0.2	> ± 0.2 and < or = ± 0.8	> ± 0.8
Dissolved oxygen	% saturation	< or = ± 5%	> ± 5% and < or = ± 15%	> ± 15%
Dissolved oxygen	mg/L	< or = ± 0.3	> ± 0.3 and < or = ± 0.8	> ± 0.8

[a] RFU = relative fluorescence unit

* Criteria expressed as a percentage of readings; for example, buffer = 100.2 µS/cm and reading = 98.7 µS/cm, so (100.2–98.7)/100.2 = 1.49% variation, which falls into the acceptable data criteria of less than 5%.

6.2.2 Targets for comparability, representativeness, and completeness

6.2.2.1 Comparability

Section 8.1 lists the SOPs to be followed for field sampling. In addition, the lab analyzing the samples for MC variants is consistent with previous Ecology studies (e.g., Johnson et al., 2013).

6.2.2.2 Representativeness

Representativeness is a measure of whether the sample media reflects the current environmental conditions. We will ensure proper representatives by adhering to the approved SOPs and sampling protocols. Samples will be preserved and stored to ensure that lab holding conditions and times are met.

6.2.2.3 Completeness

The data for this project will be considered complete if 95% of the planned samples were collected and analyzed acceptably.

7.0 Study Design

7.1 Study boundaries

The geographic area of interest encompasses lakes in the Puget Sound region within the counties of Jefferson, King, Kitsap, Pierce, and Thurston (Figure 1). Specific lakes for inclusion in this study will not be selected until Ecology receives initial cyanotoxin data from KCEL as part of the opportunistic sampling under the FACP at Ecology and DOH. The exception is Anderson Lake in Jefferson Co., which will be the location of the sediment core.

7.2 Field data collection

7.2.1 Sampling locations and frequency

Littoral Sediments

Once Ecology receives notice that a lake in the study region has a cyanobacterial bloom with MC concentrations over 60 µg/L, we will sample the nearshore waters and littoral sediments (including the porewaters) the following day during the bloom. Upon arriving at the lake in the same location sampled the day before by the county, we will collect a surface water grab sample of the nearshore waters for nutrients and MC variants. The nearshore waters will be sampled in order to characterize the MC variants in the water. All water samples will be stored on ice and submitted to the analytical lab the next day.

The littoral sediments will be sampled at three locations in the vicinity of the nearshore water samples using a sediment corer. A sediment corer will be used in order to keep the porewaters intact. The water overlying the sediment will be removed and the upper 2–3 cm of sediment will be collected. At each location enough sediment will be collected for measuring grain size, MC variants, total organic carbon and nitrogen abundance (TOC:TN), and stable isotope ratios. Samples will be stored on ice and brought back to Ecology for processing. Sediment samples for MC variants will be centrifuged to isolate the porewaters. Porewaters will then be extracted and submitted as a separate sample for MC variants.

At two of the lakes where high concentrations of total MCs are found, we will return to the lakes and sample the littoral sediments 2, 4, and 7 days after the first sample to look for changes in the MC variants. Nearshore waters will be collected for MC variants and littoral sediments and porewaters will be collected for MC variants and TOC:TN.

Sediment Core

At Anderson Lake, a sediment core will be collected from the deepest area of the lake (Figure 2). The Toxics Studies Unit of Ecology's Environmental Assessment Program (EAP) has a well-established program focused on the use of sediment cores to inform the long-term trends of persistent, bioaccumulative, and toxic chemical deposition to Washington lakes. The coring of Anderson Lake for this project will follow the same approaches and methods described in the QAPP for the sediment core program (Mathieu, 2016).

The sediment core will be transported back to the lab at Ecology for subsampling. During transportation the core will be secured vertically with intact overlying water to ensure the preservation of the sediment-water interface, wrapped in tinfoil to avoid any photo-degradation, and packed on ice.

Continuous Monitoring

At a lake in the Puget Sound region that regularly experiences cyanobacterial blooms each year, we will deploy a multi-parameter sonde for continuous measurement of cyanobacterial pigment (phycocyanin). Through local lake associations and county contacts, Dr. Joan Hardy will locate a volunteer who will allow Ecology to install the sonde for continuous measurement over the summer on their property. This approach will ensure the security of the monitoring equipment, which could be jeopardized at a public beach. The sonde will be cleaned and the calibration verified every 2 weeks at the start of the project and then as needed over the course of the summer. Following calibration verification, a grab sample will be taken for chlorophyll *a* to assess the compatibility between measurements from the sonde and laboratory.

7.2.2 Field parameters and laboratory analytes to be measured

A sonde will be deployed at one lake to continuously measure pH, DO, conductivity, temperature, chlorophyll *a*, and phycocyanin.

At the time of sampling littoral sediments, a multi-parameter sonde will be used to measure pH, DO, conductivity, and temperature. Analytes to be measured in the littoral sediments include MC variants, grain size, TOC:TN, and stable isotopes. Littoral sediment porewaters will be analyzed for MC variants. Nearshore surface waters, proximal to the littoral sediments, will be sampled for MC variants, total nitrogen, total phosphorus, nitrite-nitrate, ammonia, and chlorophyll *a*.

Analytes to be measured on the sediment core include algal pigments, ^{210}Pb radioisotopes, TOC:TN, and stable isotopes.

7.4 Assumptions in relation to objectives and study area

To test whether the MC variants persist in lake sediments, our sample design makes the assumption that the cyanobacterial bloom in the lake will attenuate over time in the water. In other words, should the bloom in the lake water continue day after day, we will not have the opportunity to test whether the MC variants from the original bloom persist in the littoral sediments.

To address the above assumption, we will work with the local county health authority to continue sampling the bloom for cyanotoxins under FACP. We will then adjust our schedule of littoral sediments sampling to capture the decline of the bloom.

7.5 Possible challenges and contingencies

7.5.1 Logistical problems

Logistical issues with sampling MC variants in the littoral sediments may include problems with connecting and collaborating with the local county health department to gain access or permission to sample a lake following a high result for MC concentrations. To alleviate this, key people in the potential counties will be contacted ahead of time to confirm approval, should a lake in their jurisdiction have a bloom with high MC concentrations.

7.5.2 Practical constraints

Cyanobacterial blooms are difficult to predict. There may not be a sufficient number of blooms in Puget Sound lakes that reach our desired 60 µg/L threshold (seven lakes). If by late-August we are having difficulty getting enough sample lakes, we will lower the threshold to the DOH guidance level of 6 µg/L.

There are no foreseeable issues with the collection of the sediment core from Anderson Lake.

7.5.3 Schedule limitations

Limitations on the project schedule are budget and QAPP review. The EPA grant for this project closes on December 31, 2018; all samples must be collected and analyzed by this date. Some lakes in the Puget Sound region are already reporting and sampling cyanobacterial blooms. The window for continuous measurements of phycocyanin and other parameters is already open. A form for approval to sample will accompany this QAPP, allowing us to begin the continuous measurements as soon as possible.

8.0 Field Procedures

8.1 Invasive species evaluation

Field personnel for this project are required to be familiar with and follow the procedures described in SOP EAP070, *Minimizing the Spread of Invasive Species* (Parsons et al., 2018). Our study areas are not considered to be of high concern for invasive species. Sampling events will be day trips, with sufficient time in between to allow for decontamination by drying (48 hours).

8.2 Measurement and sampling procedures

A number of established SOPs will be followed during sampling, including:

- EAP015 – *Manually Obtaining Surface Water Samples, Version 1.2* (Joy, 2013).
- EAP033 – *Hydrolab® DataSonde® and MiniSonde® Multiprobes, Version 1.0* (Swanson, 2007).
- EAP038 - *Collection of Freshwater Sediment Core Samples Using a Box or KB Corer* (Furl and Meredith, 2008).
- EAP040 – *Standard Operating Procedure for Obtaining Freshwater Sediment Samples* (Blakley, 2008).

8.3 Containers, preservation methods, holding times

Table 12. Sample containers, preservation, and holding times.

Parameter	Matrix	Minimum quantity required	Container	Preservative	Holding time
MC variants	water	100 ml	125 ml amber glass bottle	cool at 4°C or freeze	48 hrs (1 month frozen)
MC variants	porewaters	20 ml	20 ml amber glass vial	cool at 4°C or freeze	48 hrs (1 month frozen)
MC variants	sediments	100 g ww	4 oz amber glass jar	cool at 4°C or freeze	48 hrs (1 month frozen)
Chlorophyll *a*	water	0.25–1 L, filtered	field filter in glass tube	acetone	30 day
TP	water	60 ml	125 ml clear nalgene	HCl	28 day
NO_2–NO_3	water	60 ml	125 ml amber nalgene	H_2SO_4	28 day
NH_3	water	60 ml	125 ml clear nalgene	H_2SO_4	28 day
Total persulfate N	water	60 ml	125 ml clear nalgene	H_2SO_4	28 day
Grain size	sediment	150 g ww	8 oz HDPE jar	cool at 4°C	6 months
Loss-on-ignition	sediment	2 g ww	black 1 oz HDPE vials	cool at 4°C	6 months
TOC:TN	sediment	20 mg dw	black 1 oz HDPE vials	lyophilization	frozen 1 year
^{210}Pb radioisotopes	sediment	1.0 g dw	2 oz glass jar	lyophilization	NA
algal pigments	sediment	0.2 g dw	black 1 oz HDPE vials	lyophilization and darkness	6 months

8.4 Equipment decontamination

All sample equipment will be rinsed at EAP's operations center following use in the field. Equipment will be rinsed with site water in between samples. Decontamination protocol will also follow section *8.1, Invasive species evaluation.*

8.5 Sample ID

Laboratory sample identifications will be assigned by KCEL or MEL.

8.6 Chain of custody

Chain of custody will be recorded for all samples throughout the project.

8.7 Field log requirements

Field data will be recorded in a bound, waterproof notebook on waterproof paper. Corrections will be made with single line strikethroughs, initials, and date.

The following information will be recorded in the project field log:

- Name and location of project
- Field personnel
- Sequence of events
- Any changes or deviations from the QAPP
- Environmental conditions
- Date, time, location, identification, and description of each sample
- Field instrument calibration procedures
- Field measurement results
- Identity of quality control (QC) samples collected
- Unusual circumstances that might affect interpretation of results

8.8 Other activities

As described earlier in this QAPP, the KCEL will notify Ecology when a cyanobacterial bloom is taking place. There are number of activities needed for this communication from KCEL to be effective:

- Establish a communication plan with Francis Sweeney and Elizabeth Frame of KCEL.
- Establish points of contact with Jefferson, King, Kitsap, Pierce, and Thurston Counties.
- Organize appropriate sample containers and coolers with KCEL.
- Have an Ecology field crew ready to deploy to a lake experiencing a bloom.

In addition, the appropriate permissions will need to be confirmed with Jefferson County Public Health or Washington State Parks to recover a sediment core from Anderson Lake.

9.0 Laboratory Procedures

9.1 Lab and field procedures table

Table 13. Measurement methods (laboratory).

Laboratory	Analyte	Sample matrix	Samples	Expected range of results	Method detection limit	Reporting limit	Sample prep method	Analytical (instrumental) method
KCEL	MC-LR, MC-LA, MC-LF, MC-LY, MC-LW, MC-RR, MC-YR, NOD	Water (surface and pore)	30 surface; 30 porewater	<MDL to 100 ug/L	0.04 ug/L	0.2 ug/L	KCEL SOP 469 (Mekebri et al 2009)	KCEL SOP 473 (Mekebri et al 2009)
	as above	Littoral sediment	30	<MDL to 100 ng/g	0.16 ng/g	0.8 ng/g	KCEL SOP 469 (Chen et al 2006)	KCEL SOP 473 (Mekebri et al 2009)
MEL	Chlorophyll a	Water	15	1 µg/L to 100 µg/L	NA	0.004 to 0.05 mg/L	SM10200H1	SM10200H3
	TP	Water	7	<MRL to 1 mg/L	0.005 mg/L	0.0024 mg/L	SM4500PB5	SM4500PH
	NO2-NO3	Water	7	<MRL to 1 mg/L	0.005 mg/L	0.01 mg/L	SM4500NO3I	SM4500NO3I
	NH3	Water	7	<MRL to 1 mg/L	0.006 mg/L	0.01 mg/L	SM4500NH3	SM4500NH3 H
	Total persulfate N	Water	7	<MRL to 2 mg/L	0.013 mg/L	0.025 mg/L	SM4500NB	SM4500NB
MTC	Grain size	Littoral sediment	21	1-15%	0.1%	0.1%	NA	PSEP
ECY-TSU	LOI	Sediment core	50	1 – 80%		1%	ASTM D7348-13	LOI (Heiri et al., 2001)
Test America	^{210}Pb radioisotopes	Sediment core	20	< 0.45 - 30 pCi/g	NA	0.45 pCi/g	Eakins and Morrison, 1978	Alpha Spectroscopy (Eakins and Morrison, 1978)
Dr. Rolf Vinebrooke	algal pigments	Sediment core	20	0.1 to 2000 nmole pigment	NA	0.1 nmole	Leavitt and Hodgson, 2001	HPLC (Mantoura and Llewellyn, 1983)
UW-Isolab	TOC:N and isotopes	Littoral sediment and core	75	0.1 - 2.0 (%N); 1.0 - 15 (%C)	NA	0.10%	lyophilization	ǂ stable isotopes of N and C

KCEL: King County Environmental Lab; MEL: Manchester Environmental Lab; MTC: Materials Testing and Consulting Inc.; ECY-TSU: Ecology – Toxics Studies Unit; UW: University of Washington; LOI: loss-on-ignition;
ǂ Costech Elemental Analyzer, Conflo III, MAT253

9.2 Sample preparation method(s)

Refer to Table 13.

9.3 Special method requirements

This project will use two non-standard analytical methods for the analysis of algal pigments and MC variants. While the analysis of the algal pigments does not have a standard method or EPA approved method, there is a 40-year history of analyzing pigment abundance (*reviewed in* Leavitt and Hodgson, 2001). There are a number of analytical approaches to measuring pigment abundance. The lab of Dr. Rolf Vinebrooke has a 20 year history of using high performance liquid chromatography (HPLC), relying on standard reference materials and chromatographic libraries for positive identification of the pigments.

The analysis of the MC variants by KCEL is not accredited, but the analysis of MC variants in non-potable water is in the process of being reviewed by the Lab Accreditation Unit in Ecology's EAP for accreditation. KCEL has completed projects for other clients interested in MC variants in solids/sediments using KCEL SOP 473, but they are not seeking accreditation for this media at this time. All previous lab QC has been reviewed by Ecology and KCEL.

9.4 Laboratories accredited for methods

The vast majority of laboratory analysis for this project are not accredited methods and will be analyzed by research laboratories at universities or by KCEL. All analyses for nutrients will be carried out at Manchester Environmental Laboratory. The laboratory and analysis being conducted are:

- Water and sediment MC variants –King County Environmental Lab (not accredited; waiver approved).
- Sediment Grain size - Materials Testing and Consulting, Inc., Tukwila, WA (accredited)
- Sediment TOC-TN and isotopes – IsoLab, University of Washington (not accredited; waiver approved).
- Sediment core algal pigments – Dr. Rolf Vinebrooke, University of Alberta (not accredited; waiver approved).
- Sediment core radioisotopes – Test America (accredited).

10.0 Quality Control Procedures

10.1 Table of field and laboratory quality control

Table 14. Quality control samples, types, and frequency.

Parameter	Field Replicates	Check Standards	Method Blanks	Analytical Duplicates	Matrix Spikes
MC variants (water)	NA	1/batch	1/batch	1/batch	1/batch
MC variants (sediment)	NA	1/batch	1/batch	1/batch	1/batch
Chlorophyll *a*	10% of samples	1/batch	1/batch	1/batch	NA
TP	10% of samples	1/batch	1/batch	1/batch	NA
NO_2–NO_3	10% of samples	1/batch	1/batch	1/batch	NA
NH_3	10% of samples	1/batch	1/batch	1/batch	NA
Total persulfate N	10% of samples	1/batch	1/batch	1/batch	NA
Grain size	10% of samples	NA	NA	1/batch	NA
Loss-on-ignition	10% of samples	NA	NA	NA	NA
TOC:TN	10% of samples	1/batch	1/batch	1/batch	NA
^{210}Pb radioisotopes	10% of samples	1/batch	1/batch	1/batch	NA
algal pigments	10% of samples	1/batch	NA	1/batch	1/batch

10.2 Corrective action processes

The laboratory analysts will document whether project data meet method QC criteria. Any departures from normal analytical methods will be documented by the laboratory and described in the data package from the laboratories and the final report for the project. If any samples do not meet QC criteria, the project manager will determine whether data should be re-analyzed, rejected, or used with appropriate qualification.

Field instruments will be checked and calibrated prior to the field. The post field check of the instrument should be within the MQOs defined in Table 11. The appropriate qualification or rejection threshold is detailed in the MQOs.

11.0 Data Management Procedures

11.1 Data recording and reporting requirements

Field data will be recorded in a bound notebook with waterproof paper. Corrections will be made with a single line strikethrough, initials, and date. Data will be transferred to Microsoft Excel for creating data tables.

11.2 Laboratory data package requirements

The laboratory data package will be generated by KCEL, MEL, and Vinebrooke Lab. Labs will provide a project data package that will include a narrative discussing the following:

- problems encountered in the analyses
- corrective actions taken
- QC and spike recovery values
- changes to the referenced method
- an explanation of data qualifiers

KCEL will also include all chromatograms for each sample. Quality control results will be evaluated by the labs (discussed below in Section 13.0).

The following data qualifiers will be used:

- "J" – The analyte was positively identified. The associated numerical result is an estimate.
- "UJ" – The analyte was not detected at or above the estimated reporting limit.
- "NJ" – The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.

The qualifiers will be used in accordance with the method reporting limits:

- For non-detect values, the estimated detection limit (EDL) is recorded in the "Result Reported Value" column with "UJ" in the "Result Data Qualifier" column.
- No results are reported below the EDL.
- Detected values that are below the limit of quantitation (LOQ) are reported and qualified as estimates ("J").

11.3 Electronic transfer requirements

All laboratory data will be accessed and downloaded from MEL's Laboratory Information Management System (LIMS) or received directly from KCEL in Excel spreadsheets.

11.4 EIM/STORET data upload procedures

All completed project data will be entered into Ecology's Environmental Information Management (EIM) database for availability to the public and interested parties. Data entered into EIM follow a formal data review process where data are reviewed by the project manager, the person entering the data, and an independent reviewer. All data entered into EIM are reviewed, not simply a random subset.

EIM can be accessed on Ecology's homepage. The project will be searchable under Study ID WHOB008.

12.0 Audits and Reports

12.1 Field, laboratory, and other audits

No defined audit exists for the field work in this project. Site visits to lakes with cyanobacterial blooms will likely be conducted in partnership with the local county contacts.

The Ecology Environmental Laboratory Accreditation Program evaluates a laboratory's quality system, staff, facilities and equipment, test methods, records, and reports. It also establishes that the laboratory is capable of providing accurate, defensible data. All assessments are available from Ecology upon request, including MEL's internal performance and audits.

Labs performing analytical methods not accredited by Ecology have submitted a "Request to Waive Required Use of Accredited Lab" form through the project manager. Each form has been approved by the acting QA Officer; the form identifies the QC samples that will be analyzed to help demonstrate performance of the method (e.g., acceptable bias and precision).

12.2 Responsible personnel

The project manager will be responsible for all reporting.

12.3 Frequency and distribution of reports

Reporting for this project will be separated into two deliverables:

1. The persistence and prevalence of cyanotoxins in littoral sediments will be written as a journal publication with an accompanying EAP Fact Sheet.
2. The sediment record of cyanobacteria in Anderson Lake will be published as a short-form report compatible with previous sediment core reports.

The continuous monitoring data collected on chlorophyll and phycocyanin will be used as a visualization tool and will not be reported on. This data will be publically available in EIM.

12.4 Responsibility for reports

The journal publication will be authored by Ellen Preece (lead), Joan Hardy and William Hobbs. The sediment core report will be authored by William Hobbs and Siana Wong.

13.0 Data Verification

13.1 Field data verification, requirements, and responsibilities

The field assistant will review field notes once they are entered into Excel spreadsheets. Oversight will be provided by the project manager.

13.2 Laboratory data verification

As previously described, KCEL, MEL, and Vinebrooke Lab will oversee the review and verification of all laboratory data packages. The final data package from KCEL must include the following:

- a text narrative
- analytical result reports
- chromatograms
- spectra for all standards
- environmental samples
- batch QC samples
- preparation bench sheets

All of the necessary QA/QC documentation must be provided, including results from matrix spikes, replicates, and blanks.

13.3 Validation requirements, if necessary

It is expected that external data validation will not be necessary for this project.

14.0 Data Quality (Usability) Assessment

14.1 Process for determining project objectives were met

The project manager will determine if the project data are useable by assessing whether the data have met the MQOs (outlined in Tables 10 and 11) and discussing the data with laboratory chemists. Based on this assessment, the data will be accepted, accepted with appropriate qualifications, or rejected and considered for re-analysis.

It is expected that the project manager and laboratory project managers and chemists will be in regular contact throughout the project.

14.2 Treatment of non-detects

Non-detects will be treated as defined: the analyte was not detected at or above the estimated LOQ or reporting limit. Non-detect values (U, UJ) are assigned a value of zero for the summing process when the group of analytes being summed has both detected and non-detected results. Estimated values (J) are defined as those detected below the LOQ.

14.3 Data analysis and presentation methods

Statistical analysis will be completed in R (R Core Team, 2018) and will consist of comparisons among the sampling events for the presence and attenuation of cyanotoxins. An analysis of variance (ANOVA) with a Levene's test for equality of variance will be used to test for significant differences among the lakes and sampling events at the same lake. Non-parametric methods, such as the Kruskal-Wallis test or one-way ANOVA on ranks, may also be used to analyze non-normally distributed data rather than transforming the data. A power analysis will also be conducted to test size of the effect between re-visits to the same lake when sampling for cyanotoxin attenuation or persistence in the littoral sediments.

14.4 Sampling design evaluation

The sample design has sufficient replication at each lake to summarize local variability and make simple statistical comparisons among the lakes. The statistical power of consecutive sampling events at the same lake will allow us to say whether there is a measurable difference over time and will allow us to define the spatial variability of cyanotoxins in sediments at a public beach.

14.5 Documentation of assessment

Publications and reports will present the findings, interpretations, and recommendations from this study.

15.0 References

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16.0 Appendices

Appendix A. Glossaries, Acronyms, and Abbreviations

Glossary of General Terms

Ambient: Background or away from point sources of contamination. Surrounding environmental condition.

Clean Water Act: A federal act passed in 1972 that contains provisions to restore and maintain the quality of the nation's waters. Section 303(d) of the Clean Water Act establishes the TMDL program.

Conductivity: A measure of water's ability to conduct an electrical current. Conductivity is related to the concentration and charge of dissolved ions in water.

Cyanobacteria: Group of ancient and diverse microorganisms that have prokaryotic (bacterial) cell structure. They are photosynthetic and act like algae, but are classified as a Eubacteria. They are found in marine and freshwater. They are also called cyanophyta, blue-green algae and toxic algae.

Cyanotoxin: A term used to describe the toxins produced by cyanobacteria. It is a general term that refers to many different compounds. The toxins are a secondary metabolite produced by cyanobacteria.

Chlorophyll *a*: One of the primary pigments in algae responsible for the acquisition of energy to allow photosynthesis to occur. It is a form of chlorophyll.

Dissolved oxygen (DO): A measure of the amount of oxygen dissolved in water.

Lyophilization: Freeze-drying; the sublimation of water from a frozen sample.

Microcystin: class of toxins produced by certain freshwater cyanobacteria. So far, approximately 150 different isomers of microcystin have been observed. They are hepatotoxins, which affect the liver. They are produced mainly by genus *Microcystis*.

Nutrient: Substance such as carbon, nitrogen, and phosphorus used by organisms to live and grow. Elevated nutrient concentrations in the water can promote algal blooms and rob the water of oxygen vital to aquatic organisms.

pH: A measure of the acidity or alkalinity of water. A low pH value (0 to 7) indicates that an acidic condition is present, while a high pH (7 to 14) indicates a basic or alkaline condition. A pH of 7 is considered to be neutral. Since the pH scale is logarithmic, a water sample with a pH of 8 is ten times more basic than one with a pH of 7.

Phycocyanin: One of the primary pigments found in cyanobacteria. It has a characteristic light blue color. It is an accessory pigment to chlorophyll.

Sediment: Soil and organic matter that is covered with water (for example, a river or lake bottom).

Acronyms and Abbreviations

^{210}Pb	radioisotope of lead
CCV	Continuing calibration verification standard
CRM	Certified reference materials
DO	Dissolved oxygen (see Glossary above)
DOH	Washington State Department of Health
Ecology	Washington State Department of Ecology
EIM	Environmental Information Management database
EPA	United States Environmental Protection Agency
et al.	And others
KCEL	King County Environmental Lab
LCS	Laboratory control sample
MEL	Manchester Environmental Laboratory
MQO	Measurement quality objective
NH_3	Ammonia
NO_2^-	Nitrite
NO_3^-	Nitrate
QA	Quality assurance
QC	Quality control
RPD	Relative percent difference
SOP	Standard operating procedures
SRM	Standard reference materials
TOC	Total Organic Carbon
TN	Total Nitrogen
TP	Total phosphorus
WAC	Washington Administrative Code

Units of Measurement

°C	degrees centigrade
dw	dry weight
g	gram, a unit of mass
hr	hour
ha	hectares
kg	kilograms
mg/L	milligrams per liter (parts per million)
nmol	nanomole or one-billionth of a mole
mole	an International System of Units (SI) unit of matter
pCi/g	picocurie per gram
RFU	relative fluorescence units
µg/L	micrograms per liter (parts per billion)
µS/cm	microsiemens per centimeter, a unit of conductivity
ww	wet weight

Quality Assurance Glossary

Accreditation: A certification process for laboratories, designed to evaluate and document a lab's ability to perform analytical methods and produce acceptable data. For Ecology, it is "Formal recognition by (Ecology)…that an environmental laboratory is capable of producing accurate analytical data." [WAC 173-50-040] (Kammin, 2010)

Accuracy: The degree to which a measured value agrees with the true value of the measured property. USEPA recommends that this term not be used, and that the terms *precision* and *bias* be used to convey the information associated with the term *accuracy*. (USGS, 1998)

Analyte: An element, ion, compound, or chemical moiety (pH, alkalinity) which is to be determined. The definition can be expanded to include organisms, e.g., fecal coliform, Klebsiella. (Kammin, 2010)

Bias: The difference between the sample mean and the true value. Bias usually describes a systematic difference reproducible over time and is characteristic of both the measurement system and the analyte(s) being measured. Bias is a commonly used data quality indicator (DQI). (Kammin, 2010; Ecology, 2004)

Blank: A synthetic sample, free of the analyte(s) of interest. For example, in water analysis, pure water is used for the blank. In chemical analysis, a blank is used to estimate the analytical response to all factors other than the analyte in the sample. In general, blanks are used to assess possible contamination or inadvertent introduction of analyte during various stages of the sampling and analytical process. (USGS, 1998)

Calibration: The process of establishing the relationship between the response of a measurement system and the concentration of the parameter being measured. (Ecology, 2004)

Check standard: A substance or reference material obtained from a source independent from the source of the calibration standard; used to assess bias for an analytical method. This is an obsolete term, and its use is highly discouraged. See Calibration Verification Standards, Lab Control Samples (LCS), Certified Reference Materials (CRM), and/or spiked blanks. These are all check standards but should be referred to by their actual designator, e.g., CRM, LCS. (Kammin, 2010; Ecology, 2004)

Comparability: The degree to which different methods, data sets and/or decisions agree or can be represented as similar; a data quality indicator. (USEPA, 1997)

Completeness: The amount of valid data obtained from a project compared to the planned amount. Usually expressed as a percentage. A data quality indicator. (USEPA, 1997)

Continuing Calibration Verification Standard (CCV): A quality control (QC) sample analyzed with samples to check for acceptable bias in the measurement system. The CCV is usually a midpoint calibration standard that is re-run at an established frequency during the course of an analytical run. (Kammin, 2010)

Control chart: A graphical representation of quality control results demonstrating the performance of an aspect of a measurement system. (Kammin, 2010; Ecology 2004)

Control limits: Statistical warning and action limits calculated based on control charts. Warning limits are generally set at +/- 2 standard deviations from the mean, action limits at +/- 3 standard deviations from the mean. (Kammin, 2010)

Data integrity: A qualitative DQI that evaluates the extent to which a data set contains data that is misrepresented, falsified, or deliberately misleading. (Kammin, 2010)

Data quality indicators (DQI): Commonly used measures of acceptability for environmental data. The principal DQIs are precision, bias, representativeness, comparability, completeness, sensitivity, and integrity. (USEPA, 2006)

Data quality objectives (DQO): Qualitative and quantitative statements derived from systematic planning processes that clarify study objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions.
(USEPA, 2006)

Data set: A grouping of samples organized by date, time, analyte, etc. (Kammin, 2010)

Data validation: An analyte-specific and sample-specific process that extends the evaluation of data beyond data verification to determine the usability of a specific data set. It involves a detailed examination of the data package, using both professional judgment and objective criteria, to determine whether the MQOs for precision, bias, and sensitivity have been met. It may also include an assessment of completeness, representativeness, comparability, and integrity, as these criteria relate to the usability of the data set. Ecology considers four key criteria to determine if data validation has actually occurred. These are:

- Use of raw or instrument data for evaluation.
- Use of third-party assessors.
- Data set is complex.
- Use of EPA Functional Guidelines or equivalent for review.

Examples of data types commonly validated would be:

- Gas Chromatography (GC).
- Gas Chromatography-Mass Spectrometry (GC-MS).
- Inductively Coupled Plasma (ICP).

The end result of a formal validation process is a determination of usability that assigns qualifiers to indicate usability status for every measurement result. These qualifiers include:

- No qualifier – data are usable for intended purposes.
- J (or a J variant) – data are estimated, may be usable, may be biased high or low.
- REJ – data are rejected, cannot be used for intended purposes.
  (Kammin, 2010; Ecology, 2004).

Data verification: Examination of a data set for errors or omissions, and assessment of the Data Quality Indicators related to that data set for compliance with acceptance criteria (MQOs). Verification is a detailed quality review of a data set. (Ecology, 2004)

Detection limit (limit of detection)**:** The concentration or amount of an analyte which can be determined to a specified level of certainty to be greater than zero. (Ecology, 2004)

Duplicate samples: Two samples taken from and representative of the same population, and carried through and steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variability of all method activities including sampling and analysis. (USEPA, 1997)

Field blank: A blank used to obtain information on contamination introduced during sample collection, storage, and transport. (Ecology, 2004)

Initial Calibration Verification Standard (ICV): A QC sample prepared independently of calibration standards and analyzed along with the samples to check for acceptable bias in the measurement system. The ICV is analyzed prior to the analysis of any samples. (Kammin, 2010)

Laboratory Control Sample (LCS): A sample of known composition prepared using contaminant-free water or an inert solid that is spiked with analytes of interest at the midpoint of the calibration curve or at the level of concern. It is prepared and analyzed in the same batch of regular samples using the same sample preparation method, reagents, and analytical methods employed for regular samples. (USEPA, 1997)

Matrix spike: A QC sample prepared by adding a known amount of the target analyte(s) to an aliquot of a sample to check for bias due to interference or matrix effects. (Ecology, 2004)

Measurement Quality Objectives (MQOs)**:** Performance or acceptance criteria for individual data quality indicators, usually including precision, bias, sensitivity, completeness, comparability, and representativeness. (USEPA, 2006)

Measurement result: A value obtained by performing the procedure described in a method. (Ecology, 2004)

Method: A formalized group of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, data analysis), systematically presented in the order in which they are to be executed. (EPA, 1997)

Method blank: A blank prepared to represent the sample matrix, prepared and analyzed with a batch of samples. A method blank will contain all reagents used in the preparation of a sample, and the same preparation process is used for the method blank and samples. (Ecology, 2004; Kammin, 2010)

Method Detection Limit (MDL): This definition for detection was first formally advanced in 40CFR 136, October 26, 1984 edition. MDL is defined there as the minimum concentration of an analyte that, in a given matrix and with a specific method, has a 99% probability of being identified, and reported to be greater than zero. (Federal Register, October 26, 1984)

Percent Relative Standard Deviation (%RSD): A statistic used to evaluate precision in environmental analysis. It is determined in the following manner:

$$\%RSD = (100 * s)/x$$

where s is the sample standard deviation and x is the mean of results from more than two replicate samples. (Kammin, 2010)

Parameter: A specified characteristic of a population or sample. Also, an analyte or grouping of analytes. Benzene and nitrate + nitrite are all "parameters." (Kammin, 2010; Ecology, 2004)

Population: The hypothetical set of all possible observations of the type being investigated. (Ecology, 2004)

Precision: The extent of random variability among replicate measurements of the same property; a data quality indicator. (USGS, 1998)

Quality assurance (QA): A set of activities designed to establish and document the reliability and usability of measurement data. (Kammin, 2010)

Quality Assurance Project Plan (QAPP): A document that describes the objectives of a project, and the processes and activities necessary to develop data that will support those objectives. (Kammin, 2010; Ecology, 2004)

Quality control (QC): The routine application of measurement and statistical procedures to assess the accuracy of measurement data. (Ecology, 2004)

Relative Percent Difference (RPD): RPD is commonly used to evaluate precision. The following formula is used:

$$[Abs(a-b)/((a + b)/2)] * 100$$

where "Abs()" is absolute value and a and b are results for the two replicate samples. RPD can be used only with 2 values. Percent Relative Standard Deviation is (%RSD) is used if there are results for more than 2 replicate samples (Ecology, 2004).

Replicate samples: Two or more samples taken from the environment at the same time and place, using the same protocols. Replicates are used to estimate the random variability of the material sampled. (USGS, 1998)

Representativeness: The degree to which a sample reflects the population from which it is taken; a data quality indicator. (USGS, 1998)

Sample (field): A portion of a population (environmental entity) that is measured and assumed to represent the entire population. (USGS, 1998)

Sample (statistical): A finite part or subset of a statistical population. (USEPA, 1997)

Sensitivity: In general, denotes the rate at which the analytical response (e.g., absorbance, volume, meter reading) varies with the concentration of the parameter being determined. In a specialized sense, it has the same meaning as the detection limit. (Ecology, 2004)

Spiked blank: A specified amount of reagent blank fortified with a known mass of the target analyte(s); usually used to assess the recovery efficiency of the method. (USEPA, 1997)

Spiked sample: A sample prepared by adding a known mass of target analyte(s) to a specified amount of matrix sample for which an independent estimate of target analyte(s) concentration is available. Spiked samples can be used to determine the effect of the matrix on a method's recovery efficiency. (USEPA, 1997)

Split sample: A discrete sample subdivided into portions, usually duplicates (Kammin, 2010)

Standard Operating Procedure (SOP): A document which describes in detail a reproducible and repeatable organized activity. (Kammin, 2010)

Surrogate: For environmental chemistry, a surrogate is a substance with properties similar to those of the target analyte(s). Surrogates are unlikely to be native to environmental samples. They are added to environmental samples for quality control purposes, to track extraction efficiency and/or measure analyte recovery. Deuterated organic compounds are examples of surrogates commonly used in organic compound analysis. (Kammin, 2010)

Systematic planning: A step-wise process which develops a clear description of the goals and objectives of a project, and produces decisions on the type, quantity, and quality of data that will be needed to meet those goals and objectives. The DQO process is a specialized type of systematic planning. (USEPA, 2006)

References for QA Glossary

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