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Water Quality Sampling in Lakes and Reservoirs

**Commonwealth of Kentucky
Energy and Environment Cabinet
Department for Environmental Protection
Division of Water**

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4. Scope and Applicability

This document provides instruction for the collection, preservation, and handling of water quality samples, and the procedures for field measurements in lakes and reservoirs that are monitored by Kentucky Division of Water (KDOW). This document is used primarily by the Ambient Lakes Monitoring program but can be used for other monitoring purposes where appropriate. Many details of sampling and field measurement procedures overlap with other KDOW SOPs that are generic for all samples or were written for flowing waters (e.g. rivers and streams). Those SOPs are incorporated by reference and the elements summarized where they come into play:

- Sample Control and Management (DOWSOP03001)
- In-situ Water Quality Measurements and Meter Calibration (DOWSOP03014)
- Sampling Surface Water Quality in Lotic Systems (DOWSOP03015).

This SOP should be reviewed and updated on a regular basis to meet monitoring program and assessment data needs.

5. Executive Summary

This document summarizes water sampling and field measurement methods performed by KDOW personnel for use in lakes and reservoirs. Two methods of sample collection are described: a subsurface grab directly to a sample container, or a vertical composite created from samples at discrete depths throughout the euphotic zone. Grab samples are collected into separate bottles specific to requested analyses. Composite samples are created in a carboy and then sample bottles are filled. For routine monitoring, sample bottles collected include metals, nutrient, bulk parameters, and chlorophyll. Samples for orthophosphate, dissolved organic carbon, and total dissolved phosphorus are filtered immediately after collection to create a filtered sample for these parameters.

6. Acronyms

COC: Chain of Custody
DEP: Department for Environmental Protection
DI: De-ionized Water
DO: Dissolved Oxygen
DOC: Dissolved Organic Carbon
ESB: Environmental Services Branch
FH: Filter Holder Apparatus
HDPE: High Density Polyethylene
HVP: Hand Vacuum Pump
KDOW: Kentucky Division of Water
KS: Kemmerer Sampler

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PAR: Photosynthetically Active Radiation
PPE: Personal Protective Equipment
QA: Quality Assurance
QC: Quality Control
SD: Secchi Disc
SOP: Standard Operating Procedure
VOC: Volatile Organic Compounds
WQB: Water Quality Branch

7. Health & Safety Issues

Human Health

Field staff working in and around potentially contaminated surface waters should receive an immunization shot for Hepatitis A in accordance with DEP Policy SSE-708. In addition, staff should receive immunization for Hepatitis B and tetanus, to aid in the prevention of contracting diseases associated with those pathogens. All field staff should also be trained in CPR, First Aid and Blood Borne Pathogens in accordance with DEP Policy SSE-711.

Personal protective equipment (PPE) should be used when sampling in known waters with the potential for adverse health effects, or in unknown waters that have been determined impaired, but the pollutants have not been identified. All field staff should review the PPE modules at <http://www.laborcabinetetrain.ky.gov/courses.html>.

The following items are examples of PPE that may be used during sampling:

- Personal floatation device
- Powderless latex or nitrile gloves
- Neoprene gloves – in cold water
- Cold weather clothing such as a hat, ear-warmers, water-resistant outer wear
- UV protection including long sleeve shirts, hats and sunscreen
- Polarized sunglasses

Safety Equipment

Monitoring may include field activities during all stages of the hydrologic cycle, including high discharge/flood stage conditions. The additional precautions outlined below should be taken during high discharge events:

- Samplers shall always wear approved personal floatation devices while under way in a boat.
- The buddy system should be implemented when conducting field and boat work.

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- If high discharge conditions are determined unsafe by any field team member, do not sample during that time.

8. Cautions and Interferences

The following precautions shall be considered when collecting water samples.

- This standard operating procedure (SOP) specifically addresses water samples that are taken from lakes and reservoirs waters. It may not be appropriate to use the methods presented in this document for other types of water sampling.
- Samples should always be stored in a secure location to ensure that they cannot be tampered with.

It is important to remain cognizant of potential sources of contamination when sampling. Gloves should always be worn when collecting a sample. Immediately cap all bottles after filling with the sample water and double check that the caps are completely secured on the sample bottles prior to storing in a cooler.

Don gloves prior to performing any filtering in order to prevent contamination.

When filtering for dissolved metals, all efforts should be made to ensure that the area is free and clear of possible contaminants. Do not collect or filter samples while the boat is under way or the outboard motor is running to eliminate the motor exhaust fumes as a possible contaminant.

9. Personnel Qualifications/Responsibilities

All personnel involved in lake water quality sampling will meet at least the minimum qualifications for their job classification. In addition, all field staff will be trained in the proper water sampling collection and preservation techniques. Training will continue on-the-job through interaction with experienced field personnel and continued outside training when educational opportunities become available. Personnel performing these procedures should already be trained in the related SOPs listed in section 4.

10. Equipment, Instrumentation and Supplies

Table 1 contains a list of supplies that may be required for lake and reservoir water sampling.

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Table 1. Lake and Reservoir Water Sampling Equipment and Supplies

Sample Bottles	
30 mL Nalgene narrow-mouth, high-density polyethylene bottle	(1)
60 mL Nalgene narrow-mouth, high-density polyethylene bottle	(1)
500 mL HDPE wide-mouth Nalgene Jar	(3)
1L Natural HDPE cylinder	(1)
250 mL brown (opaque) bottle	(1)
Chemical Preservatives	
3.5 mL vial w/ 1 mL 1:1 H ₂ SO ₄ (Sulfuric Acid)	(1)
3.5 mL vial w/ 1 mL 1:1 HNO ₃ (Nitric Acid)	(1)
Sampling Equipment	
47mm filter holder	
Hand vacuum pump	
Kemmerer sampler	
Secchi disc	
YSI Pro multi-meter with 30m cable	
LiCor® 250a PAR light meter with quantum sensor	
6L carboy with spigot	
Teflon or Tygon tubing	
Sampling Supplies	
Powderless latex/nitrile gloves	
0.45µm x 47mm sterile membrane filters	
Deionized water	
Tweezers	
Sample storage coolers	
Ice	
Sealable plastic bags	
Sharpies	
Waterproof pen	
Field sheets/Chain of Custody forms	

11 Step-by-Step Procedure

11.1 Sample Container and Identification

Several analyses may be performed using source water from one sample bottle. All analytical parameters in one bottle must require the same sample bottle type and sample preservative type. An example list of variable groups can be found in Table 2. The specific analyses that will be required from these variable groups must be described on the Chain-of-Custody (COC) documentation. The specific analytes within each variable group will vary depending upon project objectives and should be outlined in project specific Quality Assurance Project Plans.

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When requesting multiple analyses from one sample bottle it is important to ensure that there is adequate volume in the sample bottle and that all test methods required from one bottle require the same preservation method. The minimum required sample volume and preservation method for specific analyses can be found in Table 1.

Table 1. Chemical parameters collected with method, preservative, reporting limits, container and volume required holding time and ESB method.

Bottle	Parameter	Preservative	Reporting limits	Method Detection Limit	Unit	Container	Volume Required	Holding Time	ESB Method
1	Nutrients	H ₂ SO ₄ , Ice				Plastic	1 L		
	Ammonia(as N)		0.05	0.025	mg/L			28 days	2000
	Nitrate-Nitrite		0.02	0.010	mg/L			28 days	2120
	Total Kjeldahl Nitrogen		0.50	0.20	mg/L			28 days	2280
	Total Organic Carbon		0.25	0.10	mg/L			28 days	2260
	Total Phosphorus		0.020	0.010	mg/L			28 days	2200
2	DOC/TDP	Ice				plastic	60 mL filtered		
	Dissolved Organic Carbon		0.25	0.010	mg/L			28 days	2260-D
	Total Dissolved Phosphorus		0.020	0.010	mg/L			28 days	2180-D
3	Ortho-P Orthophosphate (as P)	ice	0.020	0.010	mg/L	plastic	30 mL filtered	48 hours	2160-D
4	Acidity/Alkalinity Alkalinity	Ice	5.0		mg/L	Plastic	500 mL	14 days	1020/1030/1040
5	Bulk Chloride Sulfate Total Suspended Solids	Ice	5.0 4.0 1.5		mg/L mg/L mg/L	Plastic	1 L	28 days 28 days 7 days	1100 1425 1320

Information for every sampled site should be recorded on the bottle, either directly on the bottle surface, or on a waterproof bottle label sealed by clear packing tape. All marking should be done in black, permanent ink (Sharpie© marker, fine or medium point, or the equivalent). Thirty milliliter and 60mL bottles may be too small for complete ID. In that case, abbreviate the ID on the bottles and place in a food storage bag marked with the complete ID.

At a minimum, the following information should be recorded on the sample bottle and/or label:

- Site ID
- Site Location
- Date
- Time
- Initials of Sampler(s)

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- Analysis parameters (i.e. Bulk, Nutrients, Metals, etc.)
- Preservation method

Table 2. Example list of variable groups

Variable Groups	Parameters
Bulk	BOD, Bromide, Chloride, Color, Conductivity, Fluoride, Nitrite, pH, Sulfate, Total Dissolved Solids, Total Suspended Solids, Turbidity
Nutrients	Ammonia, Nitrate-Nitrite, Total Kjeldahl Nitrogen, Total Organic Carbon, Total Phosphorus
Metals	Dissolved Metals
Chlorophyll <i>a</i>	Chlorophyll <i>a</i>
Alkalinity/Acidity	Alkalinity as CaCO ₃ , Bicarbonate as CaCO ₃ , Carbonate as CaCO ₃ , Acidity

11.2 Sample Collection

The types of lake and reservoir water samples currently collected by KDOW include the following:

11.2.1 Subsurface grab Samples

Subsurface grab samples should be collected in the uppermost section (0.3m) of the water but not from the surface/air interface. The following procedure for collecting grab samples should be followed:

- Invert the bottle and submerge until your forearm is in the water (0.3m). Tilt the bottle upward to allow water to flow into the bottle from just beneath the surface.
- Withdraw the bottle and shake vigorously.
- Discard water.
- Repeat for total of three rinses.
- Submerge entire bottle and fill, leaving enough headspace to allow adequate mixing if preservation with acid is required**.
- Rinse the caps with sample water prior to capping the bottle.
- Preserve sample and place in ice chest.

** Special Consideration:

When collecting a sample to be analyzed by the Environmental Services Branch (ESB) for acidity, alkalinity or volatile organic compounds (VOC), the bottle must be completely filled. No head space should exist between the bottle cap and sample water.

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Chlorophyll a samples should have head space (25mL) to allow for mixing prior to filtering in the lab.

11.2.2 Composite Samples

Depth integrated composite samples are taken to quantify analytes of interest within the euphotic zone. The euphotic zone is defined as that strata of the lake in which at least 1% of surface photosynthetically active radiation (PAR) exists. Samples are taken at subsurface (0.3m) and again at every meter throughout the euphotic zone. WQB uses 1 liter Kemmerer samplers and 6 liter carboys. Three liters of sample are required to fill each type of sample at one site with no duplicates. The technician needs to determine from the euphotic zone, in advance, the quantity of sample water required from each depth increment to meet or exceed the 3 liter minimum while sampling equal volume at each depth interval.

Determine Euphotic Zone

The most accurate method to determine the euphotic zone is with a light meter. KDOW uses a LiCor® 250a with a quantum sensor for PAR quantification. If the light meter is not available or is malfunctioning, Secchi depth (SD) can be used to estimate the euphotic zone by multiplying SD by 2.0. Note: Determination of the euphotic zone should not be performed early in the morning or late in the evening due to the low angle of the sun to the lake surface. If the day is intermittently cloudy, insure that similar light conditions exist for both the surface and subsurface measures.

LiCor® 250a method (Preferred)

- Turn the hand-held unit “on”.
- Position the sensor near the water surface and free of any shading. Surface (ambient) PAR will be displayed in μmol . When the display equilibrates, press “hold”.
- Record surface PAR and note 1% of that value by moving the decimal point 2 places to the left. Note: The meter will not display a constant value. The tens and ones place values will be in constant flux. A typical daylight surface value may be $\sim 2437 \mu\text{mol}$. Round 2437 to 2400 and use 24 as 1%.
- Press “hold” again to reactivate the meter and slowly lower the sensor until the 1% value is reached note the depth using meter graduations on the sensor cable.
- Record the sensor depth in meters on the data sheet as the euphotic depth.

Secchi Depth

- Remove sunglasses
- From the shaded side of the boat, lower the SD until it is no longer visible and note that depth from the graduations marked on the tape.
- Raise the SD until it is again visible and note that depth.

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- Repeat both previous steps until each depth value is substantially constant
- Take the average of the two depths and record this value on the data sheet.

Kemmerer Sampler (KS) Technique

- Determine euphotic zone
- Open KS by fully extending both end caps. KS will “lock” in open position.
- Lower KS to just below the surface
- Release weighted messenger to close the KS and retrieve
- Open spigot and pour the necessary volume into the carboy
- Repeat through the euphotic zone in meter increments as indicated by the black meter graduations on the cable.
- When sample collection is complete, vigorously agitate carboy to thoroughly mix composite sample and dispense through spigot into individual sample bottles or filter holder apparatus (FH).

Filtered Samples

Ortho-P and dissolved organic carbon (DOC) samples must be filtered of particulate matter. Gloves should be worn to prevent cross contamination.

Hand Pump Technique

Rinsing Procedure:

- Don gloves
- Triple rinse the following equipment with de-ionized (DI) water: flask, funnel filter, and tweezers

Filtering Procedure:

- Use Sharpie pen to label bottles and Zip-lock bags with appropriate site information.
- After rinsing the filter holder apparatus (FH) with DI water, invert composite sample several times in order to mix the sample
- Place 0.45 μ m membrane filter into FH using tweezers
- Connect the tubing from the hand pump
- Pour ~50 mL DI water into funnel (purpose is to rinse the filter)
- Filter and discard the filtered water
- Pour ~25 mL sample water into funnel (purpose is to contaminate the funnel and filter with the sample water)
- Filter and discard the filtered water
- Pour ~125 mL sample water into funnel (purpose is to collect a filtered sample)
- Filter and fill each sample bottle to ~half full
- Shake sample bottle and discard water (purpose is to rinse the bottles)

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- Pour the remainder of the filtered water into the sample bottles leaving enough room for expansion once the sample is frozen
- Place sample bottle in the labeled Zip-lock bag and then in a cooler with ice
- Rinse filter apparatus with DI water to ready for next sample
- Upon returning from the field, place the samples in the freezer to be delivered to the ESB laboratory the following day

Note: Highly turbid water may clog the membrane filter. It may be necessary to filter separate samples for each analyte.

11.3 Sample Preservation and Handling

Sample Preservation

All surface water samples should be collected in the appropriate bottles and preserved in the correct manner. Sample preservation should occur within 15 minutes of collection in the field. All labels on sample containers that have been preserved with chemicals must include the type of preservative used.

Refer to the *Sample Control and Management SOP* (KDOW 2009) for specific requirements for sample preservation documentation as it pertains to COC records.

Sample Storage and Transport

Samples should be stored in containers that are free of possible contaminants. Sample bottles may be placed inside of sealed food grade plastic bags prior to being stored on ice in coolers if cross contamination is deemed to be a likely possibility.

Refer to the *Sample Control and Management SOP* (KDOW 2009) for specific requirements for sample storage and transport.

Chain of Custody

All surface water samples should be accompanied by accurate and traceable sample COC documentation. Refer to the *Sample Control and Management SOP* (KDOW 2009) for specific requirements.

11.4 Physiochemical Parameters

The Lake Station Field sheet (see Appendix) should be filled out with every site visit. It is possible in very clear or very shallow lakes that Secchi depth or the euphotic zone extends to the bottom substrate. If either of these cases is true, circle "yes" in the upper right hand box. If Secchi depth is less than one meter and turbidity is judged to be substantially algal, a toxin sample and/or phytoplankton sample may be taken and "yes" should be circled opposite that question. The chlorophyll worksheet is to be completed in the DOW lab.

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Depth Profile

The depth profile is an indicator of water column heterogeneity. Depending on the depth of the lake and the season, there may be layers formed by temperature or DO. Each layer is defined by the asymptote of the curve formed by plotting the independent variable of interest against the depth of the sample. Water temperature at depth defines the thermocline which defines the lake into 3 general layers, the epilimnion, metalimnion and hypolimnion.

To avoid damage to the sonde or disturbing the substrate and subsequently causing false readings, do not sample to the bottom of the lake.

Multi-meter profiling method

- Determine the deepest strata to be sampled with depth meter (hand held or fish finder). Lakes 10m or less in depth should be sampled only to within 0.5m of the bottom. Lakes greater than 10m in depth should be sampled only to within 1m of the bottom.
- Lower the sonde to just below the surface and allow time for equilibration. Record the temperature in ° C, pH, DO in mg/L, percent saturation, and conductivity in $\mu\text{S}/\text{cm}$. Note that meters using a Clark cell (membrane) consume DO in the measurement process and thus need to have some (1 ft. s^{-1}) flow past the sensor. It may be necessary to move the sonde up and down gently to obtain an accurate reading.
- Repeat as necessary at 1 meter intervals until 20 meters is sampled. Depths greater than 20m are sampled at 5m intervals. Lakes deeper than about 15m may require additional weight added to the sonde to insure a vertical profile.

12. Data and Records Management

Water chemistry results will be either entered into the database KWADE manually or automatically uploaded when available from the lab. PDF copies of results and COC documentation shall be stored in V:\DOWWQB\Lakes\.

13. Quality Assurance and Quality Control

13.1 QC Sample Types

The types of quality control samples collected for various projects must be specified in the Quality Assurance Project Plan (QAPP). The purposes of quality control (QC) samples are to provide information on background conditions, isolate site effects, and evaluate contamination during sample transit or to evaluate field and laboratory variability. Types of QC samples may include:

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Field Split Sample: A sample that is collected by initially collecting twice as much volume as is normally collected and then apportioning, after mixing, into two sets of containers. This type of sample is used to assess analysis variability.

Field Blank: A sample that is prepared in the field using de-ionized or certified ultra-pure water. The water is poured into appropriate sample containers at specific locations during a sampling event. The sample is used to assess potential contamination from the environment, not associated with the source being sampled.

Field Rinsate Blank/Equipment Blank: A sample used to assess the possible contamination level of equipment that is field cleaned and re-used on-site. The sample is taken by rinsing field cleaned equipment with de-ionized water and collecting the rinse water to be submitted for analyses of all constituents that are normally collected using that piece of equipment.

Trip Blank: A sample used to assess the potential contamination level of sample storage containers during transit

14. References

Kentucky Division of Water (KDOW). 2009. Sample Control and Management SOP. Kentucky Department for Environmental Protection, Division of Water, Frankfort, Kentucky.

Kentucky Division of Environmental Services (DES). 2007. Standard Operating Procedure for Sample Receiving and Custody. Environmental and Public Protection Cabinet, Department for Environmental Protection, Division of Environmental Services. Frankfort, KY.

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15. Appendix

Sample Lake Station Field Sheet

Station Name Energy lake @ dam					Station Number: CLN001				
Date: 03-29-2016				Time: (24h) 0914		Phytoplankton sample? Circle if <input type="checkbox"/> yes			
Secchi Depth (m) 1.15				Chl a(ug/L) 20.3		Toxin sample? <input type="checkbox"/> yes			
Depth (Max) (m) 4.3				Euphotic Zone Depth (m) 2.3		Secchi depth to bottom? <input type="checkbox"/> yes			
Euphotic zone bottom? <input type="checkbox"/> yes									
Depth	Temp	pH	DO mg / %	Cond	Depth	Temp	pH	DO mg / %	Cond
Surface	20.4	7.7	9.8/107	325	16m			/	
1m	19.6	7.6	9.5/102	325	17m			/	
2m	15.3	7.6	9.3/100	325	18m			/	
3m	12.2	7.5	8.5/89	325	19m			/	
4m	8.9	7.5	7.8/77	328	20m			/	
5m			/		25m			/	
6m			/		30m			/	
7m			/		35m			/	
8m			/		40m			/	
9m			/		45m			/	
10m			/		50m			/	
11m			/		55m			/	
12m			/		60m			/	
13m			/		65m			/	
14m			/		70m			/	
15m			/		75m			/	

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Physical Conditions:

Weather at Time of Visit: **Circle one**

Clear/Sunny Cloudy/Overcast Intermittent Showers Steady Rain Heavy Rain Snow/Sleet/Hail

Weather in Past 24 hours: **Circle one**

Clear/Sunny Cloudy/Overcast Intermittent Showers Steady Rain Heavy Rain Snow/Sleet/Hail

Other Observations: Place an 'X' if present

Fish kill _____ oil sheen _____ algae bloom _____ foam _____ odor _____

Floating woody debris _____ floating garbage _____

Comments:

Describe any unusual conditions, malfunctions or elaborate on observations

DOW Laboratory Data Chlorophyll a worksheet

Chlorophyll a

Sample volume filtered 100 mL

Extract 10 mL

Dilution ratio _____

Chlorophyll a 20.3 ug/L