

Volunteer Assisted Water Quality and Biological Monitoring of North Shore Superior Streams Project

(Surface Water Assessment-North Shore; SWANS-NRRI)

Quality Assurance Project Plan

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NRRI Technical Report: NRRI/TR-2008/24



A1. APPROVAL SIGNATURE PAGE

By their signatures below the undersigned attest that they are familiar with the requirements of this document and agree to fulfill their responsibilities as specified herein.


Richard Axler, Project Manager

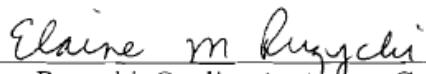
7/21/08
Date


Valerie Brady, Co-Project Manager

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Dan Breneman, Co-Project Manager

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Date


Elaine Ruzycki, Quality Assurance Coordinator

7/21/08
Date


Pam Anderson, Project Manager, MPCA

8/1/08
Date


Roger Fisher, WQ QA/QC Coordinator, MPCA

08.06.08
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Table 1. Acronyms and Abbreviations

APG : Analytical Products Group, Inc., Belpre, OH
CSMP : Citizen Stream Monitoring Partnership
DQO : Data Quality Objective
DI : Deionized
DIN : Dissolved inorganic nitrogen (= [nitrate+nitrite]-N + ammonium-N)
DO : Dissolved Oxygen
%DO : % DO saturation
EC25: specific electrical conductivity (EC normalized to 25°C)
EDA : Environmental Data Access
EPA : Environmental Protection Agency
FD : Field Duplicate
H₂SO₄: Sulfuric Acid
LIMS : Laboratory Information Management System
LSS: *LakeSuperiorStreams* Project (www.lakesuperiorstreams.org)
LSS QAQC: *LakeSuperiorStreams* Project EPA-certified Quality Assurance and Quality Control procedures (see http://duluthstreams.org/streams/QA_QC.html)
μ : Micron or Micro
μg/L : Microgram per liter (= 0.001 mg/L)
μS/cm : Microsiemens per centimeter (EC25 unit)
mg/L : Milligram per liter (=1000 μg/L)
MDH : Minnesota Department of Health
MPCA : Minnesota Pollution Control Agency
Na₂S₂O₃ : Sodium Thiosulfate
NH₄-N : ammonium nitrogen
NIST : National Institute of Standards and Technology
NO₂-N : nitrite nitrogen
NO₃-N : nitrate nitrogen
NRRI-UMD: U. of Minnesota-Duluth Natural Resources Research Institute Central Analytical Lab
NS : North Shore
NTU : Nephelometric Turbidity Units
PM : Project Manager
PO₄-P : phosphate phosphorus (aka: ortho-P [OP], soluble reactive-P [SRP], molybdenum reactive-P [MRP])
QA : Quality Assurance
QAC : Quality Assurance Coordinator
QAM : Quality Assurance Manual
QAPP : Quality Assurance Project Plan
QC : Quality Control
RPD : Relative Percent Difference
RSD : Relative Standard Deviation
SB : Sampler Blank
SLR : St. Louis River
SM : *Standard Methods (for the Examination of Water and Wastewater)*
SOP : Standard Operating Procedure
SWCD : Soil and Water Conservation District
STORET : STORage and RETrieval [federal database]

SU : Standard Unit (e.g. pH units)

SWA-NS : Volunteer Assisted Water Quality and Biological Monitoring of North Shore Superior Streams

TB : Trip Blank

TKN : Total Kjeldahl Nitrogen (total organic-N + total ammonium-N)

TN : Total Nitrogen

TP : Total Phosphorus

TSS : Total Suspended Solids

T-Tube : Transparency tube clarity in units of cms of visibility using a 100 or 120 cm tube.

WLSSD : Western Lake Superior Sanitary District Lab

WQ : Water Quality

DOCUMENT CONTROL

This document has been prepared according to the United States Environmental Protection Agency publication, *EPA Requirements for Quality Assurance Project Plans* (EPA QA/R5, March 2001). This QAPP will be reviewed annually and updated as needed. Updated versions of this QAPP will bear a new (x + 1) revision number. Elaine Ruzycki will assume responsibility for archiving outdated versions of this QAPP which will be kept at project headquarters. Archived versions of this QAPP will be retained for a minimum of ten years from the date of archival.

GROUP A. PROJECT MANAGEMENT

A3. DISTRIBUTION LIST

Each person listed on the Approval Signature Page and each person listed in Table 2 or his/her successor will receive a copy of the final approved version of this Quality Assurance Project Plan. A copy will also be made available to other persons taking part in the project and to other interested parties.

Table 2. SWA-NS Project QAPP Distribution List

Name	Title/Affiliation	Address	Phone/e-mail
Richard Axler	Project Manager, NRRI-UMD	NRRI-UMD 5013 Miller Trunk Highway Duluth, MN 55811	218-720-4316 raxler@nrri.umn.edu
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Roger Fisher	WQ QA/QC Coordinator MPCA	520 Lafayette Road North St. Paul, MN 55155-4194	651-296-7387 roger.fisher@pca.state.mn.us

A4. PROJECT ORGANIZATION

Table 3. SWA-NS Project Personnel

Name/Title	Project Responsibility
Richard Axler, NRRI-UMD Project Manager	Overall Project Administration; Project Decisions; Water Quality Lead; MPCA Liaison; Field and Lab activities. Report write-ups
Valerie Brady, NRRI & Sea Grant-UMD Co-Project Manager	Biological lead; Responsible for post-processing of historical EPA and UMD invertebrate collections and integration with SWANS data into a comprehensive data set. Report write-ups
Dan Breneman, NRRI-UMD Co-Project Manager	Field biological sampling and habitat assessment lead; Invertebrate Lab and field QA/QC, data validation; MPCA Liaison; Coordination of SWANS sampling with other concurrent MPCA-SWA projects, TMDL study surveys, and Fond du Lac Band surveys in 2008.
Jerry Henneck, NRRI-UMD	WQ surveys and Lab Analyses; sample processing and water chemistry analyses; Coordination of SWANS activities with <i>LakeSuperiorStreams.org</i> project and other research projects
Elaine Ruzycski, NRRI-UMD	WQ surveys and Lab Analyses; sample processing and water chemistry analyses; Lab QA/QC Coordinator; STORET data entry
John Ameal, NRRI-UMD	WQ analyses; Lab Manager
Norm Will, NRRI-UMD	Computer programming to facilitate transfer of data files to STORET
Robert Hell, NRRI-UMD	Field sampling for invertebrates and habitat assessments; invertebrate taxonomy and data compilation
Noel Kroening, NRRI-UMD	Field sampling for invertebrates and habitat assessments; invertebrate sorting and taxonomy
Amy Eliot	Citizen scientist/volunteer stream monitoring coordination and training
Pam Anderson, MPCA Project Manager	Technical Assistance, Data Review
Roger Fisher, MPCA WQ QA/QC Coordinator	QA/QC Support

The MPCA QA/QC Coordinator (QAC) is independent from project staff that generate data. The extent of the QAC role is to assist in the writing of this QAPP and to be available to address project QA/QC problems and concerns. The QAC is not accountable to anyone directly or indirectly associated with this project.

Richard Axler and Elaine Ruzycki are responsible for maintaining the latest official approved version of this QAPP.

A5. PROBLEM DEFINITION/BACKGROUND

A5.1 SWA-NS Project Background

An estimated 720 perennial and 127 intermittent streams flow into Lake Superior including 309 trout streams and their tributaries (>2100 miles) along the North Shore (NS) and St. Louis River (SLR) estuary alone. Steep bedrock escarpments create a high density of stream corridors in forested watersheds with steep gradients, thin erodible soils, and typically low productivity, high-quality trout streams sensitive to urbanization and rural development. Limited water quality, habitat and invertebrate community data exist for Minnesota tributaries flowing into Lake Superior and the St. Louis River Estuary, and there are very few years when all three types of data were collected contemporaneously. Visit www.lakesuperiorstreams.org for more details about these streams and consult the project Work Plan for more background information about the project.

A5.2 SWA-NS Project Problem Definition

These streams are particularly susceptible to factors raising water temperature and increasing runoff of water and sediment, such as openings in riparian cover and canopy, impervious surface within the watershed, road crossings, poor construction practices, and the potential increased frequency of severe storms predicted by climate change models and already in evidence during the past decade or so in Minnesota. The streams discharge into the sensitive coastal zone of ultra-oligotrophic Lake Superior or its St. Louis River Estuary.

Urbanization and population have increased along the North Shore over the past 15 years and 11 of the 27 major North Shore (NS) trout streams are now listed as impaired on the 2008 State 303(d) list of impaired waters; primarily for Turbidity, Temperature, and Mercury in Fish Tissue.

A6. PROJECT DESCRIPTION

A6.1 SWA-NS Project Summary

Major project elements are listed below and summarized in Table 4. For further project details consult the project Work Plan.

1. Data, field, and laboratory procedures

- Establish necessary project, laboratory, and station identification forms with MPCA
- Develop QAPP for field and lab procedures
- Develop data forms and interfaces between the NRRI Microsoft Access database, MPCA's EDA and EPA's STORET databases to facilitate historical, current, and future STORET data entry

2. Historical data

- Compile and review relevant historical (post 1996) water quality, invertebrate, and habitat data
- Data entry into STORET from prior UMD-NRRI, UMD-Biology, EPA-MED studies
- Recount historical invertebrate samples to an identical taxonomic level to the current project when appropriate

3. Monitoring current Lake Superior tributary stream water quality

- Sample 12 trout stream sites for one complete water year near mouth or lower watershed at current automated WQ monitoring sites, historical sites, or at previous MPCA sites (see Table 4); this will include approximately 10 higher flow/rain storm sampling events throughout the year and approximately 10 lower flow surveys distributed across the year.
- Parameters will include a Core Suite of field measurements (*flow, temperature, dissolved oxygen, specific electrical conductivity-EC25, pH, transparency tube clarity and CSMP visual qualitative indices*);
- Parameters will include an Advanced Suite of water chemistry parameters (*turbidity, TSS, TP, TN, TN, NO₃/2-N, NH₄-N, TKN, Cl, color*), and pathogen indicator bacteria (*E. coli*)

4. Develop and implement volunteer CSMP+ monitoring (i.e. MPCA-CSMP protocols with additional parameters) at 12 Superior trout streams

- Select volunteers in teams where possible and with the intent of maintaining data collection indefinitely
- Train CSMP+ volunteers and initiate sampling in spring 2008
- Establish field protocols consistent with MPCA impaired waters assessment guidelines
- Parameters to include: *transparency tube clarity and CSMP visual qualitative indices; EC25 and temperature; turbidity from grab samples (lab-NRRI)*

5. Assess the current status of Lake Superior trout stream macroinvertebrate ("bugs") and habitats

- Establish field protocols consistent with MPCA's new impaired waters biological assessment guidelines
- Sites selected to be paired with current or historic WQ site locations (13 total)

Table 4. Overall SWA-NS project summary.				
Water Bodies & Sites	Proposed Streams	# of samples	Parameters	Date Range
<p><u>Obj 1– Historical “Bugs” & WQ from North Shore L. Superior Streams</u></p> <p>Data compilation and entry into STORET from prior UMD-NRRI, UMD-Biology, EPA-MED studies</p>	<p>Amity, Baptism, Beaver, EB Beaver, Caribou, Cascade, Cross, Encampment, French, “Unk”, W.Knife, Lester (2), Onion, Palisade, Skunk, Sucker, Talmadge, Temperance, Blind Temperance, Two Island; Chester, Tischer, Miller, Skunk, McCarthy, Knife, E Split Rock, W.Branch Knife, Little Knife;</p>	<p>EPA, NRRI, UMD methodology similar to current NRRI methods and those specified by MPCA for biological stream assessments and TMDLs. Some recounts will be needed</p>	<p><i>Q, temp, DO, pH, EC25, turbidity, TSS, TP, ortho-P, TN, TKN, NO3/2-N, NH4-N, Cl, DOC, POC, major anions and cations</i> for EPA samples; UMD-NRRI & UMD-Biology studies included: <i>TN, TP, DIN, ortho-P (sometimes), temp, DO, EC25 & pH z(sometimes); limited anion-cations; some TSS & turbidity.</i></p>	<p>Seasonal (Spring to Fall) for WQ; during Summer base flow for invertebrates;</p> <p>Late 1990’s through 2003</p>
<p><u>Obj 2 - Current Stream Water Quality</u></p> <p>12 Stream sites sampled near mouth or lower watershed at current automated WQ monitoring sites, historical sites, or at previous MPCA sites</p>	<p>3- <u>DuluthStreams.org sites</u>: <i>Tischer, Kingsbury, Chester</i> Creeks; 3- <u>State Parks</u>: <i>Gooseberry, Split Rock, Cascade</i>; 3-<u>MPCA Superior Basin Priorities</u>: <i>Beaver, Caribou, Encampment</i>; 2-<u>MPCA Spec Interest</u>: <i>Sugarloaf, Simian</i> 1- <u>NRRI-Mitigation priority site</u> –<i>Upper Amity Cr. above Impairment Listing near confluence of East & West Branch Amity.</i></p>	<p>n_≥20 discrete grabs (site-visits); weighted to provide >50% of samples during high-flow storm & spring runoff periods as per MPCA (2003; 2007 assessment guidelines)</p>	<p><i>Q, temp, DO, pH, EC25, transparency tube, turbidity, TSS, TP, TN, TKN, NO3/2-N, NH4-N, Cl, color; CSMP qualitative indices; E. coli indicator bacteria</i></p>	<p>Snowmelt runoff until ice up; Snowmelt 2008 (~ March) through Winter 2009 – depending on hydrologic conditions and sampling success</p>
<p><u>Obj 2 – Stream Water Quality CSMP+</u> (n=12)</p>	<p>3- <u>State Parks</u>: <i>Gooseberry, Split Rock, Cascade</i>; 4- <u>MPCA Superior Basin Priorities</u>: <i>Beaver, Caribou, Encampment, Simian</i>; 1- <u>NRRI-Mitigation site</u> –<i>Upper Amity</i>; 3 –“<u>New</u>” designated trout streams in <u>Duluth</u> (<i>Buckingham, Coffee, Knowlton</i>); 1- <u>Tischer @ Mouth</u>– Duluth Trout stream with upstream monitoring</p>	<p>n_≥20 for CSMP-Plus</p> <p>weekly + storm “chasing”; ~10 low flow DOs</p>	<p>CSMP t-tubes; EC25 and /temp using calibrated portable conductivity “pens”; DO (during low summer base flows); semi-quantitative flow/stage height as per CSMP protocols (the sites are being called CSMP+; note – not sampled for nutrients)</p>	<p>Snowmelt runoff 2008 to ice up in Winter 08/09. Expectation to maintain CSMP level monitoring indefinitely</p>

Table 4. Overall SWA-NS project summary (continued)				
Water Bodies & Sites	Proposed Streams	# of samples	Parameters	Date Range
<p><u>Obj 3 –Macroinvertebrate (Bugs) sampling and habitat assessments</u></p> <p>13 Bug sites selected to be paired with current or historic WQ site locations</p>	<p>4- <u>MPCA “Superior Loading (Anderson et al. 2003)”</u>: <i>Sucker, French, Talmadge, Brule</i></p> <p>1- <u>Proposed Sentinel</u>: <i>Baptism</i>;</p> <p>1- <u>Milestone</u>: <i>Beaver (not for turbidity impairment but for pairing biota to WQ)</i>;</p> <p>3- <u>DuluthStreams.org urban</u>: <i>Tischer, Kingsbury, Chester</i>;</p> <p>3- <u>MPCA Superior Basin Priority</u>: <i>Caribou, Encampment, Flute Reed</i></p> <p>1- <u>NRRI-Mitigation priority site</u> –<i>Upper Amity Cr. above Impairment Listing near the confluence of the East and West Branch of Amity.</i></p>	<p>1 per reach/stream, sampling run, riffle, & pool habitats</p> <p>Samplers will vary depending upon substrate: Surber/Hess; Ekman grab/core; D-nets for bank, riparian vegetation, debris dams and wood, boulders, rip-rap, and run-vegetation</p>	<p>Invertebrates (quantitative & qualitative metrics)</p> <p>Stream & riparian QHEI (as per MPCA/EPA TMDL QAPPs developed by NRRI for MPCA, Benton, South St. Louis and Cook Counties in 2006 and 2007).</p> <p>The NRRI parameter list/template for biodata is being adapted to conform to the Minnesota Stream Habitat Assessment</p>	<p>Summer base flow; 2008</p> <p>Because of the potential for summer drought to prevent sampling during the standard summer baseflow period in Jul/Aug, an initial sampling will be performed after Spring runoff in early June. Only 1 set will be enumerated</p>

A6.2 SWA-NS Project Goal

The overall project goal is to develop complementary (same year) physical, biological, and chemical data sets for a range of agency – prioritized streams to process and/or incorporate historical, but modern, biological data into the overall state database.

Major objectives are:

- To screen, analyze, and entered into STORET/EDA, historical water quality and invertebrate data from ~ 30 NS stream sites sampled from the late 1990s through 2007.
- Sample 12 Superior Basin streams intensively for flow, sediments (TSS, turbidity, clarity), nutrients, pathogen indicator bacteria and other parameters and establish enhanced (+turbidity and EC25, and summer DO) Citizen Stream Monitoring Partnership (CSMP) sites in 12 priority trout streams.
- Sample benthic invertebrates at 13 Superior Basin stream sites coordinated with 2008/2009 water quality sampling for North Shore and St. Louis River (SLR) tributaries.

A6.3 SWA-NS Project Milestone Schedule

Table 5. SWA-NS Project Milestone Schedule 2008-2009

Tasks	J	F	M	A	M	J	J	A	S	O	N	D
Select and Train Volunteer Lake Monitors (2008)				•	•	•	•			•		
Volunteer and Advanced Sampling (2008)					•	•	•	•	•	•	•	•
Volunteer and Advanced Sampling (2009)	•	•	•	•	•	•						
Invertebrate Sampling (2008)					•	•	•	•				
Invertebrate Sampling (2009)	Only if needed											
Data Review and Assessment (2008)				•	•	•	•	•	•	•	•	•
Data Review and Assessment (2009)	•	•	•	•	•	•						
Data Submittal for Entry into STORET (2008)							•	•	•	•	•	•
Data Submittal for Entry into STORET (2009)	•	•	•	•	•	•						

A6.4 Samples for Laboratory Analysis

Water quality samples will be analyzed for various parameters at three State-certified Water Quality Laboratories with the majority being analyzed at NRRI-UMD’s Central Analytical Lab: *E. coli* Bacteria, total phosphorus, ammonia nitrogen, total Kjeldahl nitrogen (calculation), nitrate + nitrite nitrogen, total nitrogen, total suspended solids, turbidity, chloride, and color. The distribution of samples by laboratory is detailed in Tables 6-8.

Table 6. NRRI-UMD Analyses & Methods

Laboratory Information Form for Projects Submitting Data to MPCA STORET								Today's Date 5/27/2008		
Bold = entry required								Monitoring Year(s) 2008-2009		
Project Name <u>Volunteer assisted water quality/biological monitoring of North Shore Superior streams</u>										
Project Code (Project_ID) (8-digit ID code assigned by MPCA STORET Staff)										
Lab ID (assigned by MPCA STORET staff)										
Laboratory Name: Natural Resources Research Institute Central Analytical Laboratory										
Address: 5013 Miller Trunk Highway, Duluth, MN 55811										
Contact Name: Elaine M Ruzycki										
Phone: 218-720-4337 Fax 218-720-4328 E-mail eruzycski@nrri.umn.edu										
<u>Citation for Laboratory Manual or Handbook</u>										
Title: Analytical chemistry and quality assurance procedures for natural water, wastewater, and sediment samples.										
Author: Ameel, J., E. Ruzycki, C.J. Owen and R. Axler. Publishing Organization: Natural Resources Research Institute, University of Minnesota, Duluth										
Publication Year: 1998 (revised 2003). Volume and Page No: Technical Report NRRI/TR-98/28.										
<u>Analyses and Methods</u>										
Note: Table below reflects methods, limits, and certifications of lab for the year(s) in which the data were collected. Please include analyte information applicable to the monitoring year indicated in the first column.										
Monitoring Year	Analyte Name	Sample Fraction	Reporting Units	Comparable Standard Method	Fld. Preservation Method	Detect. Limit	Report. Limit	Lab Certified For Analyte	Duration Basis	Temperature Basis
2008-9	Chloride	filtered	mg/L	SM 4500-Cl E 97	Chill to 4°C	0.05 mg/L		Yes		
2008-9	Conductivity	total	µS/cm	SM 2510 B-97	Field measure	1 µS/cm		Yes		@25°C
2008-9	Residue, non-filterable (TSS)	total	mg/L	SM 2540 D-47	Chill to 4°C	5 mg/L		Yes		
2008-9	turbidity	total	NTRU	SM 2130 B-01	Chill to 4°C	0.4 NTRU		Yes		

Table 6 (continued). NRRI-UMD Analyses & Methods

Monitoring Year	Analyte Name	Sample Fraction	Reporting Units	Comparable Standard Method	Fld. Preservation Method	Detect. Limit	Report. Limit	Lab Certified For Analyte	Duration Basis	Temperature Basis
2008-9	phosphorus	total	mg/L	EPA 365.3	Chill to 4°C	0.02 mg/L		Yes		
2008-9	nitrogen	total	mg/L	SM 4500-P and 4500-NO ₃ ⁻	Chill to 4°C	0.01 mg/L		Yes		
2008-9	Nitrate + nitrite, as N	filtered	mg/L	SM 4500-NO ₃ ⁻	Chill to 4°C	0.002 mg/L		Yes		
2008-9	TKN (= TN - [(NO ₃ ⁻ + NO ₂ ⁻)-N])	calculated	mg/L	SM 4500-P and SM 4500-NO ₃ ⁻	Chill to 4°C	0.01 mg/L		No & Yes see *		
<p>TKN & TN: The NRRI-UMD Lab does not use the TKN method certified by the MN Dept. of Health but instead uses a more sensitive, and more limnologically accepted method for total-nitrogen (TN), that is listed in APHA Standard Methods (SM 4500-P J since 2000) and has been certified by the USGS since 2006 (USGS Water Investigations Report 03-4174 (http://nwql.usgs.gov/Public/pubs/WRIR03-4174/WRIR03-4174.html) by Charles Patton and Jennifer Kryskalla). We can calculate TKN, if that is desired (although it never is for the northern lakes and streams that we research) as TKN = TN - [(NO₃⁻ + NO₂⁻)-N] and we routinely pass the Blind Sample Audit using Certified Water Quality Standards. We can report this calculated TKN along with our direct measurement of TN.</p>										
2008-9	Ammonia, as N	filtered	mg/L	SM 4500-NH ₃ G (Auto)-97	Chill to 4°C	0.002 mg/L		Yes		
2008-9	True color	filtered	PT-Co units	SM 2120 C	Chill to 4°C	5 color units		no		
2008-9	pH	total	Standard units	SM 4500 H ⁺ B-00	Chill to 4°C	0.1 unit		yes		ambient
2008-9	Dissolved oxygen	total	mg/L	SM 4500-O G.	Field measure	0.3 mg/L		no		ambient

Table 7. WLSSD Analyses & Methods

Laboratory Information Form for Projects Submitting Data to MPCA STORET							Today's Date 5/15/2008			
Bold = entry required							Monitoring Year(s) 2008-2009			
Project Name <u>Volunteer assisted water quality/biological monitoring of North Shore Superior streams</u>										
Project Code (Project_ID)		<small>(8-digit ID code assigned by MPCA STORET Staff)</small>								
Lab ID <small>(assigned by MPCA STORET staff)</small>										
Laboratory Name: Western Lake Superior Sanitary District										
Address: 2626 Courtland Street, Duluth, MN										
Contact Name: Joseph Mayasich										
Phone 218-22-3336 ext 333 Fax E-mail										
<u>Citation for Laboratory Manual or Handbook</u>										
Title: Standard Methods for the Examination of Water and Wastewater 21 st Ed					Publishing Organization: American Public Health Association					
Publication Year: 2005			Volume and Page No.							
<u>Analyses and Methods</u>										
Note: Table below reflects methods, limits, and certifications of lab for the year(s) in which the data were collected. Please include analyte information applicable to the monitoring year indicated in the first column.										
Monitoring Year	Analyte Name	Sample Fraction	Reporting Units	Comparable Standard Method	Fld. Preservation Method	Detect. Limit	Report. Limit	Lab Certified For Analyte	Duration Basis	Temperature Basis
2008-9	Chloride	filtered	mg/L	SM 4500-Cl ⁻ C 97	Chill to 4°C	1 mg/L		Yes		
2008-9	Residue, non-filterable (TSS)	total	mg/L	SM 2540 D-47	Chill to 4°C	1 mg/L		Yes		
2008-9	phosphorus	total	mg/L	EPA 365.3	Chill to 4°C	0.01 mg/l		Yes		

Table 8. Northshore Analytical, Inc. Analyses & Methods

Laboratory Information Form for Projects Submitting Data to MPCA STORET								Today's Date 5/20/2008		
Bold = entry required								Monitoring Year(s) 2008-2009		
Project Name <u>Volunteer assisted water quality/biological monitoring of North Shore Superior streams</u>										
Project Code (Project_ID) (8-digit ID code assigned by MPCA STORET Staff)										
Lab ID (assigned by MPCA STORET staff)										
Laboratory Name: North Shore Analytical, Inc.										
Address: 4511 W. 1 st Street, Suite #1, Duluth, MN 55807										
Contact Name: Linda Christensen										
Phone: 218-729-4658 Fax 218-729-4659 E-mail: info@northshoreanalytical.com										
<u>Citation for Laboratory Manual or Handbook</u>										
Title: Determination of <i>E. coli</i> Bacteria in Surface Waters by Colilert-18 & Quanti-Tray 2000										
Author: L. Christensen and C. Gross. Publishing Organization: North Shore Analytical, INC										
Publication Year: 2008 Volume and Page No.										
<u>Analyses and Methods</u>										
Note: Table below reflects methods, limits, and certifications of lab for the year(s) in which the data were collected. Please include analyte information applicable to the monitoring year indicated in the first column.										
Monitoring Year	Analyte Name	Sample Fraction	Reporting Units	Comparable Standard Method	Fld. Preservation Method	Detect. Limit	Report. Limit	Lab Certified For Analyte	Duration Basis	Temperature Basis
2008-9	Escherichia coli	total	MPN	Colierr®-18 Quanti-Tray®	Chill @4		<1 to 2419.6 MPN	Yes		

A6.5 Samples for Field Analysis

The following parameters will be measured in the field through use of meter(s), a multi-probe, or other instrument(s):

- Temperature
- Dissolved Oxygen (concentration and % saturation)
- pH (field and Lab)
- Specific Electrical Conductivity (EC25)
- Transparency tube clarity (100-120 cm)
- Stream Velocity (measured) & Flow (estimated)

Methodology references and data quality objectives (DQOs) for field parameters, with the exception of flow, are provided in Table 9. Additional field Standard Operating Procedures (SOPs) are listed in the Appendices. Flow (stream discharge) will be estimated in several ways described below.

Flow

1. Sites with gauging stations (SWA-NS: macroinvertebrate and habitat assessments):

Three Duluth trout stream sites (Tischer, Chester, Kingsbury Creeks) are monitored for flow on a continuous basis as described in the www.DuluthStreams.org project QAPP (http://duluthstreams.org/streams/QA_QC.html). In addition, the MPCA in collaboration with the USGS operates flow gauging sites on Amity Creek (lower), Sucker R., French R., Talmadge R., Brule R. and the Baptism R.) and these data and DQOs are available from either agency.

Flow will be independently measured as part of the habitat assessments in early-mid summer from velocity measurements at 5 points on each transect to provide a description of flow characteristics. Variability in flow among different substrate size classes (e.g., cobble, boulders, etc.) and in-stream cover types (e.g., woody debris, undercut banks, etc.) will help formulate a view of conditions in a habitat context. Water depth will be recorded at each transect point along with velocity measurements at a point equivalent to 60% of total water depth. Discharge will be estimated following instructions for flow-weighted averaging (FWA) provided in the Marsh-McBirney Flowmate Operators Manual (Marsh-McBirney 1990). This is a standard procedure used by NRRI-UMD for TMDL related assessments of North Shore streams (Knife, Poplar, Miller) that has been approved by MPCA and EPA (citations below).

- *Marsh-McBirney Inc. 1990. Flow-Mate Model 2000 Installation and Operations Manual. Marsh-McBirney Inc. Fredrick, MD*
- *Breneman, D., V. Brady, and L. Johnson. 2007. Cook County Soil & Water Conservation District Biological Sampling for the Poplar River TMDL Quality Assurance Project Plan. NRRI/TR-2007/16, Natural Resources Research Institute, U. of Minnesota Duluth, 5013 Miller Trunk Highway, Duluth, MN 55811-1442*

2. Sites without gauging stations & monitored for macroinvertebrates, habitat, and water quality.

Both the midsummer “bug”/habitat surveys and the intensive (*Core + Advanced Suite* parameters) water quality surveys will determine water velocity using a field flowmeter to estimate flow as per the procedure described above.

3. Volunteer monitoring sites

Quantitative flow measurements are beyond the scope of this component of the project. Instead a qualitative, visual estimate (Low, Normal, High) and when possible, a tape down measurement made with a weighted steel measuring tape from a mark made on the rail of a bridge or culvert to the water surface will be done as per the MPCA's CSMP program

(www.pca.state.mn.us/water/csmp.html). In addition, we will attempt to install staff gauges (large metal rulers) in nearby permanent locations where stream dimensions are known and are unlikely to change (i.e. bridges and culverts). Stage height readings could be later converted to flow if a stage-discharge relationship could be developed. Data on precipitation amounts will also be collected where it is possible to install a secure CSMP rain gauge to help interpret flow and other water quality data. More accurate precipitation data will be reported on sampling days from the nearest National Weather Service or City of Duluth/WLSSD precipitation gauge sites.

A6.6 Field collections of benthic macroinvertebrates, taxonomic identification, and field habitat assessments

All field and lab measurements and data analysis will follow the same procedures used by NRRI-UMD, and approved by MPCA and EPA, for the recent TMDL studies of Superior Basin trout streams - Knife River, Poplar River, and Miller Creek (Breneman et al. 2007 cited above). These methods are fundamentally identical to those used by NRRI-UMD for other North Shore stream studies in the past and by the EPA-Mid Continent Ecology Lab (Duluth) in studies of North Shore streams from 1996-1998.

A7. QUALITY OBJECTIVES AND CRITERIA

Virtually all environmental data are only approximations of the true values of the parameters measured. These estimates are affected by the variability of the medium being sampled and by random and systematic errors introduced during the sampling and analytical procedures.

Data Quality Objectives (DQOs) are qualitative or quantitative statements of:

- Precision (a measure of random error)
- Bias (a measure of systematic error)
- Accuracy
- Representativeness
- Completeness,
- Comparability, and
- Sensitivity

The DQOs must be defined in the context of project requirements and objectives, not the test method capabilities.

Precision – This quality element measures how much two or more data values are in agreement with each other. Precision is discussed in the introductory chapter of *Standard Methods for the Examination of Water and Wastewater*, 20th Edition, 1998. Field sampling precision is

determined by using field split samples or field duplicate samples. Laboratory analytical precision is determined by comparing the results of split samples, duplicate samples, and duplicate spike samples.

Parameter	Precision (≤ % RPD)	Range	Method Detection Limit	Reporting Limits	Units	Holding Times
<i>E. coli</i> bacteria	30%* (guestimate)	0 – 50,000* (approx.)	<5*	<1 to 2420*	MPN/100 mL	24 hr **
Total Phosphorus	15%	0.002 – 1.0	<0.002	±0.001	mg/L	28 D
Ammonia Nitrogen	15%	0.002 – 1	<0.002	±0.001	mg/L	28 D
Total Kjeldahl Nitrogen (calc)	15%	0.01 – 2	<0.010	±0.001	mg/L	28 D
Nitrate + Nitrite Nitrogen	15%	0.002 – 1	<0.002	±0.001	mg/L	28 D
Total Nitrogen	15%	0.01 – 2	<0.010	±0.001	mg/L	28 D
Total Suspended Solids	20%	1 – 20,000	<5	±1	mg/L	7 D
Chloride	15%	0.1 – 200	<0.05	±0.1	mg/L	28 D
Color	10%	1 – 500	<5	5	Color Units	2 D
Turbidity	10%	0 – 4,000	0.4	±1	‡NTRU or NTU	2 D
Dissolved Oxygen†	±0.3 mg/L	0.1 – >20	<0.3	±0.1	mg/L	†
Spec. Conductivity (EC25) †	10%	10 – 2,000	1	±1	µS/cm	†
pH	[0.1 Units]	4 – 10	<0.1	±0.1	Std Units	24 hr
Temperature†	±0.2 - 0.5	0 – 25	<0.1	±0.1	°C	†
flow/stage height	See below	See below	NA	See below	cfs, m3/s, cm	†
Transparency Tube†	TBD ~10%	0 – 100	TBD (~1-2 cm)	1	cm	†

* depends on concentration and dilution: **8 hours if used for enforcement purposes; †Depends upon the optical configuration of the meter ; ‡Field measurement.

Sampling and/or analytical precision may be determined from split or duplicate samples by calculating the Relative Percent Difference (RPD) as follows:

$$RPD = (A - B) \div ((A + B) / 2) \times 100$$

where A is the larger of the two duplicate sample values and B is the smaller value.

Where three or more replicate samples or measurements have been taken, calculate the Relative Standard Deviation (RSD) instead of the RPD as follows:

$$\text{RSD} = (s/\bar{x}) \times 100 \text{ (also called the CV, Coefficient of Variation)}$$

where s is the standard deviation of the replicate values and \bar{x} is the mean of the replicate values.

Bias – This expresses the degree to which a measured value agrees with or differs from an accepted reference (standard) value due to systematic errors. Field bias should be assessed by use of field blanks and trip blanks. Adherence to proper sample handling, preservation, and holding time protocols will help minimize field bias.

Since the sampling method for all sampling will be grab sampling, no field blanks (sampler blanks) will be taken. Trip blanks are required for certain MPCA funded projects involving VOC (Volatile Organic Carbon) sampling which is not a parameter to be measured by this project. Thus bias due to field activities will not be determined. However, the Project Manager has conducted numerous research projects of this kind and his group's field collection and bottle preparation methods are well tested from previous MPCA, DNR, and EPA funded projects. No significant bias that could be corrected by collecting and analyzing field blanks is anticipated. However, laboratory bias will be determined as part of its internal quality control. Bias effects that fall outside the laboratory's acceptance limits will be flagged.

Accuracy – This expresses the degree to which an observed (measured) value agrees with an accepted reference standard (certified sample value) or differs from it due to systematic errors. NRRI analyses certified, *blind* performance standards for nutrients and other conventional water quality parameters annually as a condition for its MN Department of Health Certification. EPA and/or USGS approved methods are used for calibrating field instruments. Details can be found at http://www.duluthstreams.org/streams/QA_QC.html

Completeness – Expressed as the number of valid (usable) data points made to the total number of measurements expected according to the original sampling plan. Percent completeness is determined separately for each parameter and is calculated as follows:

$$\% \text{ Completeness} = (\text{no. of usable data points} \div \text{no. of planned data points}) \times 100$$

High or low water levels may reduce the number of samples that can be taken. A lack of major rainstorms and/or a reduced period of Spring snow melt runoff can also prevent the targeted percentage of high flow events from being collected. To some extent this may be compensated for by scheduling additional sampling events or sampling as near to the original sampling site as possible. Any such variances to the established sampling protocol will be thoroughly documented. Resulting data will also be qualified to reflect this.

Representativeness – the degree to which data accurately and precisely represents parameter variations at a sampling point, or of a process or environmental condition. Representativeness of

field data are dependent upon proper sampling program design and is maximized by following the sampling plan, using proper sampling protocols, and observing sample holding times.

Data will also be routinely screened for representativeness by comparing measured parameter values to historical project data and to current and historical data generated by other organizations when available. However, much of the data collected will essentially be “new” data for these streams and so project staff will rely on their limnological experience with other regional trout streams to identify anomalies should they occur.

Comparability –the level of confidence with which the project data can be compared to other data. Comparability is dependent upon establishing similar QA objectives for the sets being compared and is achieved by using similar sampling and analytical methods. **Sensitivity** – For laboratory analyses this represents the lowest level of analyte that can be reliably detected by the laboratory analytical method. For field measurements this represents the lowest level of analyte the field analytical method or meter can reliably detect.

Analyte Name	Minimum Volume (mL)	Container Type	Comparable Standard Method	Field Preservation	Preservation Method	U.S. EPA Recommended Holding Time ¹
Chloride	100	plastic	SM 4500-Cl ⁻ E 97	Chill to <4°C	refrigerate (4°C)	28 days
Specific Conductivity (EC25)	field	--	SM 2510 B-97	Field measure	refrigerate (4°C)	28 days
Residue, non-filterable (TSS)	200	plastic	SM 2540 D-47	Chill to <4°C	refrigerate (4°C)	7 days
Turbidity	100	plastic	SM 2130 B-01	Chill to <4°C	refrigerate (4°C)	24 hours
Total Phosphorus	100	plastic	EPA 365.3	Chill to <4°C	freeze (-20°C)	28 days
Total Nitrogen	100	plastic	SM 4500-P and 4500-NO ₃ ⁻	Chill to <4°C	freeze (-20°C)	28 days
Nitrate + nitrite (-N)	100	plastic	SM 4500-NO ₃ ⁻	Chill to <4°C	freeze (-20°C)	28 days
TKN (TN - [NO ₃ + NO ₂]-N)	calculated	plastic	SM 4500-P and SM 4500-NO ₃ ⁻	Chill to <4°C	freeze (-20°C)	28 days
Ammonia, as N	100	plastic	SM 4500-NH ₃ G (Auto)-97	Chill to <4°C	freeze (-20°C)	28 days
True color	100	plastic	SM 2120 C	Chill to <4°C	refrigerate (4°C)	48 hours
pH	200	Std units	SM 4500 H ⁺ B-00	Chill to <4°C	refrigerate (4°C)	8 hours
Dissolved oxygen	field	mg/L	SM 4500-O G.	--	--	na
<i>Escherichia coli</i>	125	Sterile plastic	Colilert®-18 Quanti-Tray®	Chill to <4°C	refrigerate (4°C)	8 hours (enforcement) 24 hrs (SWA-NS surveys)

¹ According to the EPA, samples properly preserved may be held for extended periods beyond the recommended holding time.

A8. SPECIAL TRAINING/CERTIFICATION

Training of SWA-NS Project staff, when needed, is done internally at NRRI-UMD through assistance from knowledgeable SWA-NS Project staff. All project staff are experienced with all aspects of the field sampling for this project.

Citizen scientists and other volunteer monitors will be also be trained by NRRI staff in concert with an ongoing collaboration with the Project Manager has with volunteer stream monitoring being done in Wisconsin (the WAV Program) that is also being coordinated with Lauri Sovelle's MPCA-CSMP effort. The CSMP+ sampling being done for SWA-NS incorporates water sampling with standard CSMP measurements (chiefly T-Tubes); the temperature and EC25 of the water is then measured in the field with a hand-held portable meter, and a water sample is collected and sent to NRRI for turbidity. All measurements are therefore derived from a common bucket of water.

Richard Axler, Dan Breneman and Valerie Brady as Project Managers are responsible for ensuring key project staff have or receive adequate training to effectively and correctly perform their project duties. They are also responsible for documenting such training as necessary and maintaining the training records.

A9. DOCUMENTATION AND RECORDS

All versions of the QAPP are retained at the Sponsored Projects Administration Center office. SWA-NS Project staff retain field sampling sheets indefinitely (>10 yrs). Data are entered into STORET by MPCA staff. Field sampling sheets are completed on-site at the time of sampling and data entered into electronic spreadsheets as soon as possible after sampling (usually <48 hrs). Sampling collection records, field notebooks, and all records of field activity are retained by the SWA-NS Project staff indefinitely (>10 years) following completion of the project. Volunteer monitoring data will also be submitted directly to the MPCA-CSMP program as per the program's standard protocols.

GROUP B. DATA GENERATION AND ACQUISITION

B1. SAMPLING DESIGN

The SWA-NS Project Manager and Co-Managers staff developed the sampling plan in consultation with MPCA, DNR, S. St. Louis County and Cook County SWCD staff.

Water chemistry, biological, and physical data are collected and used to assess stream condition. Samples for water quality taken during the project are considered to be a representative *yearlong snapshot* of current water quality conditions for the climatic condition of the current water year beginning with snowmelt runoff in Spring 2008. Sampling frequency follows required MPCA protocols:

- *MPCA Guidance Manual for Assessing the Quality of Minnesota Surface Waters for the Determination of Impairment 305(b) Report and 303(d) List (October 2007; <http://www.pca.state.mn.us/publications/wq-iw1-04.pdf>) and*
- *Anderson, J. M. Evenson, T. Estabrooks and B. Wilson. 2003. An assessment of representative Lake Superior Basin tributaries. Stream Water Quality Assessment Technical Report Series. Minnesota Pollution Control Agency, St. Paul, MN.55155. (<http://www.pca.state.mn.us/publications/reports/ls-tributarystreamassessment-2002.pdf>)*
- For the intensive water quality sampling effort (Obj 2- Current Stream Water Quality in Table 4) at least 20 samples will be collected over a year-long period with ≥ 10 from high flow periods split between snowmelt runoff and storm events.
- For the volunteer water quality monitoring effort (For the intensive water quality sampling effort (Obj 2 - Stream Water Quality CSMP+ in Table 4), sampling frequency will attempt to follow a weekly schedule with event-based sampling encouraged.
- Macroinvertebrate sampling and habitat surveys will be performed in early – mid-summer to maximize taxonomic diversity. Co-Project Manager Dan Breneman has worked with MPCA’s Joel Chirhart to ensure consistency with evolving MPCA protocols for stream bioassessment and habitat assessment.

B2. SAMPLING METHODS

All field work for this project, including water sample collection and delivery within the required time frame to the NRRI-UMD, WLSSD, and Northshore Analytical, Inc. Laboratories is either conducted by SWA-NS staff, or in the case of volunteer monitoring, closely coordinated by NRRI-UMD staff after training. All three laboratories are certified for their analyses by the MN Department of Health. This QAPP supports the laboratory’s QAM and SOPs and is specific for the SWA-NS Project.

1. Intensive and volunteer water quality

Field duplicates are collected 10% of the time and all samples are collected using approved methods and sampling devices. Samples are transferred from sample collection devices to pre-cleaned polyethylene bottles when necessary. Bacteriological samples are collected in sterile polypropylene or polycarbonate bottles supplied by Northshore Analytical, Inc. in the same manner as for MPCA’s Lake Superior Beach Monitoring Program.

Physical parameters are assessed on-site by use of multi-probe water quality instruments calibrated as per USGS and EPA standard methods (see http://www.duluthstreams.org/streams/QA_QC.html). Transparency tube measurements follow the method recommended by MPCA’s Citizen Stream

Monitoring Program (CSMP; <http://www.pca.state.mn.us/water/csmp.html>). Duplicates will be determined at each sampling to estimate precision.

Citizen scientist volunteers will collect a single water sample for turbidity analysis in the NRRI Lab. Water will be collected as per NRRI staff collections using a reach pole with 1 L plastic sampling bottle. At least 3 samples will be pooled in a 2 gallon clean plastic bucket. Water is swirled and agitated and then a clean 125 mL polyethylene supplied by NRRI-UMD is submersed and chilled on ice until it can be refrigerated. The EC25 of the remaining water is then determined by direct immersion of a handheld portable conductivity “pen” (Hanna Pocket Conductivity/TDS/temperature Tester -DiST 5 Hanna Scientific, Catalogue # 88230. These small, inexpensive meters (~ \$90) have been used in our Lab for the past 5 years and distributed to schools for testing local streams; they have shown excellent agreement with much more expensive multiprobe sondes and sensors from our *in situ* stream monitoring units gauges. Each conductivity “pen” will be numbered and calibrated at the start of the sampling season and then rechecked seasonally.

During initial training, multiple measurements of each parameter determined by volunteers will be made to estimate precision for each sampler as well as overall volunteer precision. This will be especially useful during group trainings.

Dissolved oxygen will be determined by NRRI-UMD staff using either a YSI 85 meter or a Hydrolab Minisonde MS5 multiprobe sonde. Instrument calibrations follow manufacturer recommendations and DO calibrations are done by air calibration after estimating elevation and barometric pressure. There are cost and logistical difficulties associated with having citizen scientists measure DO using multimeters and many chemical “kits” are not accurate enough for assessment purposes. Superior region streams DO values are usually expected to be high, but there is increasing concern by agency and academic fisheries experts about summer DO levels due to a number of unusually warm, dry summers in recent years. Therefore, we are purchasing three Hach Digital DO titrators which are EPA approved and purported to be accurate and reliable (Dissolved Oxygen Test Kit, Model OX-DT, Digital; Hach Product #: 2063100, US Price: \$238). Reported QA values are shown below (from www.hach.com) but as part of this project we will determine a Method Detection Limit and precision for the instrument and cross-compare them to our field meters:

- Range: 1-10 mg/L
- Smallest Increment (mg/L) 0.02-0.2 (Greater sensitivity {0.01 mg/L})

Sampling will focus on the late summer and fall base flow period, depending on rainfall events and flows, when low dissolved oxygen concentrations are most likely to occur. Water will be collected by immersing a 300 mL glass BOD bottle pointing it upstream of the sampler and ensuring that all air bubbles are displaced when inserting the pointed ground glass stopper. The bottle will be chilled on ice and taken home. The Winkler titration will be performed as soon as the sampler arrives home and will follow the instructions in the Hach Digital Titrator Model 16900 Manual (June 2006;

http://www.hach.com/fmmimghach?/CODE%3A1690008_24ED-210509%7C1). Site locale

will be scouted to determine if there are nearby pools that might exhibit lower DO values than riffle areas and if so a second sample will be collected.

Grab sampling

Water quality samples are collected using clean polyethylene bottles of appropriate size to provide the laboratory with sufficient sample to perform the requested analyses and re-analysis, if necessary. Multiple grab samples are collected via a one liter wide mouthed polyethylene bottle affixed to a telescoping sampling pole. Samples are depth and transverse integrated to the extent possible based on flow conditions and multiple samples pooled into a clean, ~ 10 L plastic sampling bucket or carboy. After mixing, 4 L cubitainer is filled, capped and iced for transport to NRRI. Remaining water is re-mixed and used to estimate transparency tube clarity. *E. coli* bacteria samples are collected separately in sterile polypropylene or polycarbonate bottles supplied by Northshore Analytical, Inc. in the same manner as for MPCA's Lake Superior Beach Monitoring Program. Sample containers are immersed upstream of where the sampler is positioned in all cases. All samples are labeled with a unique identifier, date and sampling time and placed in a cooler on ice. Sample information is logged on field data sheets.

Each sampling attempts to obtain a representative and well mixed sample although the method used for any particular sampling event depended on several factors including flow rate, stream depth and width, and accessibility. NRRI staff has been out in the field on North Shore streams a number of times over the past six years with MPCA-Duluth staff to ensure similar survey methods. For additional information on the grab sampling method see Appendix A. Variations of the grab sampling method which may be used as needed are described below by the MPCA Water Quality QA/QC Coordinator. Our intent is to collect an integrated and representative sample to the extent possible at each of the intensive WQ surveys. This will involve collecting 4-5 one liter samples systematically collected while moving the sampler across the stream and simultaneously up-and-down the water column and pooling them all to generate a common sample that will be used for all measurements. Conditions may not allow this scheme to be used each time, and therefore, several other permissible SOPs are described below.

Wading and hand collection

If the stream is safe to wade, the sample collector wades to the center of the stream with a sample bottle. The sample collector faces upstream taking care not to disturb any stream bottom debris or sediment which may contaminate the sample. The sample bottle is inverted and dipped below the surface, then turned upright to collect the sample while holding the bottle about one foot below the water surface. When considering wading, the general rule is that if stream depth (in feet) multiplied by its velocity (feet/second) is greater than the sampler's height (in feet), then the sampler MUST NOT WADE. Shoreline immersion of a sterile *Whirlpak* plastic container will be used for *E. coli* sampling.

Reach pole collection

When wading conditions are not safe in smaller streams, a grab sample may be collected using a reach pole. With the reach pole the sample bottle is fitted into a wire cage attached to the end of a long, telescoping reach pole. The sample bottle is inverted and dipped below the surface, then turned upright to collect the sample while holding the bottle about one foot below the water surface.

An alternative method is to use a 1-L polyethylene bottle affixed to the end of the reach pole to collect sample water which is then transferred to the sample bottles on shore. With this method the sampler bottle is triple rinsed with site water before taking samples for laboratory analysis.

Bridge and rope collection

For larger rivers where the sampling station is adjacent to a bridge, a grab sample may be collected using a Labline Polypro (or equivalent) sampler, or even a clean plastic bucket, lowered from the bridge deck near the river thalweg. The Labline sampler is lowered to the river surface and plunged into the water to an approximate depth of one meter below the water surface. The sampler is then raised to the bridge deck, and the grab sample is poured into the sample container. In this variation, both the Labline sampler and the sample bottle are triple rinsed with site water before collection of the final sample, as described above.

2. Invertebrate and habitat surveys

Survey methods are described in detail in:

- Breneman, D., V. Brady, and L. Johnson. 2007. *Cook County Soil & Water Conservation District Biological Sampling for the Poplar River TMDL Quality Assurance Project Plan. NRRI/TR-2007/16, Natural Resources Research Institute, U. of Minnesota Duluth, 5013 Miller Trunk Highway, Duluth, MN 55811-1442*
- *Natural Resources Research Institute (NRRI). 1999. Standard Operating Procedures (SOP): Benthic Sample Collection and Processing, University of Minnesota Duluth, Natural Resources Research Institute, Technical Report, NRRI/TR-1999/37, 17 p.*

Benthic macroinvertebrate and habitat sampling evaluations will be conducted at locations chosen to represent the most common in-stream and riparian conditions. A best effort is made to minimize bias from either direct or indirect landscape alterations when selecting sampling locations. Sampling sites are selected based on several parameters (e.g., biological, geomorphological, etc.), but logistical considerations including best available access also influences site selection. Sampling protocols will follow standard operating procedures outlined by the NRRI-UMD Microscopy Laboratory standard operating procedures for field collection, laboratory sample processing, and data analysis (NRRI 1999; NRRI/TR-1999/37). All procedures outlined in the NRRI document are subject to change to respond to MPCA guidance and field conditions.

Sampling protocols will provide an overview of the available habitat conditions and community composition associated each designated site and whenever possible will overlap the intensive water quality sampling site location. Sample collection equipment is selected to ensure both sample precision and accuracy to meet project objectives. Sample quantity will be sufficient to capture the inherent variability of the stream community.

B3. SAMPLE HANDLING AND CUSTODY

Jerry Henneck and Elaine Ruzycki jointly are responsible for tracking field samples and storing records of all samples taken by field personnel. Sample bottles are labeled with bottle number, site identification, and date. They are sealed tightly and packed in a cooler on ice at the sampling location. The field record includes project name, sampler's signature, unique station identification number, sample number, parameters for laboratory analysis, matrix, number and size of containers, and date and time. All laboratory samples are brought back to the NRRI Lab within 24 hours of collection. Coolers containing samples that require ice preservation are checked periodically to ensure samples remained adequately iced so sample temperatures do not exceed 4°C. In most cases turbidity measurements and TSS filtrations are performed immediately upon arrival at the Lab. If not, they are stored overnight in a 4°C walk-in refrigerated room. Raw and 0.45 micron Millipore HAWP membrane filtered water is subsampled into 125 mL polyethylene bottles and frozen for nutrients; fresh filtrate is refrigerated at 4°C for the remaining water quality analyses.

Information on field conditions, such as the weather, deviations from written procedures, operating condition of the equipment, and other unusual occurrences are also recorded for each sampling event on the field data sheets.

Laboratory Sample Handling

Sample containers are provided by the laboratory. Container cleanliness is verified by QA/QC procedures as specified in the laboratory's QAM and SOPs. The laboratory verifies sample bottle cleanliness by running a specified number of bottle blanks on each shipment received and on each batch of sample bottles following laboratory cleaning and sterilization, if reused. A preservative is added to specific bottles, as required, or accompanies the bottles in a separate container. Preservatives used and their volumes and concentrations are specified in the laboratory QAM. For this project no preservatives are used other than chilling and then freezing nutrient samples at -20°C until analysis. Details may be found in Ameel et al. (1998, rev. 2007):

- *Ameel, J. Ruzycki, E., Owen, C.J. and R. Axler, 1998 (revised 2007). Analytical chemistry and quality assurance procedures for natural water, wastewater, and sediment samples. Natural Resources Research Institute, Technical Report, NRRI/TR-98/28.*

Temperature blanks are included in the coolers provided by the laboratory to verify whether the appropriate sample temperature of $\leq 4^{\circ}\text{C}$ has been maintained.

Upon arrival at the laboratory, the condition of the samples is determined. The samples are checked for leaks and appropriate preservation and checked to ensure that temperature has remained $<4^{\circ}\text{C}$ (typically just noting the presence of ice). The information is recorded on the sample identification sheet. The sample identification sheet information is then compared to the information on the sample bottles and any discrepancies are noted. The samples are then logged into the Laboratory Information Management System (LIMS) and stored as noted above. The laboratory sample storage areas are monitored daily.

Certain water samples are analyzed by at the WLSSD Laboratory for TP, chloride and TSS as per collaborative work with the *LakeSuperiorStreams.org* project. These samples are delivered in separate polyethylene containers and treated identically to NRRI analyzed samples for the same parameters (see Tables 6 and 7).

E. coli bacteria samples are delivered directly to Northshore Analytical, Inc. On rare occasions due to problems in the field that delay return to Duluth from North Shore surveys, the (sterile) samples will be refrigerated at 4°C overnight but will still be analyzed within the holding time of 24 hrs.

Field Information Sheets

Field data sheets are the primary method for documenting most stream monitoring field activities. These sheets serve as an initial record of any field measurements and weather conditions at the time of sampling.

Field Notes

Field notes are used to document important information during sampling events. They are entered into a bound notebook with waterproof pages. Entries are made using pens with indelible ink. The field notebook becomes part of the project data and is retained with the analytical data hard copies and other project documents.

Sample Labeling

Each sample container has a label attached which is filled out in its entirety. The sample label includes the water body code or name, the site number, the date, and time of sample collection.

Sample Shipping

All samples are packed in an ice-filled cooler for transport to the laboratory. Samples are immediately brought to the Lab after sampling or in unusual circumstances stored in the NRRI walk-in cold room (4°C) overnight.

B4. ANALYTICAL METHODS

Analytical protocols are found in the NRRI Lab Manual (Ameel et al. 1998 [rev. 2007]), in WLSSDs 2007 Lab Manual, and North Shore Analytical, Inc.'s Lab manual. Analytical accuracy is routinely checked by Laboratory analysis of standard certified reference analytes as described previously and required for State Certification by MDH.

All raw data generated in the laboratory are recorded in bound notebooks, on project specific raw data sheets, or as an instrument printout. This data includes sample numbers, calibration data, calculations, results, analyst notes and observations, quality control data, date of analysis, and initials of the analyst. Completed notebooks are archived. Chromatograms, graphs, and strip

charts, if part of the data package, are kept with the laboratory raw data. All items are labeled, dated and signed by the analyst. When completed, the data are integrated into a final report.

For out-of-control situations, a corrective action plan is in place. The initial action is to repeat the analyses of the samples bracketed by the unacceptable quality control sample. Replication of unacceptable results is investigated as a matrix effect by reviewing blank spikes or laboratory knowns. If the quality control samples are still unacceptable, the entire process is repeated. This includes sample preparation or extraction. If re-analysis is not possible due to the sample being past holding times or sample quantity is insufficient, documentation of the situation will be added to the raw data. In these cases, the report is flagged.

B5. QUALITY CONTROL

Where applicable, internal reference standards are analyzed and recorded with each sample run. External reference standards and standard reference material obtained from APG are analyzed annually. All stock standard solutions are properly labeled, stored, and expiration dates visibly recorded on the label. The measured data for the certified standards must fall within the specified range as given by the provider or corrective action will be taken.

The Minnesota Department of Health (MDH) certifies NRRI's Central Analytical Laboratory, the WLSSD Laboratory and North Shore Analytical, Inc. As such the laboratories are all subject to audit by MDH and MPCA. NRRI's Lab has also been certified by EPA Region V in the past for sediment assessment studies in the St. Louis River Estuary.

One field QC grab sample duplicate for laboratory analysis is collected at the sampling site for every ten samples taken. The field duplicate for laboratory analysis is collected to determine sampling and laboratory analytical precision. If QC samples revealed a sampling or analytical problem, field and laboratory personnel attempt to identify the cause. Upon working out a plausible solution, personnel take necessary steps to ensure that similar problems do not arise during future sampling events. If possible the sampling event is repeated as soon as feasible. As per laboratory protocol, suspect data are flagged or qualified depending upon the nature and extent of the problem.

All three Laboratories have specified, and approved (by MDH) QA/QC methods and procedures for dealing with out-of-control situations. These are documented in their Lab and/or QA Manuals and SOPs, copies of which are maintained on file at MPCA and available for consultation and review upon request.

B6. INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

All hand-held instruments, when used, are inspected and tested each sampling day prior to their use in the field. Steps are taken to fix any instrument problems noted during testing. If any problems cannot be resolved a substitute instrument is used. pH buffer solutions are replaced with fresh solutions before the buffer solution expiration date. Spare batteries for all instruments are taken on all sampling trips. All maintenance procedures are documented in the meter maintenance logs or the field notebook.

B7. INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY

Temperature at volunteer monitoring stations will be determined using the thermistors reading on the specific conductivity (EC25) hand held meter. Each unit is numbered and checked for accuracy with an NIST-certified thermometer and an ice water slurry to ensure a reported accuracy of $\pm 0.1^{\circ}\text{C}$ at the beginning of each sampling season. Other field instruments are calibrated each sampling day before being taken into the field. Instrument calibration is checked periodically throughout the sampling day and recalibrated if necessary. All instrument calibration checks and procedures are documented on the instrument maintenance log or in the field notebook.

B8. INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

Supplies and consumables included buffer solutions, paper products, gloves, deionized water, and batteries. Supplies and consumables are purchased only from reputable and reliable suppliers and inspected for usability upon receipt.

B9. DATA ACQUISITION REQUIREMENTS (NON-DIRECT MEASUREMENTS)

Project staff review historical water quality data collected by previous assessment projects and from prior dates for that site, and use these data for comparative purposes when such need arises.

B10. DATA MANAGEMENT

The field samplers work as a team to complete the field data sheets. This information is entered into a spreadsheet or database and archived. Laboratory results are entered into a computer database and/or spreadsheet which is maintained by the Project Quality Assurance Coordinators who also assist with data maintenance, reduction, and transmittal. The Project Manager and co-Managers and the MPCA Project Manager will also review all data prior to its submission and approved entry into STORET.

Quality assurance data sheet checks include scanning for apparent entry errors, measurement errors, omissions, and anomalies. Suspect data are flagged and/or excluded from use. Data may be presented in table, graph, and chart format. Unusual data are rechecked to verify their accuracy. The data are then entered into STORET by MPCA data entry personnel.

GROUP C: ASSESSMENT AND OVERSIGHT

C1. ASSESSMENT AND RESPONSE ACTIONS

Richard Axler, as Project Manager is responsible for all reporting, tracking, and overall SWANS Project management including field activities, reviewing the data, reporting to the group on findings, and forwarding all data to the appropriate state regulatory agency for inspection and input into STORET. He specifically oversees all aspects of the water quality assessment component of the project, including the volunteer monitoring, while Valerie Brady and Dan Breneman are responsible for the macroinvertebrate and habitat component. The MPCA Project

Manager and QA staff are also authorized to oversee field activities during this project. The MPCA Project Manager and WQ QA/QC Coordinator are also authorized to follow up on sampling activities during the project.

C2. REPORTS TO MANAGEMENT

A draft report of the SWA-NS Project findings will be prepared for the MPCA and shared with all involved watershed districts, local resource managers, and other involved parties.

The Project Manager will submit an annual report by December 1st of each project year to the MPCA Project Manager. Problems that arise during the project are corrected and reported to all parties involved in the project via this report.

All data are recorded and tracked through use of NRRI-UMD internal Microsoft Access and Excel databases. The data compiled during this project is incorporated into spreadsheets and sent to the MPCA for perpetual storage in STORET, the EPA environmental database.

GROUP D: DATA VALIDATION AND USABILITY

D1. DATA REVIEW, VERIFICATION, AND VALIDATION

All raw data are transcribed to the data transmittal form and stored in a binder-type notebook. Where applicable, the data is organized electronically and filed in the MPCA STORET database. Statistical analyses on replicate samples are recorded so that the degree of certainty can be estimated.

All laboratory analytical results are cross-checked against the field notebook and sample tags to ensure that the raw, computer-generated summary of the laboratory analyses are assigned to the correct sampling stations. All analytical results are compared to the field sheets to ensure that the data are complete. Copies of the data transmittal form and all pertinent records of calibration, standardization, and maintenance will be archived.

All data are signed/initialed by the analyst/sampler and reviewed by the project Quality Assurance Coordinators (D. Breneman for biological data and E. Ruzycski for water quality data) to determine if the water quality data meets the DQO and QAPP objectives. The QA/QC elements of the invertebrate and habitat data are non-routine for MPCA at this time and NRRI-UMD will assist MPCA on this issue as needed. In addition, Pam Anderson, MPCA Project Manager, assists in the data review. Data is examined and outliers identified through statistical analysis. Decisions to reject or qualify data are made by the NRRI-UMD Project Managers (Axler, Breneman and Brady) in consultation with Pam Anderson from MPCA.

D2. VERIFICATION AND VALIDATION METHODS

Project staff follow the EPA *Guidance on Environmental Verification and Validation* (EPA QA/G-8) whereby the data are reviewed and accepted or qualified by project and/or MPCA staff.

D3. RECONCILIATION WITH USER REQUIREMENTS

Within 48 hours of receipt of results of each sampling event or chemical analysis, calculations and determinations of precision, completeness, and accuracy are made and corrective action implemented, if needed. If data quality does not meet project specifications, the deficient data are flagged or discarded and the cause of failure evaluated. Any limitations on data use are detailed in the project reports and other documentation.

Project data are compared to historical data, when available, and may also be used as complementary data for other monitoring efforts within the watershed.

For the data to be considered valid, data collection procedures, the handling of samples, and data analysis must be monitored for compliance with all the requirements described in this QAPP. Data are flagged and qualified if there is evidence of habitual violations of the procedures described in this QAPP. Any limitations placed on the data are reported to the data end user in narrative form.

Appendix A. Coliform Bacteria Sampling (*E. coli*)

Sample Collection, Preservation, and Storage

Because sterile conditions must be maintained during collection, preservation, storage, and analysis of indicator bacteria samples, specific procedures have been developed that must be strictly followed. These procedures vary with types of sampling equipment and source of sample (surface water, ground water, treated water, or waste water).

Surface-Water Sample Collection

The areal and temporal distribution of indicator bacteria in surface water can be as variable as the distribution of suspended sediment because bacteria commonly are associated with solid particles. To obtain representative data, one would like to use the same methods for collecting surface-water samples for bacteria analysis as for water and suspended sediment or better yet, use a single container of pooled water from which all samples are taken (after vigorous mixing). However, because of the potential risk of sample contamination, we will use sterile Whirl-Pak polyethylene bags that are directly immersed into the stream within a few minutes of the general water quality sampling.

Quality Control.

Depending on the data-quality requirements, quality-control (QC) samples (blanks and replicates) can comprise from 5 to 30 percent or more of the total number of samples collected over a given period of time. We will use the 10% rule-of thumb for SWA-NS.

Field blanks will not be collected for this project because NRRI-UMD has its own bottle preparation procedures that ensure that sampling equipment has not been contaminated.

Hand-Dip Method

If the stream depth and (or) velocity is not sufficient to use a depth-and-width integrating method, collect a sample by a hand-dip method. Although sterile point samplers such as Niskin, ZoBell, and Wheaton samplers hold a sterilizable bottle or bag, we will simply directly submerge sterile Whirl-Pak bags and non-sterile plastic bottles. To collect a hand-dipped sample:

1. Open a sterile, narrow-mouth borosilicate glass or plastic bottle; grasp the bottle near the base, with hand and arm on downstream side of bottle.
2. Without rinsing, plunge the bottle opening downward, below the water surface. Allow the bottle to fill with the opening pointed slightly upward into the current.
3. Remove the bottle with the opening pointed upward from the water and tightly cap it, allowing about 2.5 to 5 cm of headspace. This procedure minimizes collection of surface film and avoids contact with the streambed. Note that dissolved oxygen sampling requires that no headspace be present and glass BOD bottles with special pointed

stoppers and ground glass seals are preferred. This is particularly critical is low DO concentrations are anticipated.

Sample Preservation and Storage

After collection, immediately chill samples in an ice chest or refrigerator at $\leq 4^{\circ}\text{C}$. Samples will be returned to the Lab as soon as possible and processed as per Tables 9 and 10. *E. coli* samples will be delivered to North Shore Analytical, Inc. on the way back from the field so that bacterial cultures can be started as soon as possible. Since the sampling is not for possible enforcement purposes, the maximum holding time is 24-hours and a chain-of-custody form need not be used.

Preserving Sample Cleanliness

If sampling from a bridge take care to keep the rope, used to lower the sampler, coiled inside of a bucket when pulling the sampler up to keep the rope from being contaminated by substances from the bridge deck. When lowering and raising the sampler do not let the rope rub against the side of the bridge. Such rubbing could knock material from the bridge into the sampler and contaminate the sample.

Safety When Sampling From a Bridge

If you are in traffic wear a traffic safety vest. Carry a white bucket to increase your visibility. If visibility is low, set a blinking warning light next to you while you are collecting the sample.

If you are on a *Warner* truss or similar bridge and it is a sunny day, also use a warning light. Place the light in one of the shadows. The shadows of the truss work on the bridge deck will cause optical confusion for approaching drivers and will hide your presence.

Appendix B : MPCA Standard Methods for Hand-Collected (Grab) Sampling

Note – The sampling methods used by NRRI-UMD for the SWA-NS were described previously and are consistent, with minor exceptions, with the methods described here by MPCA for their own stream sampling programs.

Water is collected at the sampling point using one of the following methods depending upon physical accessibility:

- Triple sampler (MPCA design) – not used for SWA-NS
- Remote grab sampler (MPCA design - 2-liter Nalgene™ bottle clamped to a telescoping pole). This will be the primary water sampling method used by NRRI for SWA-NS.
- Sample bottle dip while wading (for SWA-NS *E. coli* sampling using a single sterile container to avoid chance of contamination)
- Sample bottle dip through hole cut in ice – for Winter SWA-NS sampling
- Kemmerer Sampler – not appropriate for SWA-NS

Follow bottle rinse and preservation methods as directed by the analyzing laboratory (NRRI Lab Manual and standard field SOPs). ~~The Minnesota Department of Health recommends that its bottles **not** be rinsed before sample collection. MDH sample bottles are pre-cleaned, disposable. Also, each lot is sampled for cleanliness as part of MDH's QA/QC Program.~~ Repeat-use sampling equipment chambers that contact sample water should be rinsed thoroughly with sample water three times before water is collected to transfer to sample containers.

When grab sampling is suitable, samples should be collected along the sample site cross-section. Sample at a point that best represents the water quality of the total flow at the cross section. Avoid sampling points that are poorly mixed or affected by local temporary conditions such as ponding across part of the stream width, obviously disproportionate sediment load, or backwater conditions. If a site is poorly mixed across the stream, integrated sample across the stream width should be used, or another site should be chosen that is well mixed across the stream width.

Collect the sample at a middle depth in the water column without disturbing stream bed sediments or collecting floating materials from the surface. When grab sampling, the bottle should be lowered mouth down to the middle depth below the water surface then turned upward to collect the sample. Always stand downstream of the sampling point to avoid contaminating the sample. During ice conditions, keep ice and snow out of the sampling hole cut in the ice.

SAFETY FIRST!

If wading, as a general rule, if stream depth (in feet) multiplied by its velocity (feet/second) is greater than your height (in feet), and then **DO NOT WADE!**

(Stream Depth) [ft.] x Stream Velocity [ft./sec.] > your height [ft.] = Do Not Wade!

Appendix C: QA Field Sampling Procedures - Sampler Blanks

Note – The sampling methods used by NRRI-UMD for the SWA-NS were described previously. Sampler blanks are not needed for SWA-NS and are not budgeted. Sampling bottles are thoroughly cleaned with DI water and/or 0.1 N HCl followed by multiple DI water rinses as detailed in the NRRI Central Analytical Laboratory Manual (Ameel et al. 1998). Records of true sample blanks from previous projects can be produced if required (the most recent being the EPA funded Great Lakes Environmental Indicators Project, 2002-2004 field studies in the Great Lakes Coastal Zone using identical methods to SWA-NS).

MPCA Recommendations for field sampling blanks (Not done for SWA-NS):

A sampler blank (also commonly referred to as a rinsate blank or equipment blank) is a sample of deionized water that is rinsed through the sampling device and collected for analysis. The first step in collecting a sampler blank is to decontaminate the sampling device in the same manner that is used to collect your regular samples. For example, if you clean the sampling device with detergent and rinse with DI water, then conduct this same procedure before you collect the blank. **If you normally rinse your sampling device with sample water before collecting your sample, then conduct this rinse with DI water instead of sample water** – this will prevent any residual sample water from being detected in your results. Eliminate as much rinse water from the sampling device as possible before you collect the blank.

To collect the blank, fill the sampling device with deionized water and transfer the water to the appropriate collection bottles. Handle the device as close to your normal sampling procedure as possible: agitate the sampling device in the same manner, try to leave the water in the sampling device for the same amount of time, and collect the same volume of water.

Trip Blanks (Not done for SWA-NS)

Trip Blanks are sample bottles of deionized water that are filled before going out into the field and are carried along the entire sampling trip in the cooler. They are typically obtained ahead of time from the laboratory and are preserved in the same manner as the regular sample. Trip blanks are generally only used when collecting samples for volatile organic compounds.

Field Duplicates (described previously for SWA-NS)

A field duplicate is a second sample taken right after an initial sample in the exact same location. Field duplicates assess the sampler's precision, laboratory precision, and possible temporal variability. The duplicate sample should be collected in the exact same manner as the first sample, including the normal sampling equipment cleaning procedures.

Lab Sheets (MPCA sheets; not relevant for SWA-NS project)

A column labeled "QA Type" has been added to the lab sheets. If you are collecting a QA sample, fill in the type of QA sample in this column. Leave the column blank if it is a normal sample. The abbreviations for the QA samples are as follows:

SB = sampler blank FD = field duplicate TB = trip blank

The sampler blanks and field duplicate samples will have the exact same station, date, time, depth, and substation as the samples with which they are associated. The only thing distinguishing the samples apart will be the specified sample type in the "QA Type" column. So please remember to fill in this column with the QA sample type (SB or FD).

Appendix D. NRRI Stream Field Data Sheets

Stream Field Sheet					
Field Info	A	B	C	D	E
Project ID					
Storet station number					
Stream Name					
Date (ddmmyy)					
Time					
Site ID*					
QA*					
Temp (°C)					
EC25 (µS/cm)					
D.O. (mg/L)					
D.O. (%sat)					
pH					
Turbidity (FNU)					
Tape Down or Staff					
Transparency Tube (cm)					
Appearance*					
Recreational suitability*					
Stream condition*					
Sampling device*					
Sample type*					
* see back for instructions					
Field Observations and notes (weather, ice condition, picture # etc)					
A					
B					
C					
D					
E					

Site ID	NRRI internal site number	
QA	Field Duplicate, sampler blank, trip blank, bottle blank, reagent blank	
Appearance	1	Clear- crystal clear transparent water
	2	Milky-not quite clear, cloudy white or gray
	3	Foamy- mineral or pollution
	4	Tea Colored-clear but tea colored due to wetland or bog influence
	5	Muddy-cloudy brown due to high sediment levels
	6	Green- might indicate excess nutrients released into the stream
	7	Green or muddy & either extensive floating scum or strong foul odor
Recreational suitability	1	Beautiful, could not be better
	2	Very minor aesthetic problem, excellent for body contact recreation
	3	Body-contact recreation and aesthetic enjoyment slightly impaired
	4	Recreation potential and level of enjoyment of the stream substantially reduced (would not swim but boating/canoeing is okay)
	5	Swimming and aesthetic enjoyment of the stream nearly impossible
Stream condition	N=normal, L=low, H=high SW=swift, SL=slow, MO=moderate C=clear, M=muddy, O=other	
Sampling device		
SIM	Simple open plastic bucket	
ROD	Telescoping rod with bottle	
ICE	Ice conditions water sampler (straight rod with bottle attached to lower through the ice)	
DI	Depth integrating (USGS type)	
WB	Weighted bucket with cover	
Other	Another type of sampler (describe in notes)	
None	Sample collected directly into sample bottle	
AS	Automatic sampler	
Sample type		
Grab-G	Sampling vessel or bottle filled at one point in water column and cross section of waterbody	
Composite F-CF	Flow weighted with auto sampler	
Composite M- CM	Samples from multiple locations on a water body, combined with churn splitter (describe)	
Composite O- CO	Composite other (describe in comments)	

APPENDIX E – NRRI-UMD Core Suite field measurements for temperature, EC25, DO, and pH

NRRI SOPs are to check all field instruments in the Lab prior to departure. This means checking to ensure that batteries are OK, calibrating for EC25 in the Lab and also performing an air calibration as per the Manufacturer’s Operating Manual instructions.

A. Measurement stabilization guidelines

Standard procedure: Before recording data values, allow sensor to equilibrate to the temperature of the water being monitored and all parameter values to stabilize. There can be no fail-safe minimum time to ensure stabilization since it is a function of sensor history and ecological conditions. Since dissolved oxygen is the most dynamic of the field meter measurements and typically takes the longest amount of time to stabilize, this is the parameter typically used by NRRI-UMD staff to assess variation across the channel and with depth. DO concentration is monitored for at least 60 seconds or until the stabilization criteria are met. In unusual circumstances where sensor drift is abnormal, best professional judgment may be necessary to obtain a measurement but this will be recorded on the field sheet and in the field notebook and brought to the attention of the appropriate Project Manager as soon as possible. Data values will be flagged as necessary. Table 11 provides stabilization criteria for most of the SWA-NS Core Suite parameters and is very similar to the quality assurance objectives and accuracy ratings for continuous water-quality records used by NRRI-UMD for their *LakeSuperiorStreams.org* project’s *in situ* sensors which are identical to those used for manual measurements (see http://www.duluthstreams.org/streams/QA_QC.html)

Table 11. MPCA Stabilization Criteria for Recording Field Measurements	
Standard Direct Field Measurement	Stabilization Criteria for Measurements
Temperature	± 0.2°C
Specific Conductance ≤ 100 µS/cm [†] → > 100 µS/cm →	± 5 % ± 3 %
pH (meter displays to 0.01)	± 0.1 SU [‡]
Dissolved Oxygen (Amperometric method)	± 0.3 mg/L
Turbidity (Turbidimetric method)	± 10 %
†Microsiemens per centimeter, ‡Standard Unit	

B. Field DO (Dissolved Oxygen) Meter Calibration Guidance (NRRI-UMD)

Instruments : YSI 85, YSI 95, YSI 58, Hydrolab Surveyor, Hydrolab Minisonde (MS5) and Quanta multiprobe DO Meters (also with temperature, EC and EC25; some also with pH):

1. DO meter is re-calibrated at or just before arrival at the first sample site, allowing at least 20 minutes for a polarographic Clarke-type probe to polarize.
2. Sensor is immersed at several points along the cross section of the stream to obtain a representative estimate of DO concentration. This is most critical during low flow conditions and in slow moving pools where water may be undersaturated in DO and/or depth variation may exist. When significant depth variation occurs, then a depth profile will be determined taking care to “jiggle” the sensor continuously during the measurement process when water movement is slow to ensure that oxygen is not depleted by the sensor. This is not necessary with the newer luminescent (LDO) sensors which consume insignificant amounts of oxygen during the measurement.
4. Record both DO concentration (mgO₂/L) and % O₂ saturation

C. Temperature – Record immediately after writing down the DO measurements

D. EC25 - Record immediately after writing down the DO and temperature measurements being extra careful to double check that EC25 (temperature compensated electrical conductivity) is being measured and **not** EC (not temperature compensated).

E. pH – Record after other sonde measurements either with the sonde sensor or a separate portable field pH meter following the meter SOP (listed in NRRI-UMD Lab Manual Appendix A. pH will also be measured using a bench pH meter following the NRRI-UMD Lab manual when a field sensor is unavailable.

Appendix F (MPCA Guidance) - The Field Notebook

We have reviewed the MPCA Field Notebook Guidance and found that our field SOPs are substantively consistent with MPCAs. A portion of the MPCA Guidance is included here while other portions were deleted because they were already included in the main body of this QAPP.

This section summarizes information, guidelines, and minimum requirements that apply generally to field measurements for all studies of water quality and the collection of basic data. Other terms commonly used for field measurements are field parameters and field analyses. Before proceeding with field work, staff must be aware and/or trained regarding the recommended methods and equipment, detailed descriptions of measurement and quality-control procedures, and guidelines for troubleshooting and data reporting.

Records, Field Instruments, and Quality Assurance

- Field-measurement data and other field information must be recorded on paper or electronically, while in the field (for SWA-NS we require a hard copy). *Reported* field measurements are defined as data that are entered into STORET. The conventions used for reporting field measurement data are described at the end of each field measurement section.
- Record field-measurement data, methods and equipment selected, and calibration information on field forms and in instrument log books.
- Field forms include study-customized field forms, analytical services request forms (for external Lab analyses), and chain-of-custody records when appropriate.
- Instrument log books for each field instrument are required to document calibrations and maintenance.
- Electronic records are maintained for each uniquely identified sampling location.

Field personnel must be familiar with the instructions provided by equipment manufacturers. Instrument Operating Manuals in some cases provide only generic guidelines for equipment use and maintenance or focus on a particular instrument or instruments that currently are in common use. NRRI-UMD field personnel routinely meet with Sales/service representatives and contact manufacturer's technical staff when there are questions.

Quality-assurance protocols are mandatory for every data-collection effort and include practicing good field procedures and implementing quality-control checks. Field measurements must minimize artifacts that can bias the result and must be routinely checked for variability (precision) and bias (accuracy plus variability). The Project Managers at NRRI-UMD believe that the most important element of Field QA is to have experienced field staff who not only are well trained and understand how their instruments and field gear function, but are well educated in aquatic ecology and familiar with the overall goals and objectives of the project.

Surface Water sampling sites

Field measurements must accurately represent the body of surface water or that part of the water body being studied. SWA-NS field measurements are made at the same location as where water samples are collected and at multiple points along benthos sampling reaches. Standard procedures for locating points of sample collection for surface-water sampling generally follow those detailed in Chapter A4 of the USGS National Field Manual:

USGS. 2000. National field manual for the collection of water-quality data. U.S. Geological Survey Techniques of Water-Resources Investigations, book 9, chaps. A1-A9, 2 v., variously paged.

If the water system is well-mixed and its chemistry is relatively uniform, a single sample will typically be sufficient to represent the water body. For SWA-NS, multiple points of measurements will be needed to characterize various riffle and pool habitats as part of the habitat sampling component that will be conducted once during early to mid-summer. These can then be used to examine if relationships exist between field water quality data and benthic invertebrate community measures.

***In Situ* and Sub-Sample Measurement Procedures**

In-situ measurements are made by carefully immersing a sensor or sonde directly into the water. Measurements made directly in the surface water body are preferable to avoid changes that result from removing a water sample from its source and are especially critical where low DO occurs. Nevertheless, in some cases where flows are low, it can be necessary to collect a grab sample being careful to avoid disturbing bottom sediments. This should be noted on the field sheet and lab book and flagged if necessary. In this situation it may not be possible to determine DO.

Appendix G. MPCA Volunteer/Citizen Scientist Monitoring Protocols

A. CSMP Transparency Tube (T-tube) protocol for estimating water clarity

1. Collect the water sample using a 1 or 2 Liter plastic attached to a 6 foot length of PVC pipe or a commercial telescoping sampler. At least five (5) individual grab samples, collected from as near to mid-stream and mid-depth as possible, should be combined in a clean bucket. This pooled sample will be used to obtain duplicate transparency tube readings.

- Wading or from Stream Bank.

Always sample safely - don't wade into fast-moving water or areas of unknown depth. If you cannot sample safely, make visual observations only (Appearance). If a sample from mid-stream and depth is not possible, avoid stagnant water and sample as far from the shoreline as is safe. Try not to stir up the bottom. Face upstream as you fill your bucket. Avoid collecting sediment from the stream bottom or materials from the water surface.

- From atop a Bridge or Culvert.

With a rope tied to its handle, lower a bucket down to the stream and collect water. Pull the bucket back up, taking care not to bounce the rope or bucket on the side of the bridge or culvert which could dislodge particulate materials that would contaminate the water sample

2. T-tube readings should be taken outside, avoiding direct sunlight by turning your back to the sun if necessary, and against a white towel background on the ground below the tube. After vigorously agitating and swirling the water to resuspend settled sediment, pour water into the tube until the symbol on the bottom is no longer visible. While looking down into your tube, open the valve at the bottom and slowly release water until you can JUST begin to make out the symbol on the bottom. Note this depth. Release a bit more water until the symbol is visible. Note this depth. Record the average of the two depths noted in steps 3 and 4 on your data sheet to the nearest centimeter. If the symbol is still visible when your tube is full, indicate this on the data sheet, for example, > 100 or 120cm depending on the type of T-tube. It may not be easy to distinguish the two readings and if so, just record your best estimate of when the black and white disc is just visible. Include the time of day that the sample was collected. This is the approximate mid-point in time of the grab sampling.

3. Quality Assurance (QA): After recording the first reading, dump the water in the T-tube, swirl the bucket well and then take a second reading in the same manner. Write down both numbers. If they agree to within 10% of their average, you are finished with this measurement. If not, take a third reading, or more until you are getting consistent readings. If you need additional stream water, note that this second bucket was used for subsequent readings since this sample may be somewhat different from the first bucket.

Example: Reading #1 = 76 cm. Reading #2 = 92 cm.

The average reading is 84 cm ($=\frac{76+92}{2}$)

Therefore, each reading differs from the average by 8 cm which is just a bit under the 10% criterion ($8/84 = 9.5\%$). If you forget to bring a calculator, just estimate as best you can. For your information, this calculated value is called the Relative Percent Difference (RPD).

B. Temperature and specific electrical conductivity (EC25) using the Hanna Scientific Pocket Conductivity/TDS/temperature Tester (Model DiST 5)

Turn the meter ON as per its instructions and simply immerse the sensor tips (1-2") into the stream at arm's length from shore. Switch back and forth between the two readings a few times, waiting at least 1 minute for the sensors to equilibrate with the water. Be sure to record the temperature compensated conductivity value (EC25) and not the un-compensated value because temperature will change the conductivity of the water. Since we are using these data as an estimate of the total dissolved ion content (essentially the same as the salt content), we let the instrument internally correct for temperature differences.

- *Note – if it is not possible to obtain direct readings from the stream, then readings can be taken from the water composite in the bucket that is used for the T-tube measurement. EC25 requires no special precautions but care must be taken to ensure that the temperature of the water in the bucket reflects the temperature of the stream. A bucket warmed or cooled by ambient air temperatures can be prevented by immersing the bucket in the stream several times prior to combining water samples in it, and then quickly taking the temperature and EC25 measurements before it changes.*

Record both values on the data sheet. Temperature is in degrees Celsius (same as centigrade) and EC25 is in microSiemens per cm. A microSiemen is usually shown as "µS" and the entire unit is µS/cm – a measure of how electrically conductive the water is between the two metal electrodes (prongs) that are exactly 1.0 centimeters apart. Ocean water would have a very high reading, ~ 43,000 µS/cm and distilled or deionized water purchased at the supermarket for your clothes iron would have a reading near zero. Lake Superior is ~ 90 µS/cm and most of the time, Duluth area and North Shore streams are anticipated to range from ~100 to 400 µS/cm unless road salt inputs are high or there is an upstream wastewater discharge of some kind.

Stream Stage

Estimate the water level each time you sample. L=low; N=normal; H=high

Appearance

Each day that you sample, record the one number that best describes the appearance of stream water within one meter of your sampling site.

1A = Clear – crystal clear, transparent water

1B = Tea-colored – transparent water which has been discolored by dissolved organic matter (lignin) from up-stream bogs or wetlands

2 = Cloudy – is not quite crystal clear; is cloudy, white, or gray

3 = Muddy – cloudy brown due to high sediment levels

4 = Green – due to algae growth; indicative of excess nutrients released into the stream

5 = Muddy AND Green – a combination of cloudy brown from high sediment levels and green from algae growth

Recreational Suitability

Use the one number each day that you sample that best describes your opinion of how suitable the stream water is for recreation and enjoyment.

1 = Beautiful, could not be better

2 = Very minor aesthetic problems. Excellent for body-contact recreation, e.g., swimming, wading, frog-catching

3 = Body-contact recreation and aesthetic enjoyment slightly impaired

4 = Recreation potential and level of enjoyment of the stream substantially reduced, e.g., you would not swim but would boat or canoe

5 = Swimming and aesthetic enjoyment of the stream is nearly impossible

Appendix H. MPCA Volunteer Monitoring Data Sheets



Minnesota Pollution Control Agency

**Citizen Stream-Monitoring Program
 2007 Site Information Sheet**

Site:

FOR OFFICE USE ONLY

Name: _____

Address: _____

Phone: _____ E-mail: _____

Organization affiliated with: _____
 (e.g. County, watershed project, etc.)

Stream Name: _____

County: _____

Location: _____ miles _____ of _____
(distance) (N, NW, etc) (nearest town)

Do you sample from a bridge? __Yes __No If yes, Rd/Hwy Name _____

Description: (describe location using landmarks that appear on a county map)

PLEASE PROVIDE THE FOLLOWING IF AVAILABLE:

Township: _____ Range: _____ Section: _____

GPS Coordinates: _____ LATITUDE _____ LONGITUDE

(Select one) Decimal Degrees UTM Deg/Min/Sec



2007 Citizen Stream Monitoring Data Sheet 1

Your Name: _____
 Stream Name: _____
 Site: NEW

- Make sure your back is to the sun when taking a measurement.
- Fill your tube until the symbol disappears.
- Release water until you can JUST make out the symbol. Note depth.
- Release a bit more water until the symbol is visible. Note depth.
- Record the average of the two depths to the nearest centimeter.
- If the symbol is visible when the tube is full, record as '>100 cm'
- If the reading is less than 60 cm, take readings with BOTH tubes

#	Date	Time	Appearance	Recr. Suit.	Transl / Trans2 (100 cm tube)	Average Transparency 100 cm tube (nearest cm)	Transl / Trans2 (60 cm tube)	Average Transparency 60 cm tube (nearest cm)	Rainfall event? (Y/N)	Stream Stage Estimate (L,N,H)	Stream Temp. (°F)	Comments/Picture Taken?
ex.	6/1	11:00 am	2	3	56 / 52	54 cm	53 / 50	52 cm	N	N	58	Picture, rained overnight
1		am pm			/	cm	/	cm				
2		am pm			/	cm	/	cm				
3		am pm			/	cm	/	cm				
4		am pm			/	cm	/	cm				
5		am pm			/	cm	/	cm				
6		am pm			/	cm	/	cm				
7		am pm			/	cm	/	cm				
8		am pm			/	cm	/	cm				
9		am pm			/	cm	/	cm				
10		am pm			/	cm	/	cm				
11		am pm			/	cm	/	cm				
12		am pm			/	cm	/	cm				
13		am pm			/	cm	/	cm				

At the end of your monitoring season, please return this form & rain gauge datasheet in the enclosed envelope (keep the sampling protocol) **THANK YOU!**



Minnesota Pollution Control Agency

2007 CSMP Rain Gauge Data sheet

Name _____

Time of daily reading _____

County _____

CSMP Site (CSMP0001) _____

	MAR	APR	MAY	JUN	JUL	AUG	SEP	OCT	NOV	REMARKS-SEVERE WEATHER-STORM DAMAGE
1										
2										
3										
4										
5										
6										
7										
8										
9										
10										
11										
12										
13										
14										
15										
16										
17										
18										
19										
20										
21										
22										
23										
24										
25										
26										
27										
28										
29										
30										
31										
Total										

PLEASE SEE BACK SIDE FOR INSTRUCTIONS ON RECORDING PRECIPITATION

Appendix I.



Citizen Stream Field Sampling Protocol

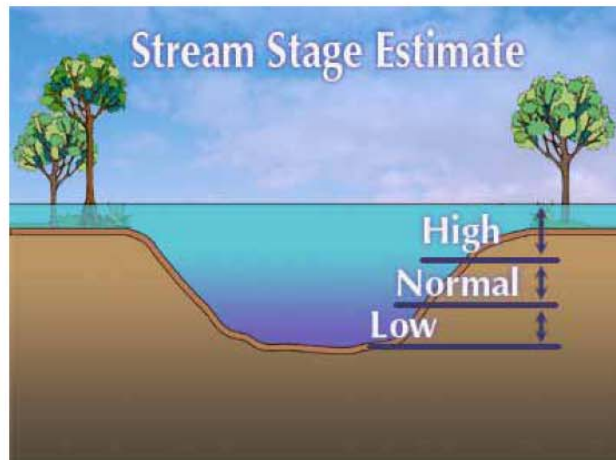
Instructions for Recording Precipitation

1. Try to record precipitation at the SAME TIME each day. **Record daily reading time at top of form**
 2. Record precipitation to the nearest 1/100 of an inch (.01, .31, 1.31), record "0" if no rainfall occurs.
 3. If precipitation is less than .01" record "T" for trace.
 4. If rain accumulates in gauge for more than a 24-hour period (e.g. you were away for a weekend), record the amount in the date on which you empty the gauge, and mark an "R" for 'Range' after the amount (e.g. ".12R").
 5. Use remarks column to note if precipitation is SNOW, or there is other severe weather;
Be sure to record date for which remarks apply.
 6. Sum precipitation for each month and record at the bottom of each column.
-


Explanation of Estimating Stream Stage

STREAM STAGE ESTIMATE (L,N,H): Required - Please estimate the water level each time you sample.
This refers to the relative amount of water flowing in the stream channel as shown by a rough visual estimate of the water level. Normal, Low and High are broad categories so don't agonize too much over which category to choose. The following graphics should help you decide on the correct category:

L=low	Water covers 1/3 or less of the distance from the stream bottom to the top of the bank.
N=normal	Water covers 1/3 to 2/3 of the distance from the stream bottom to the top of the bank.
H=high	Water covers 2/3 or more of the distance from the stream bottom to the top of the bank. Water may be over the stream bank – flooding - at some point.



Example stream monitoring field sheet (p .1)

 MINNESOTA POLLUTION CONTROL AGENCY / STREAM FIELD SHEET					
FIELD INFO:	A	B	C	D	E
PROJECT ID*					
STORET ESTAB. STATION NUMBER					
FIELD CODE OR STREAM NAME*					
DATE (YYMMDD)					
TIME (military)					
SITE ID					
QA*					
TEMP °C					
CONDUCTIVITY @ 25 ° C (umho/cm)					
DO (mg/l)					
PH					
TURBIDITY, NTU					
W.L. GAGE (ft.)*					
W.L. GAGE TYPE*					
TRANSPARENCY* 60 cm tube (to the nearest cm)					
TRANSPARENCY* 100 cm tube (to the nearest cm)					
APPEARANCE*					
RECREAT. SUIT. *					
STREAM CONDITION*					
STREAM FLOW (cfs)*					
SAMPLING DEVICE*					
SAMPLE TYPE*					
* See back of sheet for additional instructions/information. Use codes listed on back to assure STORET entry.					
FIELD OBSERVATIONS (station name/location, weather, ice condition, stream width, picture #, GPS file name, etc.)					
A					
B					
C					
D					
E					

Revised 3/0

Example stream monitoring field sheet (p .2)

ADDITIONAL INSTRUCTIONS/INFORMATION (Stream Field Sheet p. 2)

PROJECT ID

Identify Project ID for sample collection (examples: MILE_UP_MISS, SWANTMDL). If project is not established, please fill out Project Establishment form.

FIELD CODE OR STREAM NAME

If this is an unestablished site and you want the site established and data entered in STORET, please supply us with GPS reading and station description/location. Note these in the field observation section and fill out Station Establishment forms.

QA: FD = Field Dup, SB = Sampler Blank, TB = Trip Blank, BB = Bottle Blank, RB = Reagent Blank

W.L. GAGE (R.):

Water level, also called "stage", determined by reading a staff gage, recording gage, wire weight gage or by subtracting a tape down measurement to water level from a measuring point elevation. A description of the gage should be noted in "field observations", as well as any unusual conditions that affect the measurement (debris around the staff, wind catching the tape, standing waves, etc...)

W.L. GAGE TYPE	ABBREV.	DEFINITION
Tape Down	TD	Tape-down to water level subtracted from established measuring point elevation (describe in comments).
Staff Gage	S	Staff plate mounted vertically in stream.
Wire Weight	W	Weighted wire cable wound on a calibrated reel and attached to a box mounted on bridge.
Automated Stage Recorder	A	Automatic water level readout on an indoors instrument connected to water level sensor in or above stream.
Pool and/or Tailwater elev. (ie L&D)	P/TW	Pool (above dam) and tailwater (below dam) elevations, recorded in L&D station offices. (Record both; also record flow measurement if available).
Other	O	Describe in comments.

TRANSPARENCY READINGS

INSTRUCTIONS:

Make sure your back is to the sun when taking a measurement
 Fill your tube until the symbol disappears
 Release water until you can JUST make out the symbol. Note depth
 Release a bit more water until the symbol is CLEARLY visible
 (can make out screw in middle of symbol). Note depth
 Record the average of the two depths to the nearest centimeter
 If the symbol is visible when the tube is full, record as $e > 60\text{cm}$.

APPEARANCE:

- 1 = Clear - crystal, clear transparent water
- 2 = Milky - not quite clear, cloudy white or gray
- 3 = Foamy - natural or from pollution
- 4 = Tea-colored - clear but tea-colored due to wetland or bog influences
- 5 = Muddy - cloudy brown due to high sediment levels
- 6 = Green - might indicate excess nutrients released into the stream
- 7 = Green or muddy & either extensive floating scum or strong foul odor

RECREATIONAL SUITABILITY:

- 1 = Beautiful, could not be better
- 2 = Very minor aesthetic problems: excellent for body-contact recreation.
- 3 = Body-contact recreation and aesthetic enjoyment slightly impaired
- 4 = Recreation potential and level of enjoyment of the stream substantially reduced (would not swim but boating/canoeing is okay)
- 5 = Swimming and aesthetic enjoyment of the stream nearly impossible

STREAM CONDITION

N=Normal, L=Low, H=High / SW=Swift, SL=Slow, MO=Moderate / C=Clear, M=Muddy, O=Other

STREAM FLOW (cfs)

Note in Field Observations if stream flow was determined by direct measurement, rating curve, L&D gate rating or other.

SAMPLING DEVICE

ABBREVIATION	STORET CONFIG ID	NAME
SIM	SIMPLE	Simple Open Plastic Bucket
ROD	ROD	Telescoping Rod with Bottle
ICEI	ICE I	Ice Conditions Water Sampler (straight rod with bottle attached to lower through ice)
DI		Depth Integrating (USGS type)
WB	WEIGHTED	Weighted Bucket with Cover (aka triple sampler, "labline")
Other		Another type of sampler (describe in notes)
None		Sample collected directly into sample bottle
AS		Automatic Sampler

SAMPLE TYPE	ABBREVIATION	DEFINITION
Grab	G	Sampling vessel or bottle filled at one point in water column and cross section of a waterbody
Composite-F	CF	Flow-weighted with auto-sampler
Composite-M	CM	Samples from multiple locations on a waterbody, combined w/churn splitter (describe in comments)
Composite-O	CO	Composite O Other (describe in comments)

Figure I-1. How to Pack a Cooler. Properly Iced Sample Cooler with a Zip-Loc Bagged Laboratory Sheet and a Shipping Temperature-Check (the blue) Bottle.



Appendix J. *E.coli* Standard Operating Procedure (SOP)

North Shore Analytical, Inc.

4511 W. 1st St., Suite 1

Duluth, MN 55807

Standard Operating Procedure:

Determination of *E. coli*
Bacteria in Surface Waters
By Colilert-18 & Quanti-Tray 2000

Revision Number:

1

Date of Preparation:

3/14/08

Reviewers:

Christopher Gross
Linda Christensen

Last Revision:

3/14/08

Section 1: Procedural

1.1 Scope and Application

- 1.1.1 The reference method is 20th edition, Standard Methods 9223B (Colilert). This method is applicable to the determination of members of the coliform group in clean surface waters. The reporting range is the <1 to 2419.6 Most Probable Number of *E. coli*.

1.2 Summary of Method

- 1.2.1 A 100 mL sample of surface water containing Colilert-18 reagent is incubated in a Quanti-Tray/2000 at 35 ± 0.5 °C. for 18 hours. If the sample has a yellow color equal or greater than the comparator, the presence of total coliform is confirmed. If the yellow sample fluoresces in the presence of UV, greater than or equal to the comparator, *E. coli* is confirmed.

1.3 Definitions

- 1.3.1 Material Safety Data Sheet (MSDS) – Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.
- 1.3.2 Total Coliform Bacteria – Bacteria belonging to the genera *Klebsiella* sp., *Enterobacter* sp., *Citrobacter* sp., or *Escherichia* sp.
- 1.3.3 *E. coli* Positive – The yellow sample fluorescence in the present of UV.
- 1.3.4 *E. coli* Negative – The yellow sample does not fluoresce in the present of UV.

1.4 Safety Concerns

- 1.4.1 Standard laboratory safety precautions regarding the handling of chemicals and samples will be adhered to. Personal protective equipment, including but not limited to: gloves, a lab coat, and protective eyewear, will be worn when handling reagents or samples.
- 1.4.2. Solid and liquid waste materials containing or suspected to contain viable bacteria should be decontaminated using UV sterilization or by using anti bacterial reagent to disinfect before discarding.

1.5 Interferences and Corrections

- 1.5.1 Some water samples containing humic material may have an innate color. If a water sample has some background color, compare inoculated sample to a control blank of the same water sample.

1.6 Apparatus and Equipment

- 1.6.1 Air Incubator – VWR model #1500E. This incubator is capable of operating at $35 \pm 0.5^{\circ}\text{C}$.
- 1.6.2 Quanti-Tray sealer.
- 1.6.3 Quanti-Tray 2000 rubber insert.
- 1.6.4 UV 365nm lamp.
- 1.6.5 Sterile plastic 100 ml sample bottles.

1.7 Media and Reagents

- 1.7.1 IDEXX brand Colilert-18 reagent.
- 1.7.2 IDEXX brand Quanti-Tray/2000.
- 1.7.3 IDEXX Quanti-Tray/2000 color comparator (control sample).

1.8 Sample Collection, Handling, and Preservation

- 1.8.1 Sample collection containers are pre-sterilized plastic 125 ml bottles or vials with a 100 ml volume mark.
- 1.8.2 Samples should arrive at the laboratory on ice. Analyze samples within 6 hours.
- 1.8.3 Samples received beyond the holding time of 6 hours from collection will be rejected. The client is contacted in the event of a rejected sample and a new sampling is requested or the data will be flagged as out of holding time range.

1.9 Sterilization of Equipment and Apparatus

- 1.9.1 Sample bottles are purchased pre-sterilized.

1.10 Sample Analysis

- 1.10.1 Make sure the volume is 100 ml by the gradation mark on the bottle.
- 1.10.2 Aseptically add the Colilert-18 reagent to the sample bottle and recap.
- 1.10.3 Shake until dissolved.
- 1.10.4 Pour the sample directly into the Quanti-Tray/2000. Tap the small wells 2-3 times to release any air bubbles. Allow foam to settle.
- 1.10.5 Place the sample filled Quanti-Tray onto the rubber insert of the Quanti-Tray Sealer with the well side on the Quanti-Tray facing down. Seal Quanti-Tray.
- 1.10.6 Incubate for 18 hours at $35 \pm 0.5^{\circ}\text{C}$

1.11 Interpretation

- 1.11.1 After 18 hours of incubation, compare the color of the sample with the control sample.
- 1.11.2 Yellow wells are checked for *E. coli* by comparing with the control sample under UV light. If the sample fluoresces equal to or greater than the comparator, the sample is *E. coli* Positive. Count the number of wells that fluoresce. Refer to the Quanti-Tray/2000 MPN table to find the Most Probable Number (MPN) of *E. coli*.

1.12 Data Acquisition and Calculations

- 1.12.1 Data to be recorded is:

1. Sample ID #.
2. Colilert-18 Lot #.
3. Incubation Start Date and Time.
4. Initial of Analyst preparing sample.
5. Incubation End Date and Time.
6. Number of small and large wells of *E.coli*.
7. Calculated MPN of *E. coli*.
8. Initials of Analyst.

The *E. coli* Data Form (STF-MICRO-007) will be used in the logbook.

1.12.2 Incubator temperature will be recorded twice daily in the Total Coliform Incubator Temperature Log. The Total Coliform Incubator Temperature Data form (STF-MICRO-003) will be used in the log.

1.13 Data and Record Management

1.13.1 All data and records will be kept for a period of ten years.

1.14 Waste Management

1.14.1 Positive samples will be disinfected with UV prior to disposal in the trash.

Section 2: Quality Assurance/Quality Control (frequency and acceptance criteria)

2.1 Blanks

2.1.1 At least one blank will be analyzed for each lot of bottles and Colilert-18 used.

2.2 Duplicates and Spikes

2.2.1 Duplicates on 10% of sample should be performed.

2.2.2 Spikes are not applicable to this method.

2.3 Split Samples

2.3.1 Split samples are not applicable to this method

2.4 Performance Evaluation (PE) Samples

2.4.1 PE samples are performed on an annual basis.

2.5 Sterility of Colilert-18 Reagent Lot

2.5.1 Each Colilert-18 lot # is checked for sterility by incubating sterile DI with Colilert-18 for 18 hours at $35 \pm 0.5^{\circ}\text{C}$.

2.6 Sterilization Check of Sample Bottle Lots (Bottle Blanks)

2.6.1 For each lot of sample bottles, at least one bottle will be filled with sterile water. After 24 hours the sample will be tested for Total Coliform.

2.7 Positive / Negative Controls for Each Colilert-18 Lot

2.7.1 A positive and negative control will be used to check each lot of Colilert-18.

2.8 Corrective Action

2.8.1 Corrective actions for any QC failures will be documented in the *E. coli* Data Log.

2.9 Audits (external and internal)

2.9.1 Internal audits will be conducted yearly.

2.9.2 External audits will be conducted as required by regulatory agencies.

2.10 Procedure for reporting QC results

2.10.1 Results of Colilert-18 lot #'s sterility, sample bottle blanks, and positive/negative controls for Colilert-18 lot #'s will be recorded with processed samples in the *E. coli* Log Book.

Section 3: References

3.1 Published Literature

3.1.1 Quanti-Tray/2000 from IDEXX, 06-02320-06.

3.2 Methods Manuals

3.2.1 "Standard Methods for the Examination of Water and Wastewater", 20th Edition, 1998, American Public Health Association/American Water Works Association. Method 9223B.

Appendix K

**Analytical Chemistry and Quality Assurance Procedures for
Natural Water, Wastewater, and Sediment Samples**

1998

(Revised: January, 2000, March, 2007)

Natural Resources Research Institute
Center for Water and the Environment
Central Analytical Laboratory
NRRI/TR-98/28

Prepared by:

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Appendix L

COOK COUNTY SOIL AND WATER CONSERVATION DISTRICT
BIOLOGICAL SAMPLING FOR THE POPLAR RIVER
QUALITY ASSURANCE PROJECT PLAN

Prepared by

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for fulfillment of professional service agreement with Cook County dated Feb 2007 – Nov 2008

NRRI/TR-2007/16
PoplarQAPPC_1.doc