QUALITY ASSURANCE PROJECT PLAN (QAPP)

Title Sheet

Draft Version – June 2011

The Upper Yellow River Watershed
Clark, Marathon, and Wood counties, Wisconsin

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APPROVAL SHEET

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Chris Yoder, Research Director, Midwest Biological Institute	Date
Julie Sauer, Quality Assurance Manager – WDNR	Date
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SECTION A - PROJECT INTRODUCTION

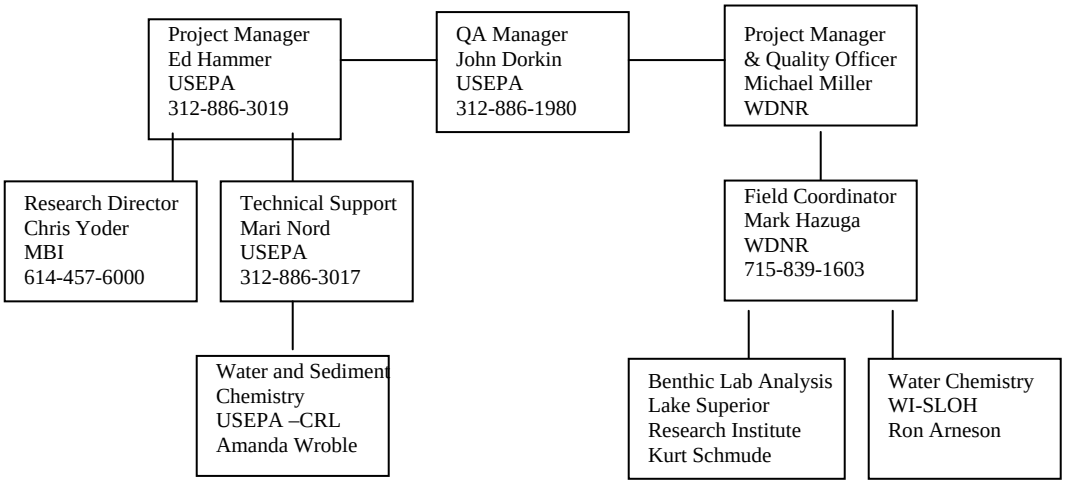
A.1 PROJECT ORGANIZATION

The U.S. Environmental Protection Agency (USEPA), the Wisconsin Department of Natural Resources (WDNR), and the Midwest Biodiversity Institute (MBI) are collaborating on a watershed assessment study in the Upper Yellow River Watershed in central Wisconsin. This study is designed to:

- Demonstrate the effectiveness of using a stratified-random (geometric) sample design used to characterize stream resource conditions,
- Apply advanced statistical methods to group stream sites of similar nature,
- Identify land use practices that are most significant in impacting stream resources,
- Explore relationships between physical and chemical stressors and biotic responses,
- Provide information to land and water resource management agencies to help guide stream and watershed management and restoration efforts.

Figure 1 provides a summary of the project organization for this project and descriptions of the duties of each individual associated with the project.

Figure 1. Organizational Chart



A.1.1 Management Responsibilities

A.1.1.1 EPA Region 5 Water Quality Branch (USEPA):
The EPA Project Manager (Ed Hammer) is responsible for:

- Coordination with the WDNR, with regard to plans for the sampling program including data collection, storage, shipment, establishment of analytical parameters and retrieval of analytical data;
- Communication between the Central Regional Lab (CRL) and WDNR Project Managers on sampling objectives, procedures and protocols;
- Organization and oversight of analytical data.

A.1.1.2 Wisconsin Department of Natural Resources:
The WDNR Project Manager (Michael Miller) is responsible for the coordination, collection, and management of field data and will be a liaison with the Lake Superior Research Institute (LRSI) aquatic entomology and WI State Laboratory of Hygiene (WI-SLOH) laboratories. Additionally, the WDNR Project Manager is responsible for working with the USEPA Project Manager and MBI staff in achieving project objectives, including data analysis and reporting

A.1.2 Quality Assurance Responsibilities
Quality Assurance responsibilities are divided between USEPA and WDNR staff (also see Section D). USEPA's Quality Assurance Manager will review and approve this QAPP.

A.1.3 Research Director Responsibilities
The Midwest Biodiversity Institute Research Director (Chris Yoder) will provide technical guidance on sample design, site evaluation, project implementation, and data analysis.

A.1.4. Technical Support Responsibilities (USEPA)
Technical support responsibilities of USEPA Water Quality Branch, Mari Nord, are to help coordinate water and sediment sample collection with CRL, and chemistry data analyses.

A.1.5 Field Coordinator (WDNR)
The Field Coordinator (Mark Hazuga) will oversee field data and sample collections, ensure crew members are trained and competent, that all stream sites and parameters are sampled, and manage sample shipping and documentation of field sampling efforts. Mark Hazuga will also be responsible for entering and proofing all stream habitat, and fish assemblage data into WDNR databases, and compiling these and water chemistry and macroinvertebrate data into electronic spreadsheets proofing all physical, chemical, and biological data collected for the study, and forwarding these data to the WDNR Project Manager in a timely fashion.

A.1.6 Laboratory Responsibilities
Three different laboratories will perform the chemical and biological analyses for this project. The following is a summary of the analytical work to be performed by each laboratory.

Analyses	**Laboratory**
Water Chemistry (Nutrients, chloride, sediment)	WI – SLOH
Water Chemistry (Pesticides, Atrazine, Metals)	CRL
Sediment Chemistry	CRL
Macroinvertebrate Identification	LSRI
Chlorophyll A and *E. Coli*	WI - SLOH

Written QA/QC reports will be filed by the laboratories each time data is submitted to the USEPA. Corrective actions will be reported to the USEPA project manager along with the QA/QC report (see Section C). Any of the laboratories may be contacted directly by USEPA or WDNR personnel to discuss QA concerns. Responsibilities of each lab and the laboratory coordinator are provided below:

A.2.5.1 Central Regional Laboratory (non-contract laboratory)
USEPA will work with the Region 5 CRL for the analysis of water and sediment chemistry analyses.

A.2.5.2 Wisconsin State Laboratory of Hygiene (non-contract laboratory)
WDNR will be responsible for analysis of water column nutrients, suspended sediment, chloride, B.O.D. Chlorophyll A and Bacteria *E. Coli* samples

A.2.5.3. Lake Superior Research Institute
The University of Wisconsin-Superior's LSRI will be responsible for identification of macroinvertebrate samples and data transfer to WDNR, following WDNR lab protocols as written in Appendix B.

A.2.5 Distribution List

U.S. EPA – Water Division
77 W. Jackson Blvd. (WQ-16J)
Chicago, IL 60604

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A.2 Watershed Background and Problem Identification

A.2.1 Background

Background on Upper Yellow River Watershed

The 213 square mile Upper Yellow River Watershed is located in Clark, Marathon, and Wood counties in central Wisconsin (Fig. 2). Agriculture is the dominant land use in the watershed. There are 171 miles of streams most of which support warm water fish assemblages. The upper watershed terminates a few miles downstream of Dexter Lake, a eutrophic impoundment on the Yellow River. Streams in the watershed lack groundwater inputs, and first and second order streams are often intermittent. Streamflows rise and fall rapidly during runoff events because of poorly drained soils and current land use practices. These highly variable stream-flow conditions degrade habitat due to channel scouring, streambank erosion and sediment deposition. Cropland runoff also delivers sediment and nutrients to surface water during these storm events. Historic stream channelization has also been documented in the watershed.

Total phosphorus concentrations in the Yellow River are some of the highest measured in central Wisconsin. Dexter Lake is listed as an impaired waterbody on the U.S. EPA 303d List because of eutrophic conditions caused by excessive phosphorus loading from the watershed. WDNR is currently monitoring Dexter Lake for Total Maximum Daily Load (TMDL) development. TMDL monitoring includes bi-weekly fixed period water chemistry sampling in the Yellow River upstream and downstream of Dexter Lake and bi-weekly summer in-lake water chemistry sampling. A long term continuous USGS streamflow gauge on the Yellow River at the town of Babcock is being used to calculate loads for the project.

Sources of Watershed Pollution

Nonpoint source pollution (NPS) contributes sediment, organic matter, and nutrients to surface waters in the Upper Yellow Watershed. According to the 2001 State of the Basin Report, excessive animal waste runoff from barnyards and pastures occurs on the main watershed tributaries. Wood County Land Conservation Department staff ranked the Upper Yellow River Watershed as highest priority for NPS erosion control practices. Smaller non-permitted farms dominate the agricultural watershed landuse, however there are at least three large permitted Confined Animal Feeding Operations (CAFOs) in the watershed. These operations are primarily located in the upper watershed in Clark County.

Urban runoff is another potential source of pollution, specifically to the East Branch of the Yellow River, Yellow River and Beaver Creek. The City of Marshfield discharges stormwater to three watersheds. Urban stormwater discharged to the Yellow River Watershed is from the City's west side.

Four Point Sources (PS) discharge to streams in the watershed including two municipal wastewater treatment facilities, a nursing home and a cheese factory. The City of Pittsville discharges directly to the Yellow River above Dexter Lake, and the Village of Chili, Bethel Nursing Home and Nasonville Cheese discharge to small unnamed tributaries to the Yellow River.

In addition to Dexter Lake there are two County Park impoundments on the Yellow River. Lake Manakiki and Kaunewinne are shallow eight and five acre impoundments, respectively. These impoundments prevent upstream movement of fish, downstream impacts are unknown.

A.2.2. Problem Definition

Assessment of stream resources should provide data and information to characterize the physical, chemical, and biological conditions of water resources, to direct management actions and evaluate the effectiveness of current watershed and point source management efforts. Primary goals of the Yellow River Watershed Assessment Project are to:

- Apply a geometric sampling design to assess stream resources, in a cost effective and statistically valid fashion.
- Use fish and macroinvertebrate assemblage data as primary indicators to assess stream conditions.
- Use physical habitat, water chemistry, and land use data to identify stream stressors and streams or reaches most impacted by poor land management or other sources of impairment.
- Apply advanced data analytical techniques to improve the data interpretation and reporting of stream assessment information to improve monitoring program efficiency and effectiveness.

Figure 2. Upper Watershed Description

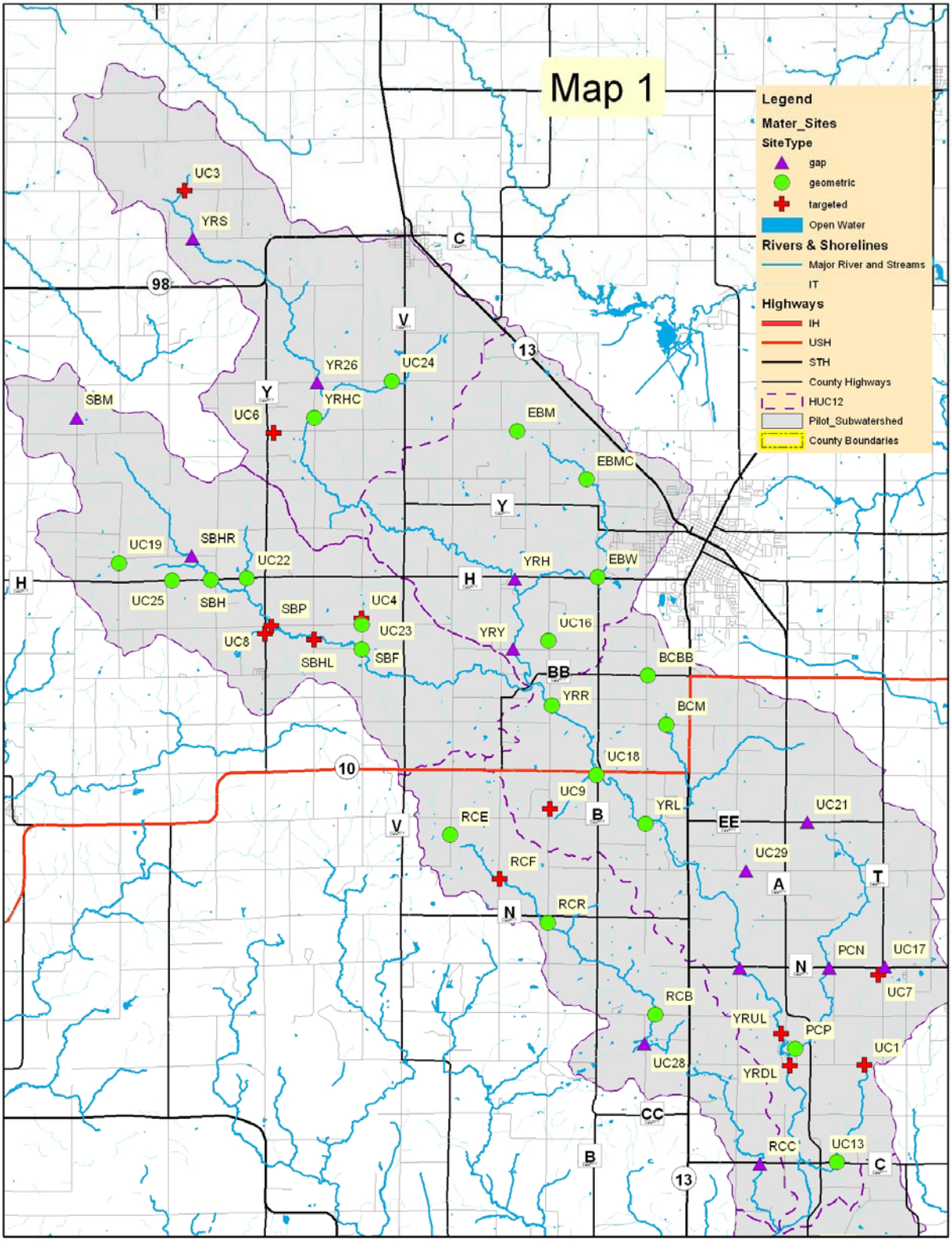
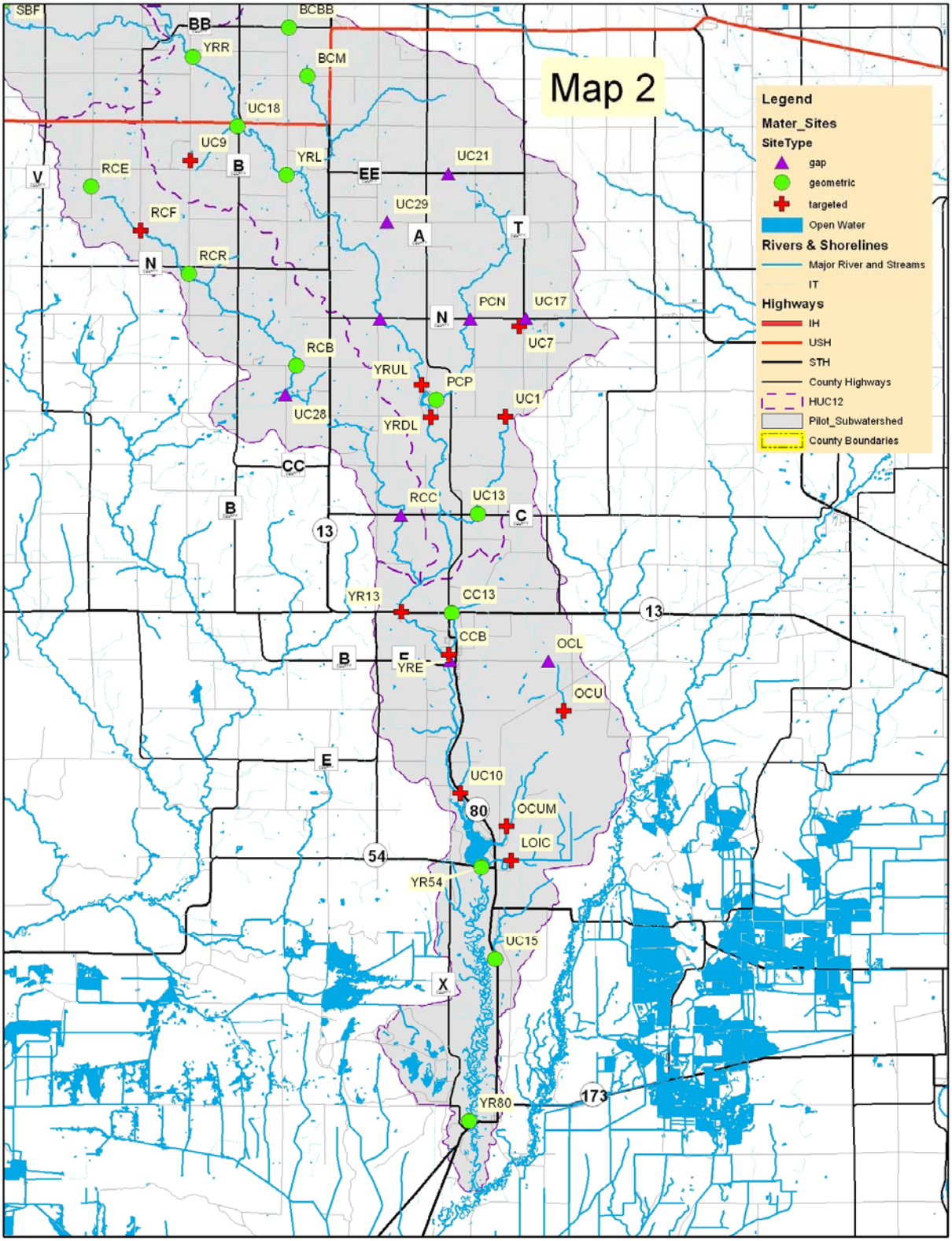


Figure 2.1 Lower Watershed Description



A.3 PROJECT DESCRIPTION

An assessment of stream resources in the Upper Yellow River watershed located in central Wisconsin will be done during the 2011 field season to evaluate the utility of a stratified random (geometric) sampling design for assessing stream resources and to provide data for land and water resources management. Biological assessments using macroinvertebrate and fish assemblage data will be the primary measures of stream quality. Riparian and in-stream habitat features will be evaluated using qualitative assessment methods. Instantaneous measures of field chemical/physical parameters will be collected using electronic meters to measure water temperature, dissolved (D.O.) concentration, percent D.O. saturation, conductivity and pH. Water column transparency will be measured with a transparency tube. Repeated sampling of stream sites for water column nutrients, Biological Oxygen Demand (B.O.D.) and suspended sediment concentrations, and a single round of chlorophyll, E. coli sampling from all sites will be used to identify stressors to stream biota and geographic areas of degradation associated with land use. One round of water column and sediment samples from all sites will be analyzed for metals, polycyclic aromatic hydrocarbon, total organic carbon (TOC) and pesticides. Sediment samples will also be analyzed for dissolved organic carbon and nutrients.

Physical, chemical, and biological field and laboratory methods will follow standard (EPA and WDNR) operating procedures (SOPs). All field and lab data will be captured in WDNR electronic databases.

Data analyses will include the use of physical, chemical and biological measures to characterize the conditions of individual monitoring sites, and aggregated data will be used to assess entire streams, and each geometrically-derived catchment area. Analyses of land cover and land use, and in-stream physical and chemical measures will be done to determine if watershed land use can be correlated with in-stream water quality or biological conditions. Physical habitat features, water chemistry measures, and land use data will be used to evaluate the response of macroinvertebrates and fish to environmental stressors. These data will be used to identify areas within the watershed that appear to be degraded, and to identify land use practices that appear to be detrimental to stream integrity, to help guide improved land management efforts.

Primary study objectives include:

1. Classify streams by Use Potential and determine if these potentials are being met.
2. Assess the physical, chemical, and biological conditions of stream survey sites, individual streams and overall watershed conditions.
3. Evaluate relationships between land use and in-stream physical and chemical stressors
4. Evaluate relationships between biota and physical and chemical stressors.
5. Identify streams and stream segments where changes in land management or Water Pollution Discharge Elimination System (WPDES) Discharge Permits will likely result in improved stream quality.

Specifically we will sample the fish and macroinvertebrate assemblages, physical stream habitat, in-situ/instantaneous water chemistry (pH, conductivity, D.O, Temp, turbidity); water grab

samples will be collected and analyzed for BOD, Chlorides, Sulfates, Total Dissolved Solids, Total Suspended Solids, Chlorophyll a, Nutrients (Total Phosphorus, Total Kjeldahl Nitrogen, Nitrate-Nitrite-N and Ammonia), organochlorine and other pesticides and metals (Cadmium, Calcium, Copper, Iron, Lead, Magnesium and Zinc). Sediment samples will be collected and analyzed for Nutrients (Total Phosphorus, Total Kjeldahl Nitrogen, Ammonia), organochlorine pesticides, polycyclic aromatic hydrocarbons (PAHs) and metals (Arsenic, Cadmium, Copper, Iron, Lead, and Zinc).

A.3.1 Project Tasks

A tentative work schedule is provided in Table 1. All personnel identified in the distribution list should be contacted regarding significant schedule changes. A description of the individual tasks is provided in the text below.

Table 1. Tentative Work Schedule

Task	Description	Date
1	Project Planning QAPP Development	April – May 2011
2	Sampling	May - October 2011
3	Lab Analyses	May 2011 – March 2012
4	Data QA Review	March-April 2012
5	Data Interpretation and Reporting	April-May 2012

Task 1. Project Planning

WDNR will work with MBI to develop the Sampling and Analysis Plan and WDNR and USEPA will write the Quality Assurance Project Plan.

Task 2. Sampling

Table 2. Sampling Schedule for 2011 Field Season

Week of…	Tasks	Samples to SLH	Samples to EPA Lab
16 May	Recon. & Chem. Round 1	Nutrients, BOD/solids (60 sites)	---
23 May	QHEI and other training		---
30 May	Fish (small stream sites)		---
6 June	Fish (small stream sites)		
13 June	Chemistry Round 2	Nutrients, BOD/solids (44 sites)	---
20 June	Fish (small stream sites)		
27 June	Fish (small stream sites)		---
4 July	Fish (small stream sites)		
11 July	Fish (larger stream sites)		---
18 July	Chemistry Round 3	Nutrients, BOD/solids (29 sites)	
25 July	Fish (larger stream sites)		

1 August	Fish (larger stream sites)		
July or -- Aug	Chemistry Round 4 (full suite of parameters)	Nutrients, BOD/solids, Chl-a, *E. coli* (60 sites)	Metals, Pesticides (60 sites total), Sediment (44 sites total)
15 Aug	Chem. Round 4 (full suite)	(continued…)	(continued…)
22 August	Fish		
29 August	Fish		
5 Sept.	Fish \ Contingency time		
12 Sept.	Contingency time		---
19 Sept.	Chemistry Round 5	Nutrients, BOD/solids (44 sites)	
26 Sept.	Macroinvertebrates		---
3 October	Macroinvertebrates		---
10 Oct.	Data entry & management		---
17 Oct.	Chemistry Round 6	Nutrients, BOD/solids (29 sites)	
24 Oct.	Contingency time		---
31 Oct.	Data entry & management		---
7 Nov.	Data entry & management		---
14 Nov.	Data compiling from SWIMS and FH Database		---
21 Nov.	Data compiling & clean up		---
28 Nov.	Data compiling &clean up		---
5 Dec.	Data analysis		---
12 Dec	Data analysis		---
19-Dec	Data analysis		---

If unforeseeable factors (e.g. inclement weather, equipment failures, staffing issues) limit the completion of sampling, certain events may be rescheduled for a later date.

Task 4. Laboratory Analysis of Samples

Samples will be sent to their respective laboratories for analysis. WDNR will coordinate all sample shipping and packaging for each sampling event. The laboratory method and laboratory requirements can be found in Appendix A (CRL), Appendix B (WDNR Lab).

Task 5. QA Review

Written QA/QC reports will be filed by each laboratory. Additionally, the technical contacts will compare the laboratory methods and results to the QA/QC Review checklists contained in Appendix H. There are separate checklists for chemistry data and benthic data.

Task 6. Interpretation

All data including chemical analytical results, habitat data, fish and benthic community analysis, and Geographic Information Systems (GIS)

analysis data will be provided to USEPA and will be sent as hardcopies or electronically transferred. Once data is validated and reviewed, USEPA will work with WDNR in developing a summary report of the watershed study.

A.4 DATA QUALITY OBJECTIVES

This section reports the major objectives of the sampling events, and addresses the most efficient means to attain them.

DQO Step 1: Statement of the Problem

The WDNR will pilot the use of a watershed sampling design that attempts to integrate multiple program data needs into one coordinated survey of the Upper Yellow River Watershed. Data generated will be used to: 1) Determine if watershed streams are properly classified; 2) Determine whether streams are meeting their Use Classification potential; 3) Assess the overall condition of stream resources in the watershed; 4) Evaluate whether wastewater discharges from publically operated treatment plants, private industry (cheese factory), or agricultural activities have a measureable affect on the streams receiving point or nonpoint source pollution; 5) Explore relationships between physical and chemical stressors and biotic responses; 6) Demonstrate the effectiveness of the sampling design used to assess stream sites, individual streams and overall watershed conditions; 7) Demonstrate the value of the sampling design in addressing various programs' (Water Quality Standards (WQS), Non-Point Source control (NPS), Total Maximum Daily Load (TMDL), data needs; 8) Demonstrate whether the design being applied improves integration and efficiencies of stream sampling relative to WDNR's current stream sampling strategy.

DQO Step 2: Decision Statement

Staff from the WDNR, EPA, and MBI will evaluate land cover and land use information to better understand potential threats to water quality from polluted run-off from crop land and urban areas, and also evaluate data from public and private wastewater treatment plants and farm sites to identify site-specific (point) sources of pollution. Data generated from geometric sampling sites will be used to characterize broad-scale water quality conditions, and targeted sampling sites-data be used to assess site-specific risks or problems.

DQO Step 3: Inputs to the Decision

Physical habitat, water chemistry, and biological data will be used to assess stream quality at individual assessment sites. Habitat, water chemistry, and biological index scoring will provide numeric criteria to assess whether or not sites are meeting physical, chemical, or biological expectations. Water chemistry reference condition data and WDNR Water Quality Standards measures will be used to assess site-specific water quality data. Data from multiple sampling sites along a stream will be used to assess the overall condition of individual streams and aggregation of all the sampling data will be used to identify overall watershed and sub-watersheds stream quality conditions and geographic areas of concern.

DQO Step 4: Specification of Project Boundaries

This assessment will focus on the rivers and streams of the Upper Yellow River Watershed in central Wisconsin.

DQO Step 5: Decision Rules - How will data be used to make decisions?

The results from this sampling event will be used to:

1) Fish assemblage data will be used to determine if stream reaches and streams are properly classified.
2) Fish indices data will be used to determine if stream reaches and streams are meeting their Designated Use and ecologic potentials.
3) Physical habitat, water chemistry, macroinvertebrate, and fish assemblage data will collectively be used to evaluate the integrity of streams and stream reaches.
4) Land use, physical habitat, and water chemistry data will be used to identify threats to stream integrity and environmental conditions stressful to stream biota.

DQO Step 6: Specifying Limits on Decision Errors

Errors in this sampling event will be based solely upon:

a) In-field sampling error,
b) Handling error post-field, pre-laboratory,
c) Laboratory handling error,
d) Laboratory analysis error, and
e) Field data transcription error

If it has been determined that:

1) The sampling has followed all necessary protocols and samples were taken successfully, *and*
2) the samples were handled properly prior to and during shipment to the laboratory, and then again after shipment by the laboratory, *and*
3) all laboratory analysis procedures have been followed, and necessary protocol and analysis were successfully completed, and lab results accurately reported,

then the analytical results of that sample will be deemed as successful and "good" data, and can be used for further evaluation.

If, during this sampling event, sample collection from a pre-determined location is unsuccessful, a new location within the vicinity of the original may be chosen at the discretion of the field crew leader. Reasons that may affect sampling from pre-determined locations include unknown site limitations such as access denial by land owner, dry stream channel, unsafe sampling conditions, etc. Documentation of the sample site(s) location change(s) will be made and will include purpose of original location, rationale for changing location and new location coordinates.

A.4.1 Optimizing Design - Selecting a resource efficient sampling design

WDNR will use a geometric sampling design to characterize the quality of all streams and specific stream reaches within the Yellow River Watershed. Targeted sampling will be done to assess potential impacts of point sources of pollution and to fill-in geometric gaps missed by the geometric sampling site design. Discussions with various program data and information users were done to identify core data needs in attempt to address as many resource assessment and management questions as possible.

A.5 SPECIAL PERSONNEL, TRAINING REQUIREMENTS/CERTIFICATIONS, AND/OR EQUIPMENT

Prior to the field-sampling season, members of the field crews will be trained to the extent that they are experts in the portion of the study for which they are responsible. At least two crewmembers will be expert in the collection of each type of data or sample (i.e., fish, macroinvertebrates, physical habitat, water samples). All crewmembers will be expertly trained in water safety and first aid. All training will be documented and records will be kept in the project file.

To minimize any potential health and safety risks related to field sampling conducted as part of this project, members of the field crew need to be physically able to conduct field work under demanding conditions and be well prepared to handle contingencies or emergencies. The following are suggested requirements for all field survey personnel:

a) Recent CPR training,
b) Recent first aid training,
b) Recent electrofishing safety training,
d) Completion of a satisfactory interview about health and safety aspects of the project with the Field Crew Leader, including routine safety precautions and a discussion of actions to be taken in the event of an emergency,
e) Crew will have a first aid kit in the field vehicle and carry a cell phone at all times in the field.

A.6 DOCUMENTATION & RECORDS

A.6.1 Field Documentation

Field logs, sampling ship logs, and chain of custody forms (for EPA-CRL lab only) will be used to record appropriate sample collection information in the field.

A.6.1.1 Field Log Sheets: An example sediment sample collection log is provided in Appendix F. The field crew will fill out a field log sheet for each sample collected. All original field data sheets shall be turned over to the Field Coordinator at the conclusion of the field sampling and shall be kept as part of the permanent project file.

A.6.1.2 Ship Log: A ship log maintaining a summary of sample collection information shall be maintained for each day of field sampling. Information to be included in the ship log shall include: sample location ID, latitude/longitude of each sampling location, and time of sample collection. The ship log shall remain with the ship files for a period of at least 2 years following the conclusion of field sampling.

A.6.1.3 Chain-of-Custody Forms: An example chain of custody form is provided in Appendix G. A chain-of-custody form will be filled out for each set of samples shipped to the laboratory.

A.6.2 Laboratory Reports

All laboratory data and records will be included in the final analytical report submitted to the project manager. A complete copy of the QAPP will be provided to the labs. The WDNR Project Manager will be responsible for maintaining the reports in the permanent project file. The following laboratory-specific records will be compiled by the appropriate laboratory and included in the final analytical report submitted to the USEPA Project Manager.

A.6.2.1 Sample Management Records. Sample management records document sample receipt, handling and storage, and scheduling of analyses. The records verify that sample tracking and proper preservation were maintained, reflect any anomalies in the samples (e.g. receipt of damaged samples), note proper log-in of samples into the laboratory, and address procedures used to ensure that holding time requirements were met.

A.6.2.2 Test Methods. Unless analyses are performed exactly as prescribed by SOPs, this documentation will describe how the analyses were carried out in the laboratory. This includes sample preparation and analysis, instrument standardization, detection and reporting limits, and test-specific QC criteria. Documentation demonstrating laboratory proficiency with each method used should be included (i.e. LCS data).

A.6.2.3 QA/QC Reports. These reports will include the general QC records, such as instrument calibration, routine monitoring of analytical performance, calibration verification, etc. Project-specific information from the QA/QC checks such as blanks (e.g., reagent, method), spikes (e.g., matrix, matrix spike duplicate, surrogate spike), calibration check samples (e.g., zero check, span check, and mid-range check), replicates, and so on should be included in these reports to facilitate data quality analysis.

A.6.2.4 Data Reporting Package Format and Documentation Control Report: The format of all data reporting packages must be consistent with the requirements and procedures used for data validation and data assessment described in Sections B, C, and D of the QAPP. The Field Coordinator will ensure that data are being recorded appropriately on the sample labels, sample tracking forms, and in the field notebook. All entries will be made using permanent ink, signed, and dated, and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark that is signed and dated by the sampler. The contract laboratories will follow a similar data entry process. Only QC/Calibration summary forms will be provided at this time, all analytical raw data shall be kept at laboratories.

A.6.2.5 Government Laboratory Chemistry Data Report:

- Case narrative for each analyzed batch of samples.
- Summary page indicating dates of analyses for samples and laboratory quality control checks.
- Cross referencing of laboratory sample to project sample identification numbers.
- Descriptions of data qualifiers.
- Sample preparation and analyses for samples.
- Sample and laboratory quality control results.
- Results of (dated) initial and continuing calibration checks.
- Matrix spike and matrix spike duplicate recoveries, laboratory control samples, method blank results, calibration check compounds, and system performance check compound results.
- Results of tentatively identified compounds for GC/MS analyses.

Notification of any deviation from these report requirements must be made to the USEPA Project Coordinator or Technical Contact.

*** When possible, an electronic copy of the Analytical Data Report will be submitted in an MS Excel format containing the analytical test results****

SECTION B - DATA ACQUISITION

B.1 EXPERIMENTAL DESIGN

B.1.1 Definition of Sample Types

Three types of sediment and water samples will be collected during this survey; Routine Field Samples (RFS), Field Blanks (FB), and Field Duplicates (FD). Each sample type is described below. FB and FD samples are collected in the field as Quality Control measures to assess laboratory and field precision and laboratory accuracy.

B.1.1.1 Routine Field Samples (RFS): Prepared by collecting a single grab water sample. Routine field samples will be collected at 60 locations. Locations of the RFS are indicated in Table 2.

B.1.1.2 Field Blanks (FB): Prepared by including deionized (DI) or distilled water in sample containers with their own unique sample ID at randomly selected sites at the frequency identified in Table 4.

B.1.1.3. Field Duplicates (FD): Prepared by collecting a second grab sample, homogenizing the material separately from the RFS and filling the required sample jars. FDs will be collected at 6 site locations (approximately 10% of the total sample population). Locations of the FDs are indicated in Table 2.

B.1.1.4. Matrix Spike/Matrix Spike Duplicates (MS/MSD): The MS/MSD samples will be collected out of the same sample as the RFS. MS/MSD samples will be generated for pesticides and atrazine analyses for 5 sites for water and for sediment.

B.1.1.5. *Polyclyclic Aromatic Hydrocarbonss (PAHs)*: Sediment samples taken for PAH samples will be collected in separate 8 oz. glass jars at 7 sites associated with point sources and 5 additional control sites. The 5 control sites will be in the smallest catchment size class, approximately 1.7 square miles.

B.1.2. Sampling Design & Rationale for Design

A “geometric” sampling design” will be used to characterize watershed-wide stream resource conditions. The geometric design divides catchment areas into increasingly smaller (by half) drainage areas (± 10% of the pre-determined geometric catchment area sizes), and the stream flowing out of each of these catchments (pour points) are sampled for physical habitat, water chemistry, and biological data. Sampling pour points of smaller catchment areas are thought to be more representative of the quality of the stream resources within these smaller catchment areas relative the representativeness of pour points of larger catchment areas in characterizing stream resources at these larger spatial scales. Since the larger streams will have multiple sampling sites along their length, this increased weight of evidence should improve the rigor of the assessments of the larger streams. Watersheds commonly have dendritic (branch-like) drainage pattern where number small stream feed into increasingly-larger streams (resembling a

tree trunk with increasingly-smaller branches). The Upper Yellow River Watershed has what is referred to as a trellised drainage pattern where many small streams feed into the main stem river (or trunk in the tree analogy). This requires adding targeted sampling sites in addition of the random-stratified geometric sites, to fill in spatial gaps not assessed by the geometric sites. Lastly, additional targeted sites have been selected in the Upper Yellow River to assess potential site-specific pollution problems emanating from wastewater treatment facilities, large herd-size cattle operations and from urban stormwater runoff. Selecting sites using a census sampling design of all streams within the entire watershed is not feasible, so sub-sampling techniques are necessary. Random or systematic sub-sampling methods are commonly used and each have their own merits and limitations. The proposed systematic sampling supplemented with targeted sites – sampling can improve the accuracy of the assessment of individual streams, but may have limitations in providing more rigorous statistical data used for characterizing populations of streams, by stream strata (e.g. stream order).

B.1.2.1 Sample Location Selection Process

Geographic information system (GIS) tools were used to determine catchment sizes for locating geometric sampling sites (Table 3). Locations of outfalls from public and private wastewater treatment facilities were mapped and site reconnaissance will be done to determine if upstream-downstream sampling of any of these point sources can be done.

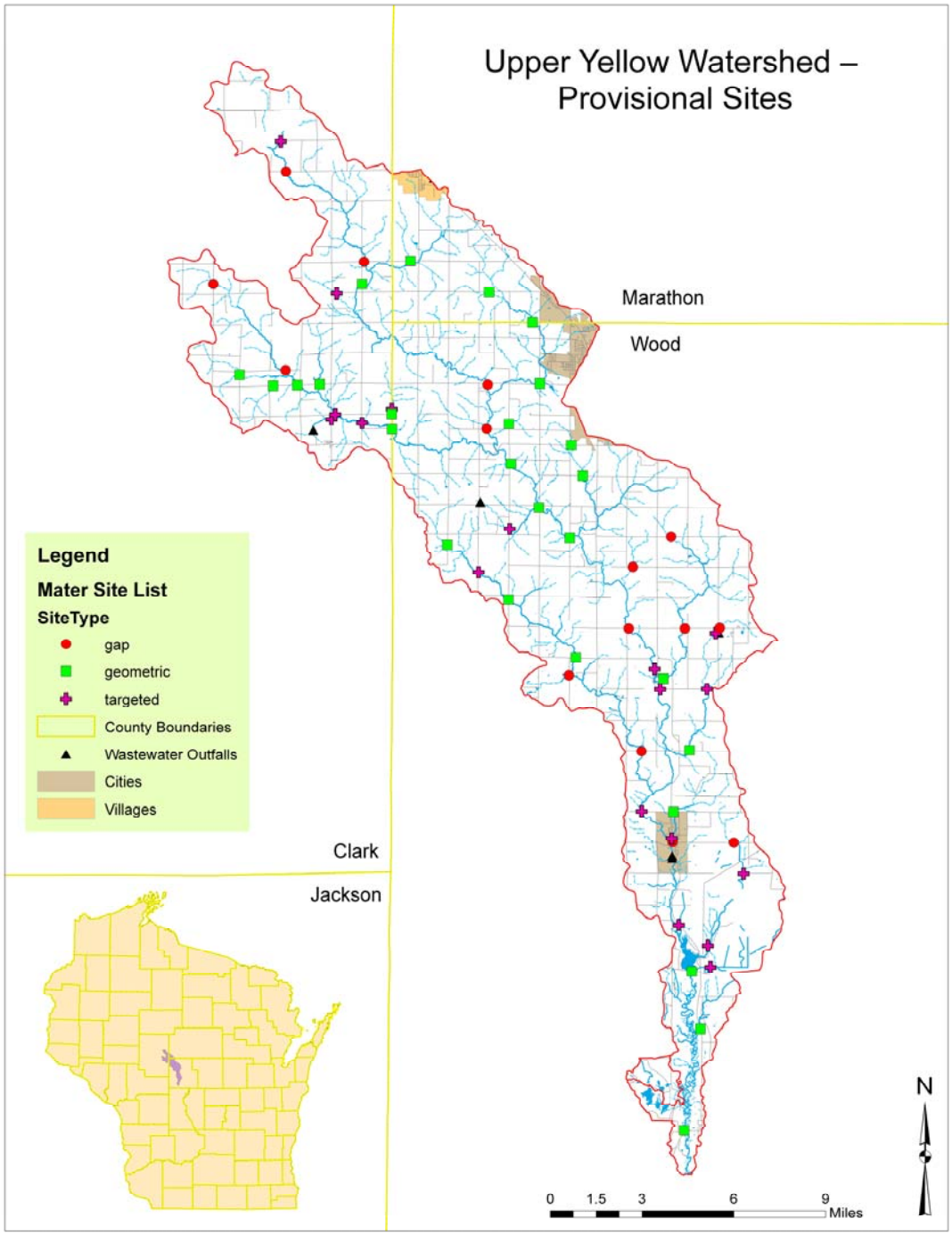
Table 3 lists the sampling panel number which determines the sampling frequency at each site, different catchment-area size classes, site type (geometric or targeted) range of catchments sizes within class, the number of sites that were selected per size class, and number of sites within each size class where duplicate samples will be collected. Panel 1 sites will be sampled 6 times, Panel 2 sites are sampled 4 times and Panel 3 sites are sampled 2 times. Sediment chemistry will be sampled once at every site (with the exception of PAHs) as well as qualitative habitat and benthic macroinvertebrates. Fish assemblage data will be collected once at every site and possibly twice at the larger mainstem sites.

Table 3. Yellow River Watershed Size Class and Panel Information

Panel	Size Class	Site Type	Approximate Area (sq. mi.)	Min area (sq. mi.)	Max area (sq. mi.)	# Sites	# Dup/Blank
1	various	targeted				18	1
1	1	Geometric/gap	213	191.7	234.3	3	0
1	2	Geometric/gap	106	95.4	116.6	3	1
1	3	Geometric/gap	53	47.7	58.3	2	0
1	4	Geometric/gap	27	24.3	29.7	3	0
2	5	Geometric/gap	13.5	12.15	14.85	6	1
2	6	Geometric/gap	7	6.3	7.7	5	1
2	7	Geometric/gap	3.5	3.15	3.85	4	0
3	8	Geometric/gap	1.7	1.53	1.87	16	2
					TOTAL	**60**	**6**

B.1.3 Sampling Locations

Figure 3. Sampling Locations



B.1.3.1 Sampling Location Identification

The latitudes and longitudes of candidate sample sites are listed in Table 4. A Global Positioning System (GPS) unit will be used to navigate to sampling sites and determine the exact location of the downstream end of each sampling reach.

Each site will be given a Field Number for site identification to be used by the field crew to quickly identify the site locations on maps (Table 4). The site ID number is based on catchment size upstream of the sampling site, and for most sites an abbreviation of the name of the roadway that crosses the stream nearest to the sampling site.

In addition to the Site Identification Number, each site will have a SWIMS Site ID. The SWIMS Site ID code is automatically generated by SWIMS (a WDNR database that stores physical, chemical, and biological stream data). The SWIMS Site ID code will be used in sample labeling (described below).

Table 4. Proposed Sampling Sites Location Coordinates (highlighted rows indicate sites where duplicate samples will be collected).

Field Number	SWIMS_id	Panel	SiteType	Dup?	Latitude	Longitude
212.6YR80	723296	1	geometric		44.30224	-90.121895
204.1YR54	723128	1	geometric		44.37783	-90.116898
185YRE	723218	1	gap		44.43909	-90.129877
153YR13	10015314	1	targeted		44.45355	-90.150217
144YRDL	10016089	1	targeted		44.51129	-90.138151
131YRUL	10016191	1	targeted		44.52083	-90.141796
128YRN	723129	1	gap		44.54032	-90.159152
108YRL	10033562	1	geometric		44.58318	-90.198185
100YRR	723130	1	Geometric	DUP	44.61811	-90.237287
59.5YRY	10017316	1	Gap		44.63484	-90.253523
41.6YRH	723145	1	gap		44.65572	-90.252938
27.8YRHC	723264	1	geometric		44.70331	-90.336595
17.6YR26	10033535	2	gap		44.71368	-90.335378
7.8YRS	10031316	2	gap		44.75645	-90.387451
4.3UC1	10033568	1	targeted		44.5115	-90.107198
2.3UC3	10033532	1	targeted		44.77059	-90.39097
2.3UC4	10033541	1	targeted		44.64401	-90.316515
1.8UC6	10033552	1	targeted		44.69878	-90.353331
1.74UC7	10012224	1	targeted		44.53824	-90.101458
1.3UC8	10017422	1	targeted		44.63908	-90.3565
0.9UC9	10033567	1	targeted		44.58746	-90.238022
0.54UC10	10033572	1	targeted		44.3997	-90.125556
6.8UC13	723139	2	geometric		44.4827	-90.118677
1.8UC15	10017309	3	geometric		44.35044	-90.11118
1.6UC16	723203	3	geometric		44.63723	-90.238791

1.5UC17	723136	3	Gap	DUP	44.54065	-90.098838
3.85UC18	10033563	2	geometric		44.59754	-90.218706
3.15UC19	10033549	2	geometric		44.65994	-90.41757
1.9UC21	10033559	3	gap		44.58374	-90.131098
1.87UC22	10033544	3	geometric		44.65559	-90.364354
1.87UC23	10033569	3	Geometric	DUP	44.6418	-90.31655
1.78UC24	10033536	3	geometric		44.71421	-90.304468
1.6UC25	10033547	3	geometric		44.6548	-90.395338
1.53UC27	10033555	3	geometric		44.3173	-90.125802
1.5UC28	10033557	3	gap		44.51782	-90.198531
1.5UC29	10033560	3	gap		44.56935	-90.156401
24.1SBHL	10033542	1	targeted		44.63744	-90.3362
20.5SBP	10033543	1	targeted		44.6414	-90.353926
27.6SBF	10033540	1	geometric		44.63448	-90.316336
14.55SBH	10033545	2	geometric		44.65512	-90.3793
7.7SBHR	10033546	2	gap		44.6622	-90.387174
2SBM	10033550	3	gap		44.70296	-90.435356
5RCF	10020508	1	targeted		44.56666	-90.258613
21.3RCC	723229	1	gap		44.48218	-90.15043
13.5RCB	10017020	2	Geometric	DUP	44.52639	-90.194038
1.85RCE	10017034	3	geometric		44.57962	-90.279369
6.5RCR	10033558	2	Geometric	DUP	44.55381	-90.238762
12.9PCP	723228	2	geometric		44.51635	-90.136024
11.1PCN	10033566	2	gap		44.54038	-90.121889
7.9OCUM	10033575	1	targeted		44.38999	-90.106308
2.3OCU	10033576	1	targeted	DUP	44.42411	-90.082735
1.5OCL	10033556	3	gap		44.43885	-90.089273
3.6LOIC	10033574	1	targeted		44.37988	-90.104524
12.5EBW	10033538	2	geometric		44.65634	-90.218412
7.7EBMC	10033539	2	geometric		44.68531	-90.223297
1.6EBM	10033564	3	geometric		44.69959	-90.252183
4.4CCB	10033571	1	targeted		44.44079	-90.130355
3.2CC13	723158	2	geometric		44.45333	-90.129216
3.3BCM	10033561	2	geometric		44.61256	-90.18973
1.5BCBB	10033565	3	geometric		44.62697	-90.197406

B.1.4 Sample Matrix, Number of Samples and Target Analytes

Table 5 summarizes the type and number of samples to be collected during this sampling event.

Table 5. Summary of Type and Number of Samples to be Collected

Sample Type	Estimated Number of Samples	Estimated Total Number of Samples	Sample Matrix	Analysis Required
Sediment Chemistry	44 + 4 dups	48	Sediment	Metals (As, Ba, Cd, Cr, Cu, Pb, Ni, Se, Ag, and Zn), Pesticides, Nutrients, TOC
Sediment Chemistry - Select Sites	12	12	Sediment	PAHs
Water Chemistry - All Sites	266 + 24 dups + 24 blanks	314	Water	Demand (BOD, TSS, TDS, Chloride, Sulfate) and Nutrients (TP, TKN, Ammonia, Nitrate-Nitrite), SSC
Water Chemistry - Round 4	60 + 6 dups + 6 blanks	72	Water	Chlorophyll-a, *E. Coli*; Metals, Pesticides, Atrazine
Benthic Community	60 + 6 Dups	66	Invertebrates	Identified to lowest practical level typically genus or species
Fish Community Sites	60 + 6 revisits	66	Fish	All fish greater than 25mm identified to species
Habitat Sites	60 + 6 revisits	66	Habitat	WI Qualitative and Ohio EPA QHEI methods used at all sites.

All of the data listed in Table 4 is considered critical to the success of this assessment project.

B.1.5 Criticality of Measurements

There are three types of information recorded for each sample collected:

1. *Laboratory tests*: for chemical analysis, and benthic community analysis.
2. *Field observations*: of fish community, habitat and in-situ water chemistry.
3. *Latitude/Longitude Location*: This data is critical for determining where samples were collected. A recreational-grade (Garmin Model 76s) GPS unit will be used to geo-locate all sites. If problems are noted, the field team should provide a qualitative description of the sampling location using permanent landmarks such as roadways.

B.2 FIELD SAMPLING METHODS

This section describes the field procedures for collecting water chemistry samples, instantaneous water quality measures using a transparency tube and electronic meters, sediment, fish assemblage, macroinvertebrates and physical habitat.

B.2.1 Sampling Procedures

B.2.1.1 Water Chemistry Sampling:

Water chemistry data will be collected from all sites at a set frequency (Table 2). The parameters to be collected for are listed in Table 5. The Wisconsin State Laboratory of Hygiene (SLOH) will provide all water and sediment chemistry sampling bottles for all samples processed at the SLOH. The EPA lab will provide sample bottles for all water column and sediment samples processed at the EPA lab.

For every sampling event, a water sample will be collected in a 1L container and preserved on ice and analyzed in the laboratory for 5-day biological oxygen demand, total suspended solids, total dissolved solids, sulfate and chloride. One water sample will also be collected in a 500 ml polyethylene bottle and preserved with H_2SO_4 then on ice and will be analyzed in the laboratory for total Phosphorus, total Kjeldahl-Nitrogen, Ammonia-Nitrogen and Nitrate-Nitrite Nitrogen.

For one sampling event at each site, a water sample will be collected in a 500 ml polyethylene container and preserved with HNO_3 and analyzed in the laboratory for unfiltered metals. Additionally, a water sample will be collected in a 1 L amber glass bottle and analyzed for Atrazine and pesticides; a sample will be collected in a 1 L plastic bottle and analyzed for Chlorophyll-a (lab filtered); and a sample will be collected in a 250 ml polyethylene bottle and analyzed for *E. Coli* (WDNR, 1998; WDNR, 2005).

The water column nutrient, B.O.D., chlorophyll and *E. coli* samples will be shipped to the WI – SLOH for analysis; all water column pesticides, metals; and all sediment samples analyzed for total organic carbon, total phosphorus, total Kjeldahl nitrogen, ammonia, pesticides, atrazine, metals and PAHs will be shipped to the EPA CRL for analysis.

In situ measurements of temperature, dissolved oxygen, conductivity, and pH will be taken using a water quality meter, and turbidity will be measured in the field with a 120 cm transparency tube, following the equipment manufacturers' operating procedure guidelines. Electronic water quality meters will be calibrated and calibration documented everyday before sampling.

Table 6. Water chemistry parameters

Water Chemistry Parameter	Type of Measurement
Conductivity	Field meter
D.O.	Field meter
Ph	Field meter
Temperature	Field meter
Turbidity	Field (turbidity tube)
Total Suspended Solids	Laboratory - Water
Suspended Sediment Conc.	Laboratory - Water
Total Dissolved Solids	Laboratory – Water
Total Phosphorus	Laboratory - Water
Ammonia-Nitrogen	Laboratory - Water
NO_3/NO_2-Nitrogen	Laboratory - Water
Total Kjeldahl Nitrogen	Laboratory - Water
Pesticides*	Laboratory - Water
Metals**	Laboratory - Water
Hardness	Laboratory - Water
BOD	Laboratory – Water
Chlorides	Laboratory - Water
Sulfate	Laboratory - Water
E. Coli Bacteria	Laboratory - Water
Chlorophyll a	Laboratory - Water

* CRL Pesticide Scans include: total DDT and metabolites, Aldrin and metabolites, Dieldrin, and metabolites, Lindane, Chlordane and metabolites, Atrazine and metabolites, Simazine, other organochlorine pesticides.

** CRL metals include:Ca, Mg, Cu, Cd, Fe, Zn, Pb, Al.

B.2.1.2 Surface Sediment Sampling:

Fine sediment samples will be collected once in the fall at each site, except at geometric sites from the smallest catchment panel (1.7 mi^2). Sediments will be collected in three separate glass containers and analyzed in the laboratory for metals and total organic carbon (TOC) (in a 16 oz. glass jar), pesticides and atrazine (in an 8 oz. glass jar), and polynuclear aromatic hydrocarbons (PAHs) (in an 8 oz. glass jar).

The fine grain sediments (silty sand, silt, clay, muck) will be collected at four or more stations along a 50 m reach of the stream, with two stations on each bank. From the wetted edge to 0.3 m deep at each station, approximately 500 ml of the top 2-3 cm of substrate will be collected with a stainless steel spoon and placed inside a stainless steel bucket. Excess water should be carefully

poured off the spoon without losing any sediment volume before being placed in the bucket. After all sediment is collected, a composite sample from the four stations will be mixed for 2-3 minutes until it is a consistent mixture, then carefully placed into each glass jar and stored on ice. To avoid contamination, the spoon and bucket should be rinsed in the river, and cleaned with Alconox and river water at the downstream end of the next location before sampling again.

B.2.1.3 Fish Community Sampling:
The procedures for collecting fish data are outlined in Table 7 and further described in WDNR's Fish Community Assessment Guidelines (WDNR 2001b, WDNR 2001c, Appendix D). For wadable streams, a single electrofishing run is made starting from the downstream to upstream end of the station. No blocking nets are used. This constitutes the one and only sampling pass. The field crew will try to capture all fish greater than 25 mm total length. Fish are identified to species and counted. Game fish total lengths are measured. After processing, the fish are returned to the stream. A small number of each species may be preserved to verify identifications. For non-wadable assessment sites a "mini-boom electrofishing boat will be used. Sampling protocols (e.g. assessment reach length, fish processing) are the same as those for wadable stream sampling, with the exception that only one fish netter is used for boatable sites.

Different data sheets are used to collect information:
- Station Summary data sheet – summarizes location, sampling characteristics, and gear used for the entire station.
- Catch Summary data sheet – used for recording the numbers, by species of fish captured.
- Individual Fish and Game Fish data sheets – used for recording total lengths, weights, and other information or observations for individual game fish captured.

Table 7. Fish sampling methods.

Sampling Aspect	WDNR Protocol (2001b)
Survey Type	a) Single-Pass Catch-Per-Effort b) No block nets
Electrofishing Gear	a) Backpack Shocker b) Stream Shocker c) Mini-boom Shocker
Fish Data Collected	a) Identify to species and count all fish greater than 26mm total length. b) Weigh game fish (excl, panfish) individually. c) Measure lengths of all gamfish listed in "b".

B.2.1.4 Macroinvertebrate Sampling:

The procedures for collecting macroinvertebrate samples are described in WDNR's Macroinvertebrate Collection Guidelines (WDNR 2000, Appendix C). These procedures will be modified so that one sample is collected at each site. Each sample is a composite of kick samples within one riffle area—ideally, a contiguous area will be sampled diagonally moving upstream. In the absence of riffle habitat overhanging vegetation and/or leafpacks will be sampled. One field data sheet is used to record sampling location identifiers, specific sample and site descriptions, and stream and watershed descriptors. This data sheet is also used for recording any special instructions to the laboratory analyzing the sample.

Table 8. Macroinvertebrate sampling methods

Sampling Aspect	WDNR Protocol (2000)
Sampling Season	Post September 15
Number of samples per site	One composite kick-sample per site
Net frame type and mesh size	D-frame net with 500 micron mesh

B.2.1.5 Habitat Assessment:
At each survey site where fish data are collected, qualitative habitat surveys will be done within the same reach length (35 times the mean stream wetted width). The habitat survey methods include WDNR Qualitative Habitat Survey and Ohio EPA's Qualitative Habitat Evaluation Index (QHEI). Analyses of data derived from both habitat assessment methods will be done to evaluate method comparability and whether biotic response variables are more strongly correlated with data derived from one habitat assessment method or the other (Appendix E).

B.2.4 Field Forms
The Field Coordinator will keep field forms. Entries will be made on waterproof paper in pencil. Corrections will be made by a single lineout deletion, initialed and dated. Field Coordinator will be responsible for maintaining the following field forms:

1. A field data sheet will be made for each site visit. An example is shown in Appendix F. This sheet will document the time that sample is taken, the GPS coordinates of that location, any deviation from the original planned sampling location, and any other pertinent field observations associated with that sample. For split and duplicate field samples, the field data sheet must reference the original location.
2. Sample container labels, and Chain of Custody (COC) forms will be maintained as described in Section B.3 Sample Handling and Custody (see Appendix G).
3. Project specific Health and Safety documentation will be submitted as required.
4. A field notebook will serve as a diary of field activities and record of pertinent data not included on the other forms described above. Recorded information will include general site conditions, daily weather conditions, equipment use, equipment problems, etc.

B.2.5 Field Corrective Action

Corrective actions will be taken if any aspect of the sampling event differs from that planned. Under circumstances where corrective action is needed, the Field Coordinator will be notified and the situation researched and a decision made. Corrective actions will be documented in the field log at the time of decision, and will accompany all reports after analytical results are returned.

B.3 SAMPLING AND HANDLING CUSTODY

B.3.1 Sample Containers, Preservation and Maximum Holding Times

After processing, samples will be placed into the appropriate sample containers as summarized in Table 9. A field log shall be filled out for each sampling location (see Appendix F).

Table 9. Container Requirements and Sample Holding Times

Matrix	Analysis	# Jars Needed per site	# Jars Needed	Holding Time	Preservation	Bottle Type
Sediment	Metals	1 - Same jar can be used for metals/nutrients/TOC for sediments; if so, then need at least 8 oz glass jar	48	6 months	Wet ice. < 6ºC	Glass 16oz. jar
	Nutrients (TKN, TP, Ammonia)	1	0	28 days	Wet ice. < 6ºC	4oz wide mouth
	Pesticides	1 – same jar for pesticides/atrazine	48	14 days /40 days for extracts	Wet ice. < 6ºC	8 oz glass jar
	Atrazine	1	0	14 days /40 days for extracts	Wet ice. < 6ºC	8 oz glass jar
	TOC	1	0	28 days	Wet ice. < 6ºC	4 oz. wide mouth glass
	PAH	1	12	14 days	Wet ice. < 6ºC	8 oz glass jar
Water	Metals	1	72	6 months	HNO3 to pH<2, Wet ice 4ºC	Polyethylene > 500 mL
	Demand (BOD)	1- Same bottle can be used for BOD/TSS/TDS/ Sulfate/Chloride for water	314	48 hours	Wet ice. < 6ºC	1000 mL Polyethylene/Glass or 2-500ml poly/glass
	Demand (Sulfate, Chloride)	1	0	28 days	Wet ice. < 6ºC	100ml Polyethylene/Glass
	Demand (TDS /TSS)	1	0	7 days	Wet ice. < 6ºC	500 mL Polyethylene/Glass
	Nutrients (Ammonia, Nitrate-Nitrite-N,TP, TKN)	1	314	28 days	Cool, <6ºC, H2SO4 to pH < 2	500ml Polyethylene/Glass
	Atrazine	1 – same bottle for pesticides; 2 additional bottles for MS/MSD for 1/20 samples	72	7 days /40 days for extracts	Wet ice. < 6ºC	1 L glass bottle, amber, narrow mouth
	pesticides	1	0	7 days /40 days for extracts	Wet ice. < 6ºC	1 L glass bottle, amber, narrow mouth
	E.Coli	1	72	6 hours (24 hours if flagged)	Wet ice. < 10°C,	250 ml Polyethylene
	Chlorophyll-A (lab filtered)	1	72	48 hours	Wet ice. < 6°C	1 L Polyethylene

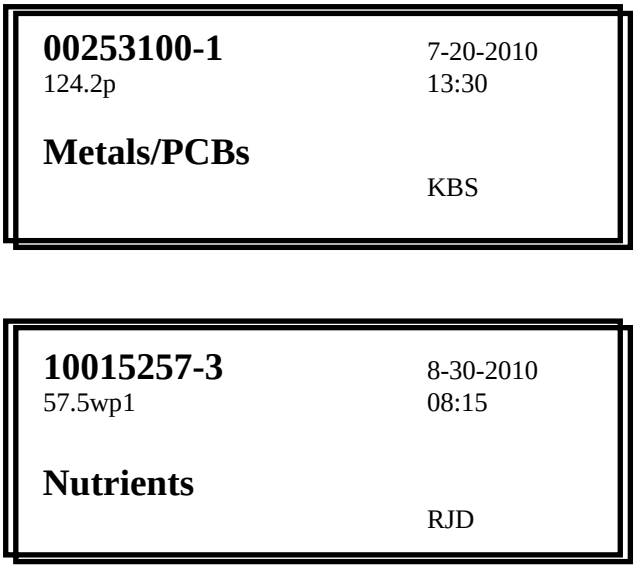
B.3.2 Sample Labeling

Each sample container shall be individually labeled using waterproof pen. The label should contain the following information:

- Sample identification: aaaaaaaa-b where: aaaaaaaa = SWIMS identification code, b = chemistry round (1 through 6).
- Site Name: as described above (B 1.3.1), between 4 and 8 letters or numerals.
- Sample Date (MM-DD-YYYY)
- Sample Time (HH:MM, on a 24-hour clock)
- Analysis to be performed (e.g. PCBs, metals, whole sediment toxicity, etc.)
- Sampler's Initials

An example label is shown in Figure 4. Clear tape will be placed over the label after the label has been completely filled out and attached to the sample container. The sample identification number and date of sample collection will be written on the sample container closure with a water proof marker.

Figure 4. Example Sample Labels



B.3.3 Shipment and Chain-of-Custody

After collection and labeling, all glass containers shall be placed in a zip-lock bag, and placed in an appropriate sample cooler with sufficient ice to maintain 4º C until receipt by the laboratory. Within 24 hours of sample collection, the samples will be sent to the respective analyzing laboratory. After samples are collected each day, the Field Coordinator shall be responsible for shipping and/or arranging pickup of samples. The Field Coordinator shall insure that:

1. The coolers contain sufficient ice to keep the samples at 4º C from time of collection and during the shipment process,
2. Are immobilized with bubble pack to reduce the risk of breakage,
3. The chain of custody form (see example in Appendix G) is properly filled out,
4. A copy of the chain-of-custody form shall be retained and provided to the Field Operations,
5. A copy of the chain-of-custody form will be placed in a "ziploc" bag and taped to the inside lid of the cooler,
6. The outside of the container will be shut using fiberglass or duct tape,
7. The laboratory name and address, as well as the return name and address, will be clearly labeled on the outside of the container (shipments to CRL must specify 10th Floor),
8. These samples will be sent to the laboratory by an overnight courier, and to ensure that the cooler does not run out of coolant while in the custody of the overnight delivery service, the samples must be shipped for delivery on the next calendar day. If a weekend or holiday will prevent delivery of the samples on the next calendar day, retain custody of the samples in the onsite refrigerator until after the weekend or holiday,

9. Receipts of bills of lading will be retained as part of the permanent documentation,
10. Commercial couriers are not required to sign off on the sample tracking form as long as it is sealed inside the sample cooler, and
11. Laboratories are contacted prior to shipment to insure they are prepared for sample arrival.

Note: *Each analyzing laboratory will supply chain-of custody forms to the Field Coordinator prior to the sampling event.*

Table 10 summarizes where each of the respective types of samples shall be shipped.

Table 10. Addresses for Shipment of Samples

Analysis Type	***Laboratory Contact Information***
Water and Sediment Chemistry	Amanda Wroble Central Regional Laboratory – EPA 536 S. Clark (ML-10C) 10th Floor Chicago, IL 60605 (312) 353-0375
Benthic Community Analysis	Dr. Kurt Schmude Lake Superior Research Institute U of Wisconsin – Superior 801 N. 28th St. Superior, WI 54880-2998 (715)-394-8421
Chlorophyll A (lab filtered)	DeWayne Kennedy-Parker Wisconsin State Lab of Hygiene, Metals 2601 Agriculture Drive Madison WI 53718 (608)-224-6282 fess@mail.slh.wisc.edu
Bacteria, *E. Coli*	Sharon Kluender Wisconsin State Lab of Hygiene, Water Microbiology 2601 Agriculture Drive Madison WI 53718 (608)-224-6262 hesk@mail.slh.wisc.edu
Water Chemistry	Tracy Fritsch Wisconsin State Lab of Hygiene, 2601 Agriculture Drive Madison WI 53718 (608)-224-6270 Tracy.fritsch@mail.slh.wis.edu
Water Chemistry QA/QC	Susan Hill Wisconsin State Lab of Hygiene, 2601 Agriculture Drive Madison WI 53718 (608)-224-6281 Susan.hill@mail.slh.wisc.edu

B.3.4 Receipt of Samples

Upon receipt of project samples, each laboratory shall

- Complete their portion of the chain-of-custody forms,
- Insure that the samples are maintained at < 4°C,
- If there are any sample shipment problems, the laboratory should contact the Technical Contact and/or WDNR Project Manager

B.4 ANALYTICAL METHOD REQUIREMENTS

B.4.1 Water and Sediment Chemistry

Water chemistry data will be compared to existing surface water quality criteria (WQCs) based on WDNR standards (Chapter NR 105, 2004). Sediment chemistry data will be compared to existing WDNR consensus-based sediment quality guidelines (SQGs) in *Solberg et al.* (2003). Often there are no criteria or guidelines determined for certain parameters; in these cases we listed available information, such as reporting limits, limits of detection for the Wisconsin Department of Agriculture, Trade, and Consumer Protection, and the Wisconsin state laboratory's limits of detection (LOD) and lower limits of quantitation (LOQ). Table 11 provides the required target concentration limits necessary to allow for water and sediment chemistry results to be compared directly to these screening criteria and guidelines. Target detection limits should be able to meet the target concentration limits.

Table 11. Criteria and Target Limits

Parameter	Water Criteria	Reporting Limits (water)	Units		Sed. Guidelines SDL/TEC/PEC (suggested detection level (always mg/L)/ threshold effect conc./ probable effect conc.)	Reporting Limit (sed.)	Units
Demand							
5 Day BOD		2	mg/L				
Chloride	395,000 ug/L	3	mg/L				
Total Dissolved Solids		20	mg/L				
Total Suspended Solids		5	mg/L				
Sulfate (anions)		.75	mg/L				
Nutrients							
Nitrogen/Ammonia (as N)	0.55	0.1	mg/L	Nitrogen/Ammonia (as N)	0.16/ /	89	mg/L
Nitrogen/Nitrate/Nitrite		0.25	mg/L	Nitrogen – Total Kjeldahl		125	mg/L
Nitrogen – Total Kjeldahl		0.5	mg/L	Phosphorus, Total	9.9/ /	560	mg/L
Phosphorus, Total	0.075/ 0.1	0.2	mg/L	TOC	0.2/ /	1	%
Metals							
Cadmium	3.82	2	µg/L	Arsenic	5/ 9.8/ 33	0.5	mg/L

Calcium		200	µg/L	Cadmium	0.6/ 0.99/ 5	.01	mg/L
Copper	21.57	5	µg/L	Copper	0.5/ 32/ 150	.01	mg/L
Iron		50	µg/L	Iron	/ 20,000/40,000	1	mg/L
Lead	54.71	15	µg/L	Lead	3/ 36/ 130	.1	mg/L
Magnesium		100	µg/L	Zinc	2/ 120/ 460	.2	mg/L
Zinc	220.7	30	µg/L	Mercury	0.015/ 0.18/ 1.1		mg/L
Pesticides							
4,4-DDD	0.011 ng/L	0.2	ug/L	4,4-DDD	/ 4.9/ 28	1.2	ug/L
4,4-DDE	0.011 ng/L	0.2	ug/L	4,4-DDE	/ 3.2/ 31	1.2	ug/L
4,4-DDT (ng/L)	0.065 ng/L	0.2	ug/L	4,4-DDT	0.01/ 4.2/ 63	1.2	ug/L
Total DDT (ng/L)	0.011 ng/L		ug/L	Total DDT	0.01/ 5.3/ 572		ug/L
Aldrin		0.1	ug/L	Aldrin	0.01/ 2/ 80	1.2	ug/L
alpha-BHC	0.0039	0.1	ug/L	alpha-BHC	/ 6/ 100	1.2	ug/L
alpha-Chlordane		0.1	ug/L	alpha-Chlordane		1.2	ug/L
beta-BHC		0.1	ug/L	beta-BHC	/ 5/ 210	1.2	ug/L
delta-BHC		0.1	ug/L	delta-BHC		2.4	ug/L
Dieldrin (ng/L)	0.0027 ng/L	0.2	ug/L	Dieldrin	0.01/ 1.9/ 62	1.2	ug/L
Endosulfan I		0.1	ug/L	Endosulfan I		2.4	ug/L
Endosulfan II		0.2	ug/L	Endosulfan II		2.1	ug/L
Endosulfan sulfate		0.2	ug/L	Endosulfan sulfate		1.8	ug/L
Endrin	0.072	0.2	ug/L	Endrin	0.01/ 2.2/ 207	1.2	ug/L
Endrin aldehyde		0.2	ug/L	Endrin aldehyde		1.5	ug/L
Endrin ketone		0.2	ug/L	Endrin ketone		2.7	ug/L
gamma-BHC (Lindane)	0.019	0.1	ug/L	gamma-BHC (Lindane)	0.01/ 3/ 5	1.2	ug/L
gamma-Chlordane		0.1	ug/L	gamma-Chlordane		1.2	ug/L
Heptachlor		0.1	ug/L	Heptachlor	0.01/ /	1.2	ug/L
Heptachlor epoxide		0.1	ug/L	Heptachlor epoxide	/ 2.5/ 16	1.2	ug/L
Methoxychlor		0.2	ug/L	Methoxychlor		1.5	ug/L
				Acenapthene	/ 6.7/ 48		ug/L
				Acenaphthylene	/ 5.9/ 67		ug/L
				Anthracene	/ 57.2/ 451		ug/L
				Fluroene	/ 77.4/ 307		ug/L
				Naphthalene	/ 176/ 369		ug/L
				2-methylnapthalene	/ 20.2/ 111		ug/L
				Pehnanthrene	/ 204/ 687		ug/L
				Benz(a)anthracene	/ 108/ 579		ug/L
				Benzo(a)pyrene	/ 150/ 800		ug/L
				Benzo(e)pyrene	/ 150/ 800		ug/L
				Benzo(b)fluoranthene	/ 240/ 6,820		ug/L
				Benzo(k)fluuroanthene	/ 240/ 6,820		ug/L
				Benzo(g,h,l)perylene	/ 170/ 1,685		ug/L
				Chrysene	/ 166/ 728		ug/L
				Dibenz(a,h)anthracene	/ 33/ 84		ug/L
				Fluoranthene	/ 423/ 1,327		ug/L
				Indeno(1,2,3-cd)pyrene	/ 200/ 1,700		ug/L
				Pyrene	/ 195/ 858		ug/L
				Total PAHs	/ 1,610/ 22,800		ug/L
				Atrazine			

B.4.2 Laboratory Analysis

Table 12 identifies analytical methods to be followed for any laboratory analysis. Any deviation from analytical methods should be notified and approved by the Technical Contact and a reference to the method should be included in the final report.

Table 12. Laboratory Analysis and Preparation Methods

Sediments

Analyte	*Analysis Method*	*Sample Preparation Method* *(CRL-SOP)*	*Sample Cleanup Method* *(CRL-SOP)*	*Laboratory SOP* *Analysis Method*
TKN	EPA 351.2	N/A	N/A	AIG022
Ammonia	EPA 350.1	N/A	N/A	AIG022
Total Phosphorus	EPA 365.4	N/A	N/A	AIG022
TOC	Black, C.A., et al. 1965. Organic Carbon. Methods of Soil Analysis, Part 2. Chemical and Microbiological Properties. American Society of Agronomy. pp. 1367-1378.	N/A	N/A	AIG009
Metals	EPA 6010B -As EPA 200.7	EPA 200.2 (Metals 025)	N/A	Metals004 -Ag
PAHs	EPA 8270C	EPA 3545	EPA Method 3640A	
Pesticides	EPA 8081A	EPA 3545 (GC013)	3620 (GC015) 3660(GC019)	GC009, GC001
Atrazine	EPA 8081A	EPA 3545 (GC013)	3620 (GC015) 3660(GC019)	GC001

Water

Analyte	*Analysis Method*	*Sample Preparation Method* *(SLOH-SOP)*	*Sample Cleanup Method* *(SLOH-SOP)*	*Laboratory SOP* *Analysis Method*
BOD	SLOH I180ALT	N/A	N/A	SM5210 B
Chloride	SLOH I240FLT	N/A	N/A	EPA 325.2
Sulfate	SLOH 1600ELT	N/A	N/A	EPA 375.2
TSS	SLOH 1650JLT	N/A	N/A	EPA160.2
TDS	N/A	N/A	N/A	640ILD

TKN	N/A	N/A	N/A	1470DLT
Nitrate/Nitrite N	SLOH 1460GLT	N/A	N/A	EPA 325.2
Ammonia	N/A	N/A	N/A	1440NLD
Total Phosphorus	SLOH 1520PLT	N/A	N/A	EPA 365.1
Metals	EPA 200.7	EPA 200.2 (Metals 025)	N/A	Metals003
Pesticides	EPA 8081A	EPA 3535 (GC011)	3620 (GC015) 3660(GC019)	GC001
Atrazine	EPA 8081A	EPA 3535 (GC011)	3620 (GC015) 3660(GC019)	GC001

n/a = not applicable

Macroinvertebrates:
Macroinvertebrate samples will be processed and analyzed using WDNR standardized operating procedures. Briefly stated: in the lab, field samples are placed in gridded trays; a random (numbered) grid square is selected and all macronvertebrate specimens within the grid square are picked from the tray and placed in a specimen jar; all macroinvertebrates within the grid square are picked; if the target number of 125+ organisms is met the sub-sampling is completed; if the target number is not met the next highest number square is picked and all macroinvertebrates are removed for identification; this process is repeated until the target number of 125+ organisms is reached. The picked specimens are identified by a qualified taxonomist to the lowest practical taxonomic level (typically genus or species) using regional taxonomic identification keys. Taxonomic analytical results data (taxa name and counts) are entered into a database and a number of metrics and indexes are computed for each sample.

B.4.3 Laboratory Turnaround Time Requirements
All sample analysis will be returned to Technical Contact within 90 days of processing. Notification of any deviation from this schedule must be made to Technical Contact.

B.5 QUALITY CONTROL ELEMENTS

All analytical procedures are documented in writing as SOPs and each SOP includes QC information, which addresses the minimum QC requirements for the procedure (see Appendices A – C). The internal quality control checks might differ slightly for each individual procedure. Examples of some of the QC samples that will be used during this project include:

- Method blanks
- Reagent/preparation blanks
- Instrument blanks
- Surrogate spikes
- Analytical spikes
- Field duplicates and splits

- Laboratory duplicates
- Matrix Spike/Matrix Spike Duplicate
- Laboratory control standards
- Internal standard areas for GC/MS analysis; control limits.

The actual QC sample requirements will be dictated by the method requirements. Details on the use of each QC check are provided in the analytical SOPs provided for each measurement. Method detection limits will be calculated for each analyte.

Note: Instrument calibration concentrations, method validation procedures, internal quality control protocols, analytical routines, maintenance and corrective actions, and the data reduction procedures are included in and will be performed as specified in the Standard Operation Procedures as required by the designated analytical methods.

B.5.1 Corrective Actions

B.5.1.1 Field
Corrective actions should only be implemented after approval by the Project Managers. If immediate corrective action is required, approvals secured by telephone from the Project Manager should be documented in an additional memorandum.

B.5.1.2 Laboratory
For noncompliance problems, a formal corrective action program will be determined and implemented at the time the problem is identified. Each Contract laboratory shall issue a nonconformance report for each nonconformance condition.

Corrective actions in the laboratory may occur prior to, during, and after initial analysis. A number of conditions, such as broken sample containers, multiple phases, and potentially high concentration samples may be identified during sample log-in or just prior to analysis. Following consultation with laboratory analysts and section leaders, it may be necessary for the Laboratory QA Officer to approve the implementation of corrective actions. The submitted SOPs specify some conditions during or after analysis that may automatically trigger corrective actions of samples, including additional sample extract cleanup and automatic re-injection/reanalysis when certain quality control criteria are not met.

Corrective actions are required whenever an out-of-control event or potential out-of-control event is noted. The investigative action taken is somewhat dependent on the analysis and the event.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the warning or acceptable windows for precision and accuracy
- Blanks contain target analytes above acceptable levels
- Undesirable trends are detected in spike recoveries or RPD between splits

- There are unusual changes in detection limits
- QC limits for sediment toxicity tests are not met
- Deficiencies are detected by the Laboratory and/or USEPA QA Officer(s) during any internal or external audits or from the results of performance evaluation samples
- Inquiries concerning data quality are received.

Corrective action procedures are often handled at the bench level by the analyst, who reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, experimental set-up, and so on. If the problem persists or cannot be identified, the matter is referred to the Laboratory Project Manager and/or Laboratory QA Officer for further investigation. Once resolved, full documentation of the corrective action procedure is filed with the Laboratory QA Officer.

These corrective actions are performed prior to release of the data from the laboratories. The corrective actions will be documented in both the laboratories corrective action log and the narrative data report sent from the laboratory to the technical contact.

If corrective action does not rectify the situation, the analytical laboratory will contact the USEPA Project Manager and or Technical Contact, to discuss details of the corrective actions and required future actions. The benthic laboratory will contact the WDNR Project Manager. In general communications from the laboratories should follow the chain-of-command as shown in Figure 1.

B.5.2 Procedures used to Calculate QC Statistics

B.5.2.1 Bias

Bias is the systematic or persistent distortion of a measurement process that causes errors in one direction. Bias assessments for environmental measurements are made using personnel, equipment, and spiking materials or reference materials as independent as possible from those used in the calibration of the measurement system. When possible, bias assessments should be based on analysis of spiked samples rather than reference materials so that the effect of the matrix on recovery is incorporated into the assessment. A documented spiking protocol and consistency in following that protocol are important to obtaining meaningful data quality estimates. Spikes should be added at concentrations approximately at the mid-range. Spiked samples shall be used in accordance with the specified method.

Bias will be assessed through the use of certified reference materials (CRMs), standard reference materials (SRMs: a reference material certified by the U.S. National Institute of Standards Technology [U.S. NIST]), or other standards, such as, matrix spikes.

Matrix spike and matrix spike duplicate samples (MS/MSD) also will be used to assess bias as determined by the laboratory. Acceptable recovery values will be within the recoveries specified as determined by the laboratory. Control samples for assessing bias will be analyzed at a rate as specified in the analytical SOPs or specified analytical methods.

B.5.2.2 Precision

Precision is a measure of agreement among replicate measurements of the same property, under prescribed similar conditions. This agreement is calculated as either the range (R) or as the standard deviation(s). It may also be expressed as a percentage of the mean of the measurements, such as relative percent difference (RPD) or relative standard deviation (RSD) (for three or more replicates).

Laboratory precision is assessed through the collection and measurement of laboratory duplicates. The laboratories shall follow the protocols in the specified method and corresponding SOPs regarding the frequency of laboratory duplicates. This allows intra-laboratory precision information to be obtained on sample acquisition, handling, shipping, storage, preparation, and analysis. Both samples can be carried through the steps in the measurement process together to provide an estimate of short-term precision. An estimate of long-term precision can be obtained by separating the two samples and processing them at different times, or by different people, and/or analyzed using different instruments. Acceptable RPDs will be in accordance to those specified in Table 12.

For duplicate measurements, relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100\%$$

RPD = relative percent difference
D_1 = sample value
D_2 = duplicate sample value

For three or more replicates:

$$RSD = (s/x) \times 100$$

RSD = relative standard deviation
s = standard deviation of three or more results
x = mean of three or more results

Standard deviation is defined as follows:

$$s = ((\sum(y_i - \text{mean } y)^2 \times 1/(n-1)))^{0.5}$$

s = standard deviation
y_i = measured value of the replicate
mean y = mean of replicate measurements
n = number of replicates

Field duplicates are collected from slightly different locations (<3 feet away) than the original sample. Field duplicates provide a measure of the variability inherent in the entire sampling and analysis process, including, small-scale variability of site conditions, consistency of sampling and homogenization process, and laboratory analysis. The field duplicates provide a general picture of the amount of variability that can be expected between this and future sampling events, even if site conditions do not change substantially. This is an important consideration since this data will be compared to historical and possible future sampling events. Since site variability can greatly influence RPD for field duplicates, no strict RPD measures will be used to evaluate

this measure. However, most sediment guidance recommends that RPD measures for field duplicates be in the same range as that for field splits.

Ohio EPA (1987) extensively tested the reproducibility, accuracy, and precision of their boat electrofishing sampling protocols in both wadeable streams and non-wadeable rivers. Based on a combination of data analyses from specially designed methods testing studies and the aggregate Ohio database, the reproducibility of an IBI score was determined to be 4 units out of a 12 to 60 scoring scale (Rankin and Yoder [1999] later revised the scoring range, 0-60). Rankin and Yoder (1990) showed coefficient of variations (CV) were on the order of 8-10% at least impacted and high quality sites. CVs increased at sites with lower IBI scores, presumably due to the effect of stressors at increasingly impacted sites. Fore et al. (1993) performed more extensive statistical analyses of the Ohio database and determined that IBI scores were reproducible to an error margin of 2-3 units. Their power analysis confirmed that the Ohio IBI was capable of distinguishing 6 discrete scoring ranges that approximate the delineations of the IBI scale into the qualitative descriptions of exceptional, good, fair, poor, and very poor. Angermier and Karr (1986) analyzed other statistical properties of the IBI focusing on the extent of redundancy among metrics. The results of their analysis showed that careful construction and derivation of an IBI following the original guidance of Karr et al. (1986) should produce a robust and non-redundant set of metrics.

Quality control limits for Precision, Accuracy, and Completeness are summarized in Table 13.

Table 13. Quality Control Limits (Sediment/Water Matrices)

Analyte	Precision (RPD)	Accuracy (%)	Completeness (%)
Metals	≤35	*As determined by Laboratory*	80%
Pesticides	≤40	*As determined by Laboratory*	80%
Nutrients	≤40	*As determined by Laboratory*	80%
TOC	≤20	50-130	80%
Demand		*As determined by Laboratory*	80%

RPD = Relative Percent Difference

B.5.2.3 Accuracy

Accuracy measures how close analytical results are to a true or expected value. Accuracy objectives will be determined by calculating the percent recovery range of laboratory matrix spikes and matrix spike duplicates. Accuracy measures are calculated using the RPD between the expected value and the actual analytical results.

Accuracy can also be examined in terms of the assessment produced by the subject method. Biological assessments are viewed as a direct measure of the aquatic life protection goals of the Clean Water Act (CWA) and State water quality standards (as opposed to the surrogate assessment provided by chemical water quality criteria). This has given rise to the concept and interest in biological criteria and adoption by U.S. EPA of a national program (U.S. EPA 1990), methods (Barbour et al. 1997), and the development of formal implementation procedures (U.S. EPA Aquatic Life Use Working Group). The issue at stake here is the accuracy of the delineation of waters as impaired or unimpaired for CWA purposes (e.g., TMDLs). Historically, States and U.S. EPA based these decisions on chemical water quality data and comparison to State and national water quality criteria. However, studies that compared the relative performance of chemical and biological data and their respective abilities to detect impairment showed that biological data was far superior in its ability to detect impairment and minimize type II assessment error (Rankin and Yoder 1990b; Yoder and Rankin 1998). It is implicit in these studies that the better standardized and calibrated the biological assessment method and assessment criteria, the more able the method is to detect impairment and establish a relative degree of departure from a baseline criterion.

B.5.2.4 Representativeness

Representativeness is the degree to which the sampling data properly characterize the study environment. For the field-sampling phase, the sampling sites reasonably cover the Upper Yellow River and tributaries.

The collection of biological data includes the use of standardized sampling procedures designed to produce a sufficiently representative sample of the assemblage at a site with a reasonable expenditure of effort (i.e., 1-3 hours/site). As such this type of assessment is distinguished from the much more resource intensive efforts using multiple collection gear and those required to obtain estimates of population (standing crop) or a complete inventory of all species present. Numerous

large river IBI development studies that followed Gammon's pioneering work have substantially confirmed the utility and representativeness of the approach. Lyons et al. (2001) correctly observed that single gear assessments might not be as useful for rare or single species issues or for detailed management needs such as stock assessments of commercially or recreationally important species. However, broad agreement between overall assemblage condition assessments and the correspondence of suitable conditions for rare species and management goals has been demonstrated (Hughes and Gammon 1987; Yoder and Rankin 1995).

In the analytical phase, and as specified elsewhere in this document, appropriate sample storage and preservation, and sample homogenization will insure that the samples analyzed adequately reflect conditions as they existed in the natural environment.

B.5.2.5 Comparability

Comparability states the confidence with which one data set can be compared to another. Comparability will be enhanced by the consistent use of standardized sampling methods and specified protocols for the sampling phase and through the use of standard documented methodologies for analyte determinations. Any deviations from the standardized, selected methods or protocols will be clearly documented by the laboratories and noted in the final analytical report. There are a number of issues that can make two data sets comparable, and the presence of each of the following items enhances their comparability:

- Two data sets should contain the same set of variables of interest
- Units in which these variables were measured should be convertible to a common metric
- Similar analytical procedures and quality assurance should be used to collect data for both data sets
- Time measurements of certain characteristics (variables) should be similar for both data sets
- Measuring devices used for both data sets should have approximately similar detection levels
- Rules for excluding certain types of observations from both samples should be similar
- Samples within data sets should be selected in a similar manner
- Sampling frames from which the samples were selected should be similar
- Number of observations in both data sets should be of the same order or magnitude.

These characteristics vary in importance depending on the final use of the data. The closer two data sets are with regard to these characteristics, the more appropriate it will be to compare them. Large differences between characteristics may be of only minor importance, depending on the decision that is to be made from the data.

While there is no theoretical upper limit to many of the raw data parameters that comprise the baseline data that will be produced by the proposed study, most have practically limited expectations. The practical range of these parameters is dependent on the natural attributes of

the regional fish assemblage and the effectiveness of the sampling gear and procedure. For example, in a warmwater river in Ohio we expect boat electrofishing to produce a sample of 20-30 species and several hundred fish among those species. In exceptional quality rivers, the number of species might increase to more than 35-40 among thousands of individuals. In the large cold water rivers of the western U.S., many fewer species and individuals are usually collected. However, in terms of regional reference condition and potential, the resulting biological assessment should rate the samples from Ohio and the Western U.S. the same with respect to its similarity to or departure from a regional reference condition. This is critical to establishing biological assessments that are comparable across the U.S. Thus the derivation of reference condition is a critical step in the bioassessment process and is one of the factors that influences comparability.

The resulting assessments and biological indices have discrete scoring ranges, within which the raw data is stratified and compressed. For example, the original IBI and many of its contemporary applications used a scoring range of 12-60, i.e., metric scores of 5, 3, and 1 are assigned to each of 12 metrics. Newly developed IBIs have employed a scoring range of 0-100 (e.g., Lyons et al. 2001; Mebane et al. 2003), which is intuitively more meaningful as a theoretical scoring range and communication tool. The rigor, adequacy of the method, development, and calibration ultimately determines the accuracy, precision, and reproducibility of the index, its statistical rigor, and its resulting assessment.

For this investigation, comparability will be satisfied by ensuring that the field sampling plan is followed, standard EPA Methods of analysis are used for sample analysis and that proper sampling techniques are used.

B.5.2.6 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that the project is expected to obtain under normal conditions. Field completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. Field completeness objectives for this project will be greater than 90% because sample locations were picked based on sampleability. However, due to river currents and areas with little sediment deposition, an invalid sample is the analysis of sediment depth and the recording of “No Sediment Present”. Sites with no water will be noted. If over 3 sites are dropped in levels 1-4 than sites will be replaced if over 4 sites are dropped in Levels 5 and 6 than sites will be replaced. Sites that are dangerous to sample or where access is denied by landowners may result in moving of the site to the nearest sampleable location. Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. Laboratory completeness for this project will be greater than 80% of the total number of samples submitted to the analytical laboratories.

The calculation for percent completeness is as follows:

$$\%C = 100\% \times (V/n)$$

%C = percent completeness

V = number of valid[(1)] measurements
n = number of measurements planned

[(1)] For this sampling event, a valid measurement is defined as the arrival at a sampling location and collection and analysis of a sediment sample.

B.6 INSTRUMENT EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE REQUIREMENTS

The purpose of this section is to discuss the procedures used to verify that all instruments and equipment are maintained in sound operating condition, and are capable of operating at acceptable performance levels.

B.6.1 Testing, Inspection, and Maintenance

The success of this project is dependent on well functioning field, analytical, and toxicological equipment. Preventative maintenance of this equipment is the key to reduce possible project delays due to faulty equipment.

As part of each laboratory's QA/QC program, a routine preventative maintenance program will be conducted to minimize the occurrence of instrument failure and other system malfunctions. All laboratory instruments are maintained in accordance with manufacturer's specifications and the requirements of the specific method employed. This maintenance is carried out on a regular, scheduled basis and is documented in the laboratory instrument service logbook for each instrument.

B.7 INSTRUMENT CALIBRATION AND FREQUENCY

This section concerns the calibration procedures that will be used for instrumental analytical methods and other measurement methods that are used in environmental measurements. Calibration is defined as checking physical measurements against accepted standards.

B.7.1 Calibration Methods That Will Be Used For Each Instrument

Instrument calibration procedures are dependent on the method and corresponding SOP. All ongoing calibration measurements must be within the requirements of the corresponding SOP to be considered adequate.

Equipment logbooks will be maintained at each laboratory, in which will be recorded the usage, maintenance, calibration, and repair of instrumentation. These logbooks will be available during any audits that may be conducted.

B.8 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

The purpose of this section is to establish and document a system for inspecting and accepting all supplies and consumables that may directly or indirectly affect the quality of the project or task.

B.8.1 Identification of Critical Supplies and Consumables

Critical supplies and consumables include sample bottles, gases, reagents, hoses, materials for decontamination activities, ethanol and distilled/deionized water. Each of the laboratories will utilize high quality supplies and consumables to reduce the chances of contaminating the samples. All water purification systems are tested on a regular basis to ensure that water produced is acceptable for use. Solvent blanks are run to verify the purity of solvents used in the organic analyses. The laboratories may also incorporate other measures, such as the dedicated use of glassware for certain analyses (e.g., inorganics, organics).

B.8.2 Establishing Acceptance Criteria

Acceptance criteria must be consistent with overall project technical and quality criteria. Each of the laboratories should utilize their own acceptance criteria for normal operations with analyzing and/or testing contaminated sediments.

B.8.3 Inspection of Acceptance Testing Requirements and Procedures

Each laboratory should document inspections of acceptance testing, including procedures to be followed, individuals responsible, and frequency of evaluation. In addition, handling and storage conditions for supplies and consumables should be documented.

B.8.4 Tracking and Quality Verification of Supplies and Consumables

Procedures should be established to ensure that inspections or acceptance testing of supplies and consumables are adequately documented by permanent, dated, and signed records or logs that uniquely identify the critical supplies or consumables, the date received, the date tested, the date to be retested (if applicable), and the expiration date. The responsible individual(s) at each laboratory should keep these records. In order to track supplies and consumables, labels with the information on receipt and testing should be used. These or similar procedures should be established to enable project personnel to: 1) verify, prior to use, that critical supplies and consumables meet the project objectives; and 2) ensure that supplies and consumables that have not been tested, have expired, or do not meet acceptance criteria are not used for the project.

B.9 DATA ACQUISITION REQUIREMENTS FOR NON-DIRECT MEASUREMENTS

WDNR will make an effort to access historical information about the fish, macroinvertebrate fauna, water chemistry and sediment chemistry of the study area. This will be especially valuable in evaluating the historical trends through time. Some expert judgment may be necessary to evaluate the quality and accuracy of this information.

Additionally, sets of screening values will be used to evaluate samples collected during this survey. All parameter data will be compared to existing sediment quality guidelines and water quality standards available in *MacDonald et. al*. (2000), Chapter NR 105, and *Solberg et al.* (2003).

B.10 DATA MANAGEMENT

This section will present an overview of all mathematical operations and analyses performed on raw data to change their form of expression, location, quantity, or dimensionality. These operations include data recording, validation, transformation, transmittal, reduction, analysis, management, storage, and retrieval.

B.10.1 Data Recording

The gathering and organization of data for this project will begin with field observations. Handwritten accounts of each individual sampling event will be recorded on data log sheets.

B.10.2 Data Validation

After analytical data has been returned, Technical Contact will verify the quality of the data. In-field sampling technique, results, returned analytical data will all be taken into consideration when verifying the data.

B.10.3 Data Transformations

Transformations do not occur when analytical data is entered into the database. Transformations can, however, be performed during database query. For example, a parameter that had been analyzed for, by the lab, in parts per billion, can be extracted to show results in parts per million. This transformation is only temporary, and does not alter the database in any way.

B.10.4 Data Transmittal

Field sample forms will accompany all samples taken during this sampling event (See Appendix F). These forms will be preserved after the sampling event has been completed and kept on record with WDNR.

B.10.5 Data Analysis

Both WDNR and the USEPA group will perform data analysis for this project.

B.10.6 Data Tracking

The time-frame for completing this sampling event will be tracked internally by the USEPA for completion of goals.

B.10.7 Data Storage and Retrieval

USEPA will retain all project documentation until the summary report based on the validated data is completed. At that point, all project documentation and reports will be submitted to WDNR for storage. WDNR will capture all fish assemblage, water column water chemistry,

Wisconsin Qualitative Habitat Survey and macroinvertebrate data, and subsequent study reports in the Departments SWIMS database.

SECTION C - ASSESSMENT AND OVERSIGHT

C.1 ASSESSMENT AND RESPONSE ACTIONS

During the planning process, many options for sampling design, sample handling, sample cleanup and analysis, and data reduction are evaluated and chosen for the project. In order to ensure that the data collection is conducted as planned, a process of evaluation and validation is necessary. This section of the QAPP describes the internal and external checks necessary to ensure that:

- All elements of the QAPP are correctly implemented as prescribed.
- The quality of the data generated by implementation of the QAPP is adequate.
- Corrective actions, when needed, are implemented in a timely manner and their effectiveness is confirmed.

The most important part of this section is documenting all planned internal assessments. Generally, internal assessments are initiated or performed by the QA Officers of the respective organizations.

C.1.1 Assessment of Subsidiary Organizations

Two types of assessments of the subsidiary organizations can be performed as described below.

- *Management Systems Review (MSR).* A form of management assessment, this process is a qualitative assessment of a data collection operation or organization to establish whether the prevailing quality management structure, policies, practices, and procedures are adequate for ensuring that the type and quality of data needed are obtained. The MSR is used to ensure that sufficient management controls are in place and carried out by the organization to adequately plan, implement, and assess the results of the project.
- *Readiness Reviews.* A readiness review is a technical check to determine if all components of the project are in place so that work can commence on a specific phase.

It is anticipated that a readiness review by each contract laboratory project manager will be sufficient for this project. No management systems review is anticipated for this project.

C.1.2 Assessment of Project Activities

Assessment of project activities can involve the following tasks:

- Surveillance
- Technical Systems Audit (TSA)
- Performance Evaluation (PE)
- Audit of Data Quality (ADQ)
- Peer Review

- Data Quality Assessment.

Surveillance will be the primary assessment technique of project activities. This will most readily occur by the Project Officer and/or QA Officer of each laboratory.

C.1.3 Number, Frequency, and Types of Assessments
Due to the short-term nature of this project for the laboratories, no types of assessments are planned other than general surveillance.

C.1.4 Assessment Personnel
Internal audits of the laboratories are regularly performed by their respective QA Officers.

C.1.5 Schedule of Assessment Activities
External audits by the Project Managers is up to his/her discretion. The scheduling of regular internal audits at labs is at the discretion of the respective QA Officers.

C.1.6 Reporting and Resolution of Issues
Any audits or other assessments that reveal findings of practice or procedure that do not conform to the written QAPP need to be corrected as soon as possible. The Laboratory Manager and/or Laboratory QA Officer need to be informed immediately of critical deviations that compromise the acceptability of the test. For any critical deviations from the QAPP (i.e., elevated detection levels, surrogate recoveries outside control limits, etc.) that cannot be corrected within the laboratories standard procedure, the Laboratory Project Manager must contact the Site Coordinators and or WDNR Project Manager within 24-hours of being informed of the deviation at which time the laboratory project manager should be ready to provide suggestions for corrective action. For non-critical deviations, the USEPA Project Manager and or WDNR Project Manager need to be informed by the next business day.

C.2 REPORTS TO MANAGEMENT

C.2.1 Responsible Organizations
Written QC data and appropriate QA/QC reports generated by the laboratories shall be included in the Analytical Data Report. The QC section of the Analytical Data Report should include the QC data (including results, recoveries, and RPDs), any non-conformance reports, and chains of custody. The report should give detailed results of analysis of QC samples, and provide information on the precision, accuracy, and completeness for each sample run. These written reports will note any significant QA/QC problems encountered during sample analyses, as well as state the corrective actions taken.

SECTION D – DATA VALIDATION AND USABILITY

The USEPA Project Manager and WDNR Project Manager will make a final decision regarding the validity and usability of the data collected during this project. The Project Managers will evaluate the entire sample collection, analysis, and data reporting processes to determine if the data is of sufficient quality to meet project objectives. Data validation involves all procedures used to accept or reject data after collection and prior to use. These include screening, editing, verifying, and reviewing through external performance evaluation audits. Data verification procedures ensure that objectives for data precision and bias will be met, that data will be generated in accordance with the QA project plan and SOPs, and that data are traceable and defensible. The process is both qualitative and quantitative and is used to evaluate the project as a whole.

D.1.1 Procedures Used to Verify Field Data

Procedures to evaluate field data for this project primarily include checking for transcription errors and reviewing field notebooks. This task will be the responsibility of the site coordinators.

D.1.2 Procedures Used to Verify Laboratory Data

USEPA staff and/or WDNR may conduct a systematic review of the analytical data for compliance with the established QC criteria based on the spike, duplicate, and blank results provided by the laboratories. All technical holding times will be reviewed, the laboratory analytical instrument performance will be evaluated, and results of initial and continuing calibration will be reviewed and evaluated.

The data review will identify any out-of-control data points and data omissions. Decisions to repeat sample collection and analysis may be made by the USEPA Project Manager based on the extent of the deficiencies and their importance in the overall context of the project.

Additionally, the Field Operations will compare all field duplicates for RPD. Based on the results of these comparisons, the USEPA project manager will determine the acceptability of the data. One hundred percent of the analytical data will be verified and validated. Reconciliation of duplicates and field splits shall be the responsibility of the Field Operations.

Finally, the Field Operations may compare the laboratory methods and results to the QA/QC Review checklists contained in Appendix H. Separate checklists are for chemistry data. Any critical problems identified by these checklists that we are unable to rectify through corrective actions may be cause for rejecting portions or all of the data provided.

SECTION E – REFERENCES

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