Report Concerning the Sampling Program for the Oklahoma Poultry Litigation

Prepared by:

Darren L. Brown, P.G.
Camp Dresser & McKee Inc.
345 Riverview, Ste 520
Wichita, KS 67026

Submitted on
May 15, 2008

Darren L. Brown, P.G.

I have worked for Camp, Dresser and McKee ("CDM") since August 1987, with a five-month hiatus between August 1990 and January 1991 and a two-year hiatus between July 2003 and July 2005. I have worked in the environmental consulting field since May 1986. I earned a Bachelor of Science Degree in Geological Engineering from the Colorado School of Mines in 1984 and Master of Science Degree in Geology from the University of Oklahoma in 1987. I am a Licensed Geologist in the State of Kansas (License No. 403) and received my Engineer in Training Certification from the State of Colorado in 1984 (#12425).

I have been the project manager for the Oklahoma Poultry Project since 2006. In addition to project management, my duties have included: working to develop the groundwater sampling program, the soil and litter sampling program, and the 2006 and 2007 river sampling programs; staffing and coordinating all field programs in 2006 and 2007; training and/or supervising the training of field staff; and field sampling, including soil and litter sampling, when necessary. Sampling programs conducted while I have been project manager include: litter and soil sampling; high and base flow sampling; edge of field (runoff) sampling; groundwater sampling (both residential well sampling and direct push sampling); spring sampling; river sampling (including fish and benthic macroinvertebrate sampling); lake sampling (Tenkiller Ferry Reservoir, Stockton, and Broken Bow); public water supply sampling; and manure sampling.

My development and scoping of the soil and litter sampling programs, the groundwater sampling program, and most of the other programs identified above was based upon my twenty-one years of environmental consulting experience. I have developed work plans, field sampling plans, and quality assurance project plans for the United States Environmental Protection Agency (US EPA) Superfund Sites under the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA); various state regulatory agencies; and private clients.

I have led portions of the field sampling programs for five separate US EPA Superfund projects. I have developed the scopes of work, written the work plans, conducted and/or supervised the conduct of the field investigation programs, written the reports, and served as project manager on several multi-million dollar state-lead Remedial Investigation/ Feasibility Study (RI/FS) programs.

1.0 Introduction

Field investigations in support of the State of Oklahoma's litigation against the poultry industry integrators were conducted between 2005 and 2008. The field investigations were conducted under a series of Standard Operating Procedures (SOPs). The purpose of the SOPs was to ensure that the work was conducted in a consistent manner. The SOPs were designed to provide data that met the data quality

objectives for the program. Data quality objectives are provided in Roger Olsen's Expert Witness Report.

Objectives

The purpose of this document is to present a summary of the field investigation efforts associated with the Illinois River Watershed 2005 through 2008 field investigations. The SOPs under which the investigations occurred will be introduced. This document will also provide a discussion of the training and supervision involved with the program as well as documentation and support structure to assist the court in its understanding of how the field investigation program was implemented.

Summary of Sampling Program

The field investigation program was conducted to investigate and document the links between the application of poultry waste on the fields within the Illinois River Watershed and the impacts on the soils, sediments, groundwater, surface water, and aquatic biology within the watershed. The field investigation program consisted of the following.

- Poultry litter sampling
- Manure sampling of beef cattle, dairy cattle, ducks, geese, swine, and humans
- Soil sampling at litter applied and non-litter applied properties
- Sediment sampling within the streams of the watershed
- Sediment sampling within Tenkiller Ferry Reservoir (Lake Tenkiller) and two lakes outside the watershed
- Water quality sampling within the streams and rivers of the Illinois River Watershed and at two streams outside of the watershed
- Water quality sampling within Lake Tenkiller and two lakes outside the watershed
- Aquatic biological observations and sampling within the streams, rivers, and Lake Tenkiller.

Report Structure

The remainder of this report discusses the field sampling program for the Illinois River Watershed investigations (also referred to as the Oklahoma Poultry Project). Section 2 provides an overview of the SOPs used during the field sampling program. Section 3 discusses the training, supervision, and chain-of-command during the field sampling program. Section 4 discusses the general structure of the SOPs and specific activities associated with the SOP. Section 5 presents an overall evaluation of the field

program. Section 6 provides references for this report. Section 7 provides the Rule 26 disclosure information including considered material.

2.0 An Overview of Standard Operating Procedures

The purpose of a SOP is to provide a standard working tool that can be used to document technical activities as well as quality control and quality management. The 2007 EPA guidance document replaces EPA's March 2001 Guidance for Preparing Standard Operating Procedures (SOPs) EPA/240/B-01-004. The newer document provides examples of both technical and administrative SOPs.

The SOPs for this program were developed consistent with the US EPA guidelines for the preparation of SOPs. The US EPA SOP guidance document is:

> Guidance for Preparing Standard Operating Procedures (G-6) (PDF 60pp, 336K About PDF) - April 2007, EPA/600/B-07/001.

The field investigation program conducted during this investigation was conducted under the protocols presented in the SOPs. These SOPs provide the basis for the work conducted; the procedures by which the work was to be performed; the sample containers and preservatives to be used; the analyses that were to be conducted; and the decontamination procedures required.

As the field program was conducted, the SOPs were revised for a variety of reasons. The revision number is provided in the SOP as well as the revision dates. The primary reasons for the revisions were:

- To add additional steps to account for changes in scope;
- To eliminate unnecessary or unneeded steps;
- To adjust or account for site specific conditions as a result of the initial field efforts; and/or
- To alter text formatting relative to the physical SOP document.

In general, SOPs provide the following information:

- Overview and Application (also introduced as Background and Scope)
- Sampling Methods and Procedures
- Decontamination Procedures
- Sample Containers and Preservation Techniques
- Quality Control

- Documentation
- Sample Shipment
- Additional Contact Information
- References (where applicable)

The remainder of this section presents a brief discussion of the major SOP subsections noted above.

2.1 Overview and Application (also Background and Scope)

Each SOP provided a brief introduction on the purpose of the task at hand. A discussion of the procedures to be implemented and the overall purpose of the task(s) was provided in this section.

2.2 Sampling Methods and Procedures

Each SOP provided a description of the sampling methods (if applicable) and procedures to be followed during the implementation of the task. In general, this portion of the SOP presented the following information:

- Description of the sampling locations
- Types of samples to be collected
- Sample equipment and general supplies
- Sample nomenclature
- Sample equipment installation (if appropriate)
- Detailed sampling procedures
- Description of field processing requirements (filtering, mixing, etc.)

The primary purpose of a SOP is to provide detailed information on the sampling procedures so that data are collected consistently. The bulk of the SOP text focused on this task.

2.3 Decontamination Procedures

Decontamination procedures are an important aspect of the SOPs associated with the collection of samples to be submitted for analytical parameters. Decontamination procedures were designed to reduce the number of variables which could impact data evaluation and interpretation. They helped limit the impact of potential cross-contamination between sampling locations and sampled properties. They could also be used as biosecurity protocols to limit the potential of disease transmittal between

growers' farms. Decontamination requirements were developed based upon the planned use of the data, the degree of impact upon laboratory reporting limits, and the biosecurity protocol requirements.

In general, decontamination efforts to reduce cross-contamination impacts during this program only required rinsing/scrubbing/wiping sampling equipment with distilled or deionized water, followed by a rinse/scrub/wipe by phosphate free detergent mix, followed by another rinse/wipe with distilled or deionized water. Additionally, sample devices associated with water sampling were typically rinsed with the water at the sample location prior to collection of the samples. For contaminants of concern, the decontamination procedures were proved sufficient as evidenced by the results of the rinsate blanks collected during the program.

The above level of decontamination was appropriate for the sampling programs not required to meet biosecurity protocols. The decontamination effort associated with the soil and litter sampling program required additional effort to meet the biosecurity protocols. Decontamination associated with biosecurity protocol generally employed the above procedures with the added step of a rinse or wipe with a ten percent bleach solution before the second distilled/deionized water rinse. A more detailed description of the decontamination procedures implemented for the soil and litter sampling program is provided in Section 4.7.

2.4 Sample Container and Preservation

SOPs related to the collection of analytical data presented a discussion of the sample containers required for the program. Where appropriate, sample preservation requirements were also provided.

2.5 Quality Control

SOPs provided descriptions of the quality control measures to be implemented during the program. Where appropriate, the SOPs provided descriptions of field quality control (QC) samples. QC samples included blind standards, splits, duplicates, decontamination rinsate blanks, and field analyses.

Blind standards consist of prepared samples of known composition that have undergone independent multi-laboratory analysis using standard methods. Blind standards, also known as performance evaluation samples, are submitted to a laboratory to evaluate the laboratory's test results. Blind standards are submitted to independently evaluate accuracy of laboratory results.

Splits are co-located samples collected identically and consecutively and within a minimum time frame. Grab samples are typically split samples. Duplicates are samples collected from the same composite samples. Splits and duplicates are submitted to laboratories to provide a measure of the system variability (field and laboratory) including the variability component resulting from the inherent heterogeneity of the field sources.

Decontamination rinsate blanks are collected to evaluate the effectiveness of the decontamination process.

A more detailed discussion of the QC results for this sampling program is provided in Roger Olsen's Expert Witness Report.

2.6 Documentation

SOPs provided descriptions of the documentation efforts to be conducted. Examples of documentation efforts include the following:

- Decision-making processes in the field
- Field notebooks
- Photographic records
- Video records
- Field work sheets

Field books and work sheets are used to document aspects of sample collection and handling as well as visual observations. Documentation was also provided in the chain-of-custody documents completed during the field investigation. Chain-of-custody documents are discussed in more detail in Section 4.17 (SOP 9-1)

2.7 Sample Shipment

Where applicable, SOPs provided information related to sample shipment including shipping requirements and the addresses of the laboratories receiving the sample shipments.

2.8 Additional Contact Information

When appropriate, SOPs provided additional contact information. The contact information was provided for field staff in the event that questions arise during the field program.

2.9 References

Where applicable, SOPs may provide references to other documents. The referenced documents were either used as a source to develop the SOP or the SOP was modeled after the referenced document.

3.0 SOP Training, Supervision and Chain-of-Command

This section discusses the training, supervision, and chain-of-command structure implemented for the primary activities conducted during this investigation. Training activities included the training of CDM staff, Lithochimeia staff, and subcontractor

staff such as Environmental Priority Service who conducted groundwater sampling by direct push methods.

3.1 Training

Training of field staff was accomplished through a number of different procedures. The following training support was provided to the field staff:

- Biosecurity Training by the Oklahoma Department of Agriculture, Food and Forestry (ODAFF) in Oklahoma City
- Pre-field deployment training sessions by experienced CDM staff
- Practice sessions conducted by experienced CDM staff
- Direct supervision during field work by experienced CDM staff
- In-the-field review sessions conducted by CDM task leaders or the project manager
- Health and Safety review sessions conducted by experienced CDM staff

3.1.1 Biosecurity Training

All staff involved with direct sample collection from poultry grower properties were provided biosecurity training by ODAFF staff in Oklahoma City. This training was focused on preventing the transmission of poultry disease from one farm to another, or from one poultry house to the next. Decontamination efforts relative to the above goal were reviewed in detail. The biosecurity decontamination efforts were incorporated into SOP 5-1.

3.1.2 Pre-field deployment training sessions

Pre-field deployment training sessions were conducted for the large scale (requiring a number of different staff) sampling programs. Pre-field deployment training sessions were conducted for programs such as:

- The 2005 Lake Event sampling
- The 2005 Sediment sampling
- The 2005, 2006, and 2007 Biologic sampling events
- The 2006 and 2007 River sampling events
- The 2006 Soil and Litter Sampling events
- The 2006 Spring Sampling events
- The 2007 Synoptic Sampling event

The pre-field deployment sessions were conducted by experienced CDM or Lithochimeia staff and included review of the program objectives, discussion of the sampling procedures, review of health and safety issues where appropriate, and sample handling, and equipment usage and calibration where applicable.

3.1.3 Practice Training Sessions

Practice field exercises were also conducted for several of the large scale sampling programs. The exercises were conducted to refine the sampling process and to help familiarize the field staff with the sampling protocols. Practice field exercises were conducted for the following sampling programs:

- 2006 Soil sampling program
- 2007 River sampling
- 2007 Fish survey

3.1.4 Direct Supervision by Experienced Field Staff

In-field training was conducted for all field sampling programs. In-field training consisted of experienced personnel, primarily CDM staff, training field sampling crews. In-field training included proper equipment calibration and set-up, sample collection, and sample documentation. Staff members were supervised until they demonstrated proficiency in the sampling program. Training supervisors achieved their experience from other field sampling programs or during previous sampling efforts under other training supervisors.

3.1.5 In-the-field Review Sessions

CDM task leaders or the project manager conducted periodic in-the-field review sessions during extended sampling programs. The in-the-field review sessions were generally conducted at the beginning of the day and included a review of the day's planned sampling effort, sampling goals, and any questions or concerns by sampling team members. Field notes and other documentation were also reviewed for appropriateness and completeness.

3.1.6 Health and Safety Sessions

Training efforts also included Health and Safety sessions. These sessions were conducted by Task Leaders or the Project Manager at the beginning of each sampling program. Health and Safety concerns and procedures were reviewed. Review sessions were conducted at the discretion of the task leader.

3.2 Supervision and Chain-of-Command

CDM and Lithochimeia field staff were supervised in the field by personnel experienced in the sample protocols for the various field programs. Sampling experience was achieved through previous industry experience outside of this

program or through direct supervision by other experienced staff during previous sampling effort under the same sampling program.

The chain-of-command consisted of field teams working under the direct supervision of a field team leader. Field team leaders were supervised by task leaders. Depending upon the scale of the sampling program, the task leader and field team leader may have been the same person. On larger scale sampling efforts, the task leader may not have been directly supervising the field team(s). Task leaders were responsible for the conduct of their portion of the sampling program and reported to the project manager. The task leaders, in conjunction with the project manager also had the ability to review and or change the field sampling protocols after consultation with either the senior CDM supervisor, or the outside expert witnesses for whom the data were being collected. Changes or deviations to the SOPs were to be documented by the field teams in their field books if the changes or deviations occurred during the actual sampling efforts. SOPs were revised under the supervision of the project manager or senior CDM supervisor associated with the SOP to be revised.

4.0 Sampling Program SOPs

This section discusses the primary activities conducted during this investigation and the applicable SOPs. A total of 22 SOPs were developed for this program. The SOPs are provided in Appendix A. Each of the SOPs used during this program are presented below.

4.1 SOP 1-1 Tenkiller Ferry Reservoir Sampling

SOP 1-1 applies not only toward Lake Tenkiller sampling, but was also used in the sampling of Stockton Lake and Broken Bow Lake. This SOP describes field procedures used for collection of surface water samples from Lake Tenkiller in the Illinois River Watershed of eastern Oklahoma and western Arkansas. This SOP includes water chemistry, zooplankton, phytoplankton, bacteria, and other water sampling protocols. Procedures for on site measurement of various water quality parameters such as temperature, conductivity, turbidity, pH, oxidation-reduction potentials, and dissolved oxygen are also provided in this SOP. This SOP follows the guidelines described in the Standard Operating Procedures (SOP) for Field Sampling Efforts of the Oklahoma Water Resources Board's 2001 Beneficial Use Monitoring Program and also as updated in 2004.

The purpose of the work was to evaluate and document 1) the linkage and relationship, if any, between the disposal of poultry wastes and environmental contamination within Lake Tenkiller, and; 2) the resulting harm/injury to natural resources that may have resulted from the disposal of poultry wastes within the Illinois River Watershed.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated the collection of reliable data under repeatable collection procedures. Based upon my review of the field books and work sheets

associated with this sampling program along with my personal field observations and the data results, it appears that the SOP was correctly implemented. As noted in Roger Olsen's Expert Witness Report, the data can be used for all of its intended purposes. The data were used in the impact assessment of the lake and its fauna.

4.2 SOP 1-2 Public Water Sampling

This SOP describes field procedures used for the collection of public drinking water samples from water supply distribution system in the Illinois River Watershed for analysis of trihalomethanes (THM) and haloacetic acids (HAA). The SOP also includes procedures used for collection of surface water samples at the raw water intake locations for the water supply systems. THM and HAA are a group of chemicals that are produced by a reaction between inorganic and organic compounds in water (e.g., algae and organic matter) with chorine used for disinfection to control microbial contaminants. The US EPA regulates the amount of THM and HAA that can be found in drinking water because of the potential risk of increased cancer rates associated with exposure to these chemicals. The amount of THM and HAA found in a water supply is dependent on certain characteristics of the source water and of the disinfection process as described in the Oklahoma Department of Environmental Quality (DEQ) 2005 document titled: Water: Trihalomethane (THM) Fact Sheet.

Samples were also collected in order to assess the THM formation potential, and the actual presence and concentrations of THM and HAA with a water system at Gore PWA, Cherokee RWD #2, and Tahlequah water systems.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated the collection of reliable data under repeatable collection procedures. Based upon my review of the field books associated with this sampling program along with the data results, the SOP appears to have been correctly implemented. As noted in Roger Olsen's Expert Witness Report, the data can be used for all of its intended purposes. The data were used in the impact assessment of the local drinking water systems.

4.3 SOP 2-1 Automated High Flow Sampling

This SOP presents instructions for collecting representative high flow samples from streams during runoff events using Teledyne Isco, Inc. automated samplers. This SOP describes in detail the protocol for sampling high flow events within the Illinois River Watershed.

The purpose of the SOP was to facilitate the consistent collection of data that would allow the direct measurement of flow-weighted concentrations of key pollutants of concern associated with runoff events in small watershed tributaries. The SOP also allows the investigation of potential correlations between the runoff concentrations and land use characteristics, poultry operations, and storm event hydrographs characteristics.

The SOP recommends the equipment to be used and outlines the basis for the selection of site sampling locations. The SOP also provides information tips and troubleshooting guidance based upon past sampling experiences with the specified equipment. This guidance system was very useful to field staff trying to solve encountered programming and sample collection issues in the field.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated the collection of reliable data under repeatable collection procedures. Based upon my review of the field books associated with this sampling program along with the data results, the SOP appears to have been correctly implemented. As noted in Roger Olsen's Expert Witness Report, the data can be used for all of its intended purposes. The data were used by CDM as well as outside expert witnesses in the evaluation of potential impacts to the surface waters from storm and baseflow events.

4.4 SOP 2-2 High Flow Sample Compositing and Filtering

This SOP provides compositing and handling procedures to be followed by the CDM Denver Laboratory when preparing high flow samples for shipment to the analytical laboratories. Compositing is the process of combining sample portions from various sample bottles into a uniform sample.

Data downloaded from the Teledyne Isco, Inc. sampler via Rapid Transfer Device (RTD) was evaluated by a CDM specialist. The evaluation consisted of using the data provided by the RTD which includes depth and velocity at the specific flow station as well as the cross-sectional characteristics of the stream channel at the sampler location. The specialist used to data to develop a compositing scheme in spreadsheet format which showed the volumes to be used from each bottle to generate a composite sample representative of the high flow event.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated consistent sample handling and compositing at the CDM Denver laboratory in order to prepare samples for analysis. Based upon my discussion with the laboratory manager and the data results, it appears that the SOP was correctly implemented. As noted in Roger Olsen's Expert Witness Report, the data can be used for all of its intended purposes. The data were used by CDM as well as outside expert witnesses in the evaluation of potential impacts to the surface waters from storm and baseflow events.

4.5 SOP 3-1 Spring Sampling

Instructions presented in this SOP are for collecting representative water samples from flowing spring water bodies. A spring is defined as a place where the water table crops out at the ground surface and water flows out more or less continuously. This SOP describes in detail the spring sampling for the Illinois River Watershed sampling.

During the 2005 sampling program, over 50 springs or seeps were identified in the Illinois River Watershed from USGS maps and University studies and reports. Access was gained to eighteen springs which were sampled in the spring of 2005 and eight of the springs were resampled in the late fall of 2005. Additional springs were identified during 2006 and 2007 field reconnaissance activities. Twenty-nine springs were sampled in 2006 with fourteen of the springs sampled in 2005 resampled in 2006. Two springs were sampled in 2007. Springs typically result from groundwater seepage or outflow at specific locations during periods of high rainfall. During other times of the year, the groundwater may enter streams as base flow (gaining stream).

The purpose of the sampling program was to collect reliable and repeatable data to evaluate the degree of groundwater contamination present in selected areas by sampling springs. The data were used to provide a preliminary evaluation of potential groundwater contamination.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated the collection of reliable data under repeatable collection procedures. Based upon my review of the field books associated with this sampling program, photographic review, my field observations of some of the sampling events, and the data results, the SOP appears to have been correctly implemented. As noted in Roger Olsen's Expert Witness Report, the data can be used for all of its intended purposes. The data were used in the evaluation of impacts to the springs within the watershed.

4.6 SOP 4-1 Sediment Sampling in Streams and Small Impoundments

Instructions presented in this SOP are for collecting representative sediment samples from surface water bodies. Sediment can be considered as solid material that is submerged/saturated (at least temporarily) or suspended in any surface water body. This includes sludges, lake bottom sediments, perennial and intermittent stream sediments, and marine sediments.

The purpose of the work was to provide data to allow the evaluation and documentation of 1) the linkage and relationship, if any, between the disposal of poultry wastes and environmental (primarily aquatic system) contamination within the Illinois River Watershed, and; 2) the resulting harm/injury to natural resources that may have resulted from the disposal of poultry wastes within the Illinois River Watershed.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated the collection of reliable data under repeatable collection procedures. Based upon my review of the field books associated with this sampling program along with the data results, the SOP appears to have been correctly implemented. As noted in Roger Olsen's Expert Witness Report, the data can be used for all of its intended purposes. The data were used in the evaluation of impacts to the sediments within the watershed.

4.7 SOP 5-1 Litter and Soil Sampling

This SOP provides guidance for the collection of poultry waste (litter) from active poultry houses and soil samples from pastures and control field locations within the Illinois River Watershed. Sample locations were selected from either contract growers' farms or company-owned facilities. Fields where documentation of litter application from a specific farm and Integrator was available from the ODAFF were selected for sample collection. Fields associated with a contract grower farm or company-owned facilities were identified with a LAL (Litter Applied Location) prefix. In addition, control soil samples were collected from pastures where no documented poultry litter application occurred and from forested property. These samples are identified with a CL (Control Location) prefix.

The purpose of this SOP was to provide a consistent protocol for the collection of soil and litter samples. The purpose of the work was to provide data to allow the evaluation and documentation of 1) the linkage and relationship, if any, between the disposal of poultry wastes and environmental contamination within the Illinois River Watershed, and; 2) the resulting harm/injury to natural resources that may have resulted from the disposal of poultry wastes within the Illinois River Watershed.

This SOP includes detailed procedures on how to establish the grid pattern (also referred to as a subarea within a grower's property) for the soil sampling program[1]. It also provides instructions on the collection of soil litter samples from the poultry houses. Decontamination procedures are established and differ depending upon the type of collection being conducted. The most stringent decontamination procedures are the result of biosecurity protocols required by the ODAFF and the poultry industry. Biosecurity protocols are focused on the prevention of transfer of poultry viruses and harmful bacteria between individual growers' properties. Biosecurity protocols are not necessarily intended to address sampling and cross-contamination issues, but it can be an unintended side benefit when applied.

Decontamination processes employed for soil sampling on litter applied pastures incorporated the requirements of the biosecurity protocols, the use of the data, and the laboratory reporting limits. Decontamination protocols were set forth in SOP 5-1. Full decontamination procedures were consistent with the biosecurity requirements and were employed when sampling teams left an individual grower's property and

---

[1] I have reviewed the affidavit/report titled "Report of Sampling Oversight Observations" prepared by Jay Churchill ("Mr. Churchill") on behalf of the Defendants in connection with the Preliminary Injunction Hearing. I have also read Mr. Churchill's testimony as a witness for Defendants during the hearing on the State's Motion for Preliminary Injunction. Mr. Churchill identifies a document titled "Soil and Litter/Manure Sampling Protocol" and refers to it as a "Work Plan" in his report and testimony. However, Mr. Churchill fails to specify which "Soil and Litter/Manure Sampling Protocol" he is referring to. Based on the text citations, it appears that Mr. Churchill relied on either Revision 6 (May 11, 2005) or Revision 7 (April 24, 2006). Also, CDM has never characterized the referenced documents as "Work Plans." CDM characterized these documents as SOPs.

entered onto public right-of-ways. A more detailed discussion of the decontamination protocols in SOP 5-1 are provided below.

SOP 5-1 describes two levels of decontamination protocols.[2] Full decontamination procedures of sampling equipment involved the removal of organic and soil debris by scrubbing and using a phosphate free detergent rinse. This was followed by a rinse with a ten percent bleach solution and then a deionized or distilled water rinse. The implementation of a ten percent bleach solution rinse was primarily to meet biosecurity protocols.

The second level of decontamination protocols in SOP 5-1 involves decontamination between subareas within a LAL where sampling crews did not exit onto public right-of-ways before entering the next subarea within an LAL. In these instances, full decontamination procedures were not required. The second level of decontamination consisted of removing soil material from the corer barrel and the knife or implement used to cut the soil samples prior to collection of the first soil sample from the next LAL subarea. In addition, the corer was driven into the soil at the first grid location of the following subarea and the material removed and discarded prior to commencing the grid sampling program. This was done to further reduce the possibility of cross-contamination between LAL subareas.

Within a subarea, the SOP did not require decontamination between soil samples within a single core sample, between cores within a grid location, or between grid locations. All zero to two-inch sample intervals, two to four-inch sample intervals, and four to six-inch sample intervals within a subarea were composited at the CDM laboratory before being submitted for analytical testing. Decontamination efforts were not specified for the above situations for the following reasons:

- Cross-contamination between sample depths within a core sample from material remaining on the cutting implement and/or on sample gloves was evaluated by CDM to be insignificant by weight when compositing processes are considered.

---

[2] I have reviewed the affidavit/report titled "Report of Sampling Oversight Observations" prepared by Jay Churchill ("Mr. Churchill") on behalf of the Defendants in connection with the Preliminary Injunction Hearing. I have also read Mr. Churchill's testimony as a witness for the Defendants during the hearing on the State's Motion for Preliminary Injunction. As a general matter, Mr. Churchill's criticisms of CDM's sampling program are without merit. For example, Mr. Churchill's assertion that CDM violated Section III.A.4.b and Section III.A.6 of the Soil and Litter SOP is false. First, Version 9 of the Soil and Litter SOP does not even have Sections identified as III.A.4.b or III.A.6. Second, neither Revision 7, 8, or 9 of SOP 5-1 requires that decontamination be performed between discrete in-core soil samples or between soil samples within a sampling area grid. One of the primary differences between Revision 7 and Revision 9 of SOP 5-1 was the modification to the decontamination section (Section 4.6). The SOP was modified to clearly state that full decontamination procedures were only required when equipment and staff left a grower's property and/or passed on to a public right-of-way. Revision 7 indicated that full decontamination procedures were required between subareas.

- Cross-contamination between core locations by material potentially remaining within the core barrel, on the cutting implement, and/or on sample gloves was evaluated by CDM to be insignificant by weight when compositing processes are considered.

Poultry litter sampling devices were one time use materials. Shovels, trowels, and five-gallon buckets used in the collection of samples were either donated to the grower or discarded as trash. No decontamination of sampling tools occurred. Sampling personnel were decontaminated per biosecurity protocols to prevent the possible transfer of poultry viruses from one grower to another. Biosecurity decontamination protocols required the disposal of all personnel protective equipment and the rinses of rubber boots by a phosphate free detergent solution; ten percent bleach solution; and deionized or distilled water.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated the collection of reliable data under repeatable collection procedures. Based upon my review of the field books associated with this sampling program, photographic review, my field observations of some of the sampling events, and the data results, the SOP appears to have been correctly implemented. As noted in Roger Olsen's Expert Witness Report, the data can be used for all of its intended purposes. The data were used in the assessment of poultry waste characteristics and evaluation of impacts to the soils within the watershed.

4.8 SOP 5-2 Litter and Soil Sample Compositing

This SOP describes procedures used for compositing of soil and poultry litter samples from the Illinois River watershed of eastern Oklahoma and western Arkansas. As with water samples, soil compositing requires mixing of a select number of collected soil samples to generate one representative soil sample. Two types of soil compositing were performed during the litter sampling effort.

The first type of litter and soil compositing consisted of a rough compositing of the collected litter sample as described under SOP 5-1, Version 9, Section 5.2.4.c. This was done to generate a litter sample to be submitted directly to the laboratory for bacterial analyses. This rough compositing effort was dictated by the need to get the bacteria samples to the laboratory as soon as possible to provide a representative result of the bacteria population in the litter at the time of sampling. The time delay involved with sending the sample to the CDM Denver Laboratory, plus the additional handling efforts, would not have provided as representative a result of actual bacteria conditions in the poultry house at the time of sampling. It should be noted that previous versions of SOP 5-1 stated that compositing of the litter sample would not occur in the field. The laboratory requirements for bacterial analyses, dictated a modification to this portion of SOP 5-1.

A thorough compositing effort was conducted under SOP 5-2 on the soils and the litter samples shipped to the CDM Denver Laboratory to generate representative samples. The resulting composited sample was designed to provide a sample

representative of the entire sampled subarea. The composited soil sample was also split between CDM's chosen laboratories and the Defendant's laboratories. The compositing of the soil and litter samples was performed at the CDM Denver Laboratory. The SOP includes handling, processing, and shipping protocols relative to the soil and litter samples.

The purpose of the SOP was to provide soil and litter samples representative of the sampled subareas and poultry houses. The goal was also to provide a homogeneous soil sample that could be split with the Defendant per their requirements.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated consistent sample handling and compositing at the CDM Denver laboratory in order to prepare samples for analysis. Based upon my discussion with the laboratory manager and the data results, it appears that the SOP was correctly implemented. As noted in Roger Olsen's Expert Witness Report, the data can be used for all of its intended purposes. The data were used in the assessment of poultry waste characteristics and evaluation of impacts to the soils within the watershed.

4.9 SOP 5-3 Manure Sampling for DNA Analysis

This SOP describes field procedures used for collection of fecal matter for identifying the types and abundance of bacterial DNA. The bacterial DNA is first amplified by polymerase chain reaction (PCR), and then digested with a restriction enzyme. The enzyme cuts DNA strands into different size fragments whose length is dependent upon the DNA sequence, and the last (terminal) fragment is labeled for detection. Each terminal fragment length is represents approximately one bacterial species. This program was designed to identify DNA fragments from bacteria that reside in fecal material from various animals, including cattle, swine, ducks, geese and humans.

The SOP provides collection and handling guidelines, decontamination procedures, and shipping requirements. The purpose of this program was to provide data which would determine whether unique bacteria DNA strands were associated with poultry waste but not the other types of fecal material sampled within the watershed. The results of the sampling may help assess the degree of impact of poultry litter within the watershed.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated the collection of reliable data under repeatable collection procedures. Based upon my review of the field books associated with this sampling program along with the data results, it appears that the SOP was correctly implemented. The data are being used for its intended purposes.

4.10 SOP 6-1 Water Sampling of Rivers and Streams

This SOP describes field procedures used for collection of surface water samples from rivers and streams in the Illinois River watershed of eastern Oklahoma and western

Arkansas. This includes water chemistry, nutrients, bacteria, and other water sampling protocols. Procedures for on site measurement of various water quality parameters such as temperature, conductivity, turbidity, pH, and dissolved oxygen are also discussed. This follows the guidelines described in the Standard Operating Procedures (SOP) for Field Sampling Efforts of the Oklahoma Water Resources Board's 2001 Beneficial Use Monitoring Program (updated in 2004).

The purpose of the work was to provide representative and repeatable data to allow the evaluation and documentation of 1) the linkage and relationship, if any, between the disposal of poultry wastes and environmental contamination within the Illinois River Watershed, and; 2) the resulting harm/injury to natural resources that may have resulted from the disposal of poultry wastes within the Illinois River Watershed.

Sampling conducted under this SOP includes baseflow sampling, water quality sampling conducted in conjunction with the biologic assessments, grab samples during high flow events, and periodic scheduled water quality monitoring.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated the collection of reliable data under repeatable collection procedures. Based upon my review of the field books associated with this sampling program along with the data results, the SOP appears to have been correctly implemented. As noted in Roger Olsen's Expert Witness Report, the data can be used for all of its intended purposes. The data were used in the evaluation of impacts to the aquatic systems within the watershed.

The SOP is provided in Appendix A. Review of the field books associated with this sampling program indicates that the SOP was appropriate and resulted in repeatable collection procedures and reliable data in the evaluation of impacts to the aquatic systems within the watershed. As noted in Roger Olsen's Expert Witness Report, the data can be used for all of its intended purposes.

4.11 SOP 7-1 and SOP 7-1.1 Fish Community Sampling

These SOPs describe field procedures used for collection of resident fish from rivers, lakes, and streams. SOP 7-1 was used for the 2005 field collection of samples by electrofishing gear and by seining and was based upon the USEPA Rapid Bio-assessment Protocols (RBP) for use in wadeable streams and rivers (Barbour, 1999). SOP 7-1.1 was modified from SOP 7-1 in 2007 after CDM staff collaborated with staff from the Oklahoma Department of Wildlife Conversation to update the 2007 field sampling program.

The purpose of the work was to provide representative data to allow the evaluation and documentation of 1) the linkage and relationship, if any, between the disposal of poultry wastes and environmental contamination within the Illinois River Watershed, and; 2) the resulting harm/injury to resident fish populations that may have resulted from the disposal of poultry wastes within the Illinois River Watershed.

The SOPs are provided in Appendix A. The SOPs are good guidance documents for the work conducted. The SOPs facilitated the collection of reliable data under repeatable collection procedures. Based upon my review of the field books and work sheets associated with this sampling program along with the data results, the SOPs appear to have been correctly implemented. The data are being used for their intended purposes. The data were used in the evaluation of impacts to the fish communities within the watershed.

4.12 SOP 7-2 Periphyton Sampling

This SOP describes field procedures used for the 2005 collection of benthic algae (periphyton) from streams in the Illinois River watershed in eastern Oklahoma and western Arkansas. Periphyton are a vital part of many stream communities and are often used as indicators of water quality conditions. By comparing the composition, density, and growth rates of benthic algal assemblages between affected and reference streams at similar times of year, a valuable assessment of the environmental impact of various form of pollution can be formulated. This SOP follows the standard laboratory based approach for periphyton sampling as outlined in the USEP A's Rapid Bioassessment Protocols (RBP) for Use in Wadeable Stream and Rivers (Barbour et aI., 1999).

The purpose of the work was to provide representative data to allow the evaluation and documentation of 1) the links and relationship, if any, between the poultry waste disposal and environmental contamination with the Illinois River Watershed, and; 2) the resulting harm/ injury to resident periphyton communities that may have resulted from the disposal of poultry wastes with the Illinois River Watershed.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated the collection of reliable data under repeatable collection procedures. Based upon my review of the field books associated with this sampling program along with the data results, the SOP appears to have been correctly implemented. The data are being used for their intended purposes. The data were used in the evaluation of impacts to the periphyton communities within the watershed.

4.13 SOP 7-3 Benthic Macroinvertebrate Sampling

This SOP describes field procedures used for the 2005 collection of benthic macroinvertebrates from streams in the Illinois River watershed in eastern Oklahoma and western Arkansas. Benthic macroinvertebrates are found in most flowing water bodies and are often used as indicators of water quality conditions. By comparing the composition and density of macroinvertebrate populations between affected and reference streams at similar ties of year, a valuable assessment of the environmental impact of various forms of pollution can be formulated. This SOP was used for collecting samples using fine-meshed dip nets and benthic seines (kick nets) and followed the Oklahoma Water Resources Board's Beneficial Use Monitoring Program (BUMP) protocols. Procedures for collecting samples using standard Hester-Dendy

samplers are also included. A Hester-Dendy sampler is a specialized device designed to present an artificial substrate for benthic macroinvertebrates to attach to. The device facilitates the assessment of a benthic macroinvertebrate community.

The purpose of the work was to provide data to allow the evaluation and documentation of 1) the links and relationship, if any, between the disposal of poultry wastes and environmental contamination within the Illinois River Watershed, and; 2) the resulting harm/ injury to resident macroinvertebrate populations that may have resulted from the disposal of poultry wastes within the Illinois River Watershed.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated the collection of reliable data under repeatable collection procedures. Based upon my review of the field books associated with this sampling program along with the data results, the SOP appears to have been correctly implemented. The data are being used for their intended purposes. The data were used in the evaluation of impacts to the benthic macroinvertebrate communities within the watershed.

4.14 SOP 7-4 Stream Habitat Assessment

This SOP describes field procedures used for the 2005 collection of stream habitat data in the Illinois River watershed of eastern Oklahoma and western Arkansas. In this protocol, the term "habitat' refers to the characteristics of the in-stream and riparian habitat that may influence the biotic community of a stream. This includes the physical characteristics such as stream width, flow, depth, and substrate. Procedures for onsite measurement of various water quality parameters such as temperature, conductivity, and dissolved oxygen are also discussed. This SOP follows the standards outlined in the US EPA's Rapid Bioassessment Protocols (RBP) for Use in Wadeable Streams and Rivers (Barbour et aI., 1999).

The purpose of the work was to provide data to allow the evaluation and documentation of 1) the links and relationship, if any, between the disposal of poultry wastes and environmental contamination within the Illinois River Watershed, and; 2) the resulting harm/ injury to resident biotic communities that may have resulted from the disposal of poultry wastes with the Illinois River Watershed.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated the collection of reliable data under repeatable collection procedures. Based upon my review of the field books and worksheets associated with this sampling program along with the data results, the SOP appears to have been correctly implemented. The data are being used for their intended purposes. The data were used in the evaluation of impacts to the aquatic systems within the watershed.

4.15 SOP 7-5 Combined River and Biological Sampling Program

This SOP describes the field procedures used for sampling during the 2006 and 2007 River and Biological sampling program. The purpose of these biological investigations was to determine the level of biological productivity (eutrophication) associated with various water quality parameters, primarily; phosphorus, nitrogen, and dissolved oxygen. In 2006, a two-phased approach involved preliminary site investigations of over 200 stream locations followed by more intensive water quality and biological community assessments of approximately 70 sites within and around the Illinois River watershed. Most of these 70 locations were reinvestigated in 2007.

In the first phase, sampling teams were sent to select locations throughout the watershed to collect field water quality measurements including pH, Dissolved Oxygen (DO), water temperature, conductivity, turbidity, and water samples were collected for analysis of phosphorus concentrations. During the second phase, a subset of the original locations was selected for additional surface water sampling along with benthic and sestonic chlorophyll analysis, macroinvertebrate community sampling, algal community sampling, and habitat evaluations. This SOP was developed so that consistent collection and sampling procedures were followed. This SOP was based in part upon the USEPA's Rapid Bioassessment Protocols (RBP) for Use in Wadeable Streams and Rivers (Barbour et al., 1999). Modifications to these protocols were made based upon direct consultation with Dr. R. Jan Stevenson of Michigan State University. Dr. R. Jan Stevenson was acknowledged as a key contributor to the periphyton chapter (Chapter 6) of the above document. Dr. Stevenson's modifications were primarily to provide focused and streamlined sampling protocols applicable to the proposed sampling program.

The purpose of the work was to provide data to allow the evaluation and documentation of 1) the linkage and relationship, if any, between the disposal of poultry wastes and environmental contamination within the Illinois River Watershed, and; 2) the resulting harm/injury to natural aquatic systems that may have resulted from the disposal of poultry wastes within the Illinois River Watershed.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated the collection of reliable data under repeatable collection procedures. Based upon my review of the field books and worksheets associated with this sampling program along with the data results, the SOP appears to have been correctly implemented. The data are being used for their intended purposes. The data were used in the evaluation of impacts to the aquatic communities and systems within the watershed.

4.16 SOP 8-1 Water Quality Meters

This SOP describes field procedures for using an electronic water quality meter to conduct on-site measurements of surface waters. This includes calibration and maintenance procedures. Specific procedures for on-site measurement of various water quality parameters at various sample locations provided in other SOPs. This

SOP follows the guidelines described in the E*nvironmental Investigations Standard Operating Procedures and Quality Assurance Manual.* (USEPA, 1996).

The procedures in this SOP are designed for use with YSI-650 multi-meters. These procedures are generally applicable for most types and models of electronic water quality measurement devices. When other water quality meters were used, experienced staff were used to train other staff in the use of the equipment. In addition, the operation manual for the instrument provided an additional level of support for maintenance or calibration procedures to be used. As noted in Section 3, field and laboratory staff were trained by experienced personnel in the calibration and use of water quality meters.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP was appropriate to provide repeatable measurement procedures and reliable data during this program. Based upon my review of the field books associated with this sampling program along with the data results, the SOP appears to have been correctly implemented. As noted in Roger Olsen's Expert Witness Report, the data can be used for all of its intended purposes. The data were used to evaluate water quality aspects of surface water, springs, and groundwater systems sampled during the program.

4.17 SOP 9-1 Shipping and Chain of Custody

This SOP describes procedures for shipping and maintenance of chain of custody (COC) documents for all samples collected during the course of the project. Chain of custody documents provide the sample identification, collection date, and times. They specify the analyses to be performed and document the custody of the samples between collection and arrival at the samples at the laboratory. Proper packing and documentation helps keep collected samples viable and in suitable condition for the laboratory contracted to do the analyses. The SOP also provides the contact and shipping information for the laboratories used during this program.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated consistent sample handling, shipping, and shipping documentation under repeatable procedures. Based upon my review of the chain of custody documents and field book notes associated with this sampling program along with the data results, the SOP appears to have been correctly implemented. As noted in Roger Olsen's Expert Witness Report, the data can be used for all of its intended purposes.

4.18 SOP 10-1 Edge of Field Sampling

This SOP is applicable to the collection of representative aqueous samples of runoff from fields ("Edge of Field Samples" or "EOF Samples") and includes samples collected directly from naturally ponded or flowing water and from passive samplers installed to collect water runoff from fields. Passive samplers were sample collection containers placed in areas where runoff from applied fields was likely to flow during

rainfall events. After the rainfall event, the sampler was removed and the collected water transferred to an appropriate container for sample shipment.

The purpose of the work was to provide data to allow the evaluation and documentation of 1) the linkage and relationship, if any, between the disposal of poultry wastes and environmental contamination within the Illinois River Watershed, and; 2) the resulting harm/injury to natural systems that may have resulted from the disposal of poultry wastes within the Illinois River Watershed.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated the collection of reliable data under repeatable collection procedures. Based upon my review of the field books and work sheets associated with this sampling program along with the data results, the SOP appears to have been correctly implemented. As noted in Roger Olsen's Expert Witness Report, the data can be used for all of its intended purposes. The data were used in the evaluation of impacts to the surface waters within the watershed.

4.19 SOP 11-1 Residential Well Sampling

This SOP describes field procedures used for collection of groundwater samples from the Illinois River watershed (IRW) of eastern Oklahoma. Groundwater samples were collected from existing residential wells within the Oklahoma portion of the IRW. Locations within various potentially impacted areas and areas suspected to have minimal impact were selected for sampling. Procedures for on site measurement of various water quality parameters such as temperature, conductivity, turbidity, pH, oxidation-reduction potential, and dissolved oxygen were provided.

The purpose of the sampling program was to collect usable data to assess the potential impact from the application of poultry house waste to fields within the watershed. The data were used to provide a preliminary assessment of the extent and degree of potential groundwater impact.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated the collection of reliable data under repeatable collection procedures. Based upon my review of the field books associated with this sampling program, my field observations of some of the sampling events, and the data results, the SOP appears to have been correctly implemented. As noted in Roger Olsen's Expert Witness Report, the data can be used for all of its intended purposes. The data were used in the evaluation of impacts to the groundwater in the watershed.

4.20 SOP 11-2 Direct Push Groundwater Sampling

This SOP describes field procedures used for collection of groundwater samples by direct push sampling methods from the Illinois River Watershed of eastern Oklahoma. Locations anticipated to have saturated unconsolidated sediments within various impacted areas were selected for sampling.

The purpose of this work was to provide data to allow the evaluation and documentation of 1) the linkage and relationship, if any, between the disposal of poultry wastes and environmental contamination within the Illinois River Watershed, and; 2) the resulting harm/injury to natural systems that may have resulted from the disposal of poultry wastes within the Illinois River Watershed.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated the collection of reliable data under repeatable collection procedures. Based upon my review of the field books associated with this sampling program, my field observations of some of the sampling events, and the data results, the SOP appears to have been correctly implemented. along with the data results, it appears that the SOP was correctly implemented. As noted in Roger Olsen's Expert Witness Report, the data can be used for all of its intended purposes. The data were used in the evaluation of impacts to the groundwater within the watershed.

4.21 SOP 12-1 General Water Preparation and Filtering - EOF, Springs, and Groundwater

Instructions presented in this SOP are for preparing water samples collected as edge-of-field, springs or groundwater. This SOP provides detailed protocols for the handling and preparation of samples collected under the guidance of other SOPs for this program. The prepared samples were then shipped to the appropriate laboratories for analysis.

The SOP is provided in Appendix A. The SOP was a good guidance document for the work conducted. The SOP facilitated consistent sample handling and preparation of water samples at the CDM Denver laboratory in order to ship samples for analysis. Based upon my discussion with the laboratory manager and the data results, it appears that the SOP was correctly implemented. As noted in Roger Olsen's Expert Witness Report, the data can be used for all of its intended purposes. The data were used in the evaluation of impacts to surface water and groundwater within the watershed.

5.0 Overall Evaluation of Field Program

This section provides a summary of the evaluation of the SOP program's field sampling protocols, documentation, decontamination, shipping, and processing.

The SOPs developed for the Illinois River Watershed Investigation program are consistent with the guidance for the development and preparation of SOPs (US EPA 1996, 2007). The individual sampling programs were designed to acquire data consistent with the data quality objectives presented in Roger Olsen's Expert Witness Report.

The field sampling protocols were either modified from existing protocols in the documents referenced within the specific SOP or were modified from industry

standards by experienced personnel to achieve the data quality objectives for the particular sampling program. The decontamination procedures were also developed based on both industry standards and the data quality objectives for the program. The decontamination procedures presented in the SOPs addressed the biosecurity concerns of the ODAFF and poultry industry and were sufficient to minimize the potential impact of cross-contamination to below significantly measurable analytical levels.

The SOPs addressed sample handling and shipping measures as well as sample processing.

The SOPs serve as good guidance documents for the work conducted. The SOPs provide methods to produce repeatable sample collection and sample handling procedures. Based upon my site visits, photographic review, review of the field books, and review of work sheets, the SOPs appear to have been correctly implemented. Data resulting from the sample collection and handling under the SOPs are reliable and meet the data quality objectives as noted in Roger Olsen's Expert Witness Report. As noted in Roger Olsen's Expert Witness Report, the data can be used for all of its intended purposes.

6.0 References

Barbour, M.T., J. Gerritsen, B.D. Snyder, and J.B. Stribling. 1999. Rapid Bioassessment Protocols for Use in Wadeable Streams and Rivers: Periphyton, Benthic Macroinvertebrates, and Fish, 2nd Edition. EPA 841-B-99-002. U.S. Environmental Protection Agency; Office of Water; Washington, D.C.

Oklahoma Water Resources Board (OWRB). 2001. *Standard Operating Procedures (SOP) for Field Sampling Efforts of the Oklahoma Water Resources Board's Beneficial Use Monitoring Program.* Oklahoma Water Resources Board; Water Quality Programs Division; Oklahoma City, Oklahoma.

Oklahoma Water Resources Board (OWRB). 2004. Water Quality Monitoring Program: Field Sampling Protocol for Water Quality Assessments of Streams and Rivers, Draft Copy. Oklahoma Water Resources Board; Water Quality Programs Division; Oklahoma City, Oklahoma.

Oklahoma Department of Environmental Quality (DEQ). 2005. Water: Trihalomethane (THM) Fact Sheet. Available online at http://ww.deq.state.ok.us/factsheets/water /THMfactsheet.pdf

U.S. Environmental Protection Agency (US EPA). 1996. *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual*, U.S. Environmental Protection agency; Region 4, Athens, Georgia.

U.S. Environmental Protection Agency (US EPA). 2007. *Guidance for Preparing Standard Operating Procedures* (G-6) (PDF 60pp, 336K About PDF) - April 2007, EPA/600/B-07/001.

7.0 Rule 26 Disclosure Information

My billed salary rate is $150 per hour.

I have not provided expert witness testimony in the previous four years.

My considered materials are being provided under separate cover.

My resume is provided under separate cover.

My publications in the last ten years are provided in my resume.

_____

Darren L. Brown, P.G.

Appendix A

Standard Operating Procedures

Oklahoma Poultry Project

Illinois River Watershed

Darren L. Brown Expert Witness Report

Contents

Section 1.0- Lake **SOP**

1.1 Tenkiller Ferry Reservoir Sampling 1-1
1.2 Public Water Sampling 1-2

Section 2.0- High Flow

2.1 Automated High Flow Sampling 2-1
2.2 High Flow Sample Compositing and Filtering 2-2

Section 3.0- Springs

3.1 Spring Sampling 3-1

Section 4.0- Sediment

4.1 Sediment Sampling in Streams and Small Impoundments 4-1
4.2 Sediment Sampling, Tenkiller Ferry Reservoir – Not Completed 4-2

Section 5.0- Litter and Soil

5.1 Litter and Soil Sampling 5-1
5.2 Litter and Soil Sample Compositing 5-2
5.3 Manure Sampling for DNA Analysis 5-3

Section 6.0- River and Stream Water

6.1 Water Sampling of Rivers and Streams 6-1

Section 7.0- Biological

7.1 Fish Community Sampling 7-1
7.2 Periphyton Sampling 7-2
7.3 Benthic Macroinvertebrate Sampling 7-3
7.4 Stream Habitat Assessment 7-4
7.5 Combined River and Biological Sampling Program 7-5

Section 8.0- Water Quality Meters

8.1 Water Quality Meters 8-1

Section 9.0- Shipping and COC

9.1 Shipping and Chain-of-Custody 9-1

Section 10.0- Edge of Field

10.1 Edge of Field Sampling 10-1

Section 11.0- Groundwater

11.1 Residential Well Sampling 11-1
11.2 Direct Push Groundwater Sampling 11-2

Section 12.0- General Water Preparation

12.1 General Water Preparation and Filtering 12-1

STOK0020762

Prepared: Brian Bennett Review: Ronald French

Approved: Roger L Olsen Date Approved: 02/06/07

1.0 Overview and Application

This standard operating procedure (SOP) describes field procedures used for collection of surface water samples from Tenkiller Ferry Reservoir in the Illinois River watershed of eastern Oklahoma and western Arkansas. This will include water chemistry, zooplankton, phytoplankton, bacteria, and other water sampling protocols. Procedures for on site measurement of various water quality parameters such as temperature, conductivity, turbidity, pH, oxidation-reduction potential, and dissolved oxygen will also be discussed. This SOP will be used during field work and will follow the guidelines described in the *Standard Operating Procedures (SOP) for Field Sampling Efforts of the Oklahoma Water Resources Board's Beneficial Use Monitoring Program.* (ORWB, 2001).

The Illinois River watershed in eastern Oklahoma and western Arkansas has been receiving inputs from agricultural runoff, including inputs from fields where poultry waste has been applied. These wastes, along with the chemical constituents that make up the poultry wastes, have impacted the water quality and sediments in the streams and lakes in the Illinois River Watershed. The purposes of the work being performed are to evaluate and document 1) the linkage and relationship, if any, between the disposal of poultry wastes and environmental contamination within the Illinois River Watershed, and; 2) the resulting harm/injury to natural resources that may have resulted from the disposal of poultry wastes within the Illinois River Watershed, such as the level of euthrophication in Lake Tenkiller.

2.0 Sampling Methods Summary

Sampling at Tenkiller Ferry Reservoir will consist of several different tasks. At each sampling location, various water samples must be collected and on site water quality parameters must be measured. Water samples are to be collected at multiple, pre-determined depths in each location in order to obtain a representative sample of the entire water column. Likewise, the water quality parameters should be measured and recorded at 1 meter intervals from the surface to the lake bottom.

For certain parameters, samples must be preserved immediately after collection. This is accomplished by placing the samples in bottles provided by the analytical lab which already contain the necessary types and amounts of preservatives (sulfuric acid, nitric acid, hydrochloric acid, etc.). In some cases, it may be necessary to add certain preservatives after sample collection (e.g. formalin in zooplankton samples). Some samples must be filtered prior to preservation in the appropriate sample bottles by using a peristaltic pump to force the sample water through a filter pad and into a beaker or flask.

2.1 Sampling Locations

Four locations have been identified on Tenkiller Ferry Reservoir for water quality sampling. These locations are distributed throughout the main channel of the reservoir and are referred to as LK01, LK02, LK03, and LK04. They are distributed in such a way as to allow for the changes in water chemistry between the upper and lower ends of the reservoir to be detected. Additional sampling will be conducted at several raw water intakes during some of the sampling events. Table 1 highlights the maximum number of samples for each parameter that will be collected during a single sampling event. Note that all parameters will not be sampled during every sampling event.

2.2 Sample Types

Water samples can either be discrete or a composite of several discrete samples. A discrete sample is defined as a single aliquot from a specific location or depth at a given point in time. Composites are samples composed of two or more discrete samples collected from one or several sampling locations or depths. This type of sample represents an average value and can, in certain instances, be used as an alternative to analyzing a number of individual discrete samples and calculating an average value. It should be noted, however, that compositing can mask the presence of contaminants by diluting isolated concentrations of an analyte that may be present in the environmental matrix.

2.3 Sampler Selection

The choice of samplers is dictated by sampling objectives (surface versus subsurface samples) and parameters to be examined. Each sampling technique presents various advantages and disadvantages for its application. For example, sample disturbance, sample volume, chemical

and physical reactivity between potential contaminants and sampling tool materials, and ease of decontamination vary from technique to technique.

Discrete samples from any depth can be collected using a sampling device known as a Van Dorn water sampler. This sampler consists of a horizontal PVC tube that can be lowered to any depth and then closed to collect a discrete sample from that depth.

Phytoplankton and zooplankton samples will be collected with a Wisconsin-style tow net. This net is lowered through the water column to the desired depth and retrieved vertically. As it passes through the water, plankton are collected by the netting and can then be rinsed into a sample container for preservation and identification by a qualified laboratory.

2.4 Data to be Collected

At each sampling location, a variety of physical and water quality parameters will be measured and recorded. The exact location of the sampling should be measured with a handheld GPS unit and recorded. The depth of the water should also be measured and recorded either manually or with an electronic depth-finder. Water quality parameters will be measured using a YSI model 650, In-Situ Troll 9500, or similar multi-meter which will be calibrated and verified before and after each sampling event as described in Standard Operating Procedure 9.0: *Water Quality Meters*. At each sampling location, the multi-meter will be slowly lowered through the water column, and measurements will be recorded every meter once readings are stable for the following parameters:

- Temperature in degrees Celsius
- pH
- Dissolved Oxygen (DO) in milligrams per liter
- Specific Conductance in micro-Siemens per cm
- Oxidation Reduction Potential (ORP) in milli-volts
- Turbidity in Nephelometric Turbidity Units (NTU)

Additionally, Secchi depth measurements should be taken at each location during every sampling event. Secchi depths are used to assess how deeply sunlight is penetrating into the water column and are measured using a Secchi disk. This disk is weighted and lowered into

the water column until it reaches a depth at which it first becomes impossible to see from the surface. This process is repeated 3 times until an exact depth can be determined. This depth is measured and recorded as the Secchi depth. It is important that the Secchi depth measurement be taken on the side of the boat that receives the most sunlight.

2.5 Sample Nomenclature

In general, samples taken from Tenkiller Ferry Reservoir will be labeled according to the identification scheme: **AA-BB-CC-DD.**

AA is defined by the location type. The main reservoir stations are LK and each raw water intake will have a code based on the name of that intake, its location, or operator.

BB is defined by a two digit station identification number.

CC is defined by a two digit number which identifies the sample depth or location along a transect. Samples taken from the surface should be labeled SF or 0. Tow samples should be identified with a "T".

DD is defined by a two digit number which identifies the type of sample, i.e. the original sample (01), field blank (02), or a duplicate sample (03).

2.6 General Supplies

The following is a list of the minimum supplies needed for surface water sampling at Tenkiller Ferry Reservoir:

- YSI 650 or In-Situ Troll 9500 multi-meters for collecting water quality parameters
- 50 meters of cable for YSI marked at 1 meter increments
- Van Dorn water sampler
- 50 meters of rope marked at 1 meter increments for Van Dorn sampler
- Secchi Disk with 10 meter rope marked at 0.5 meter increments and 2-5 lb weight
- Vertical column sampler

- Churn splitter or similar mixing container
- Wisconsin tow net with 15 meter rope marked at 1 meter increments
- 10% formalin solution
- Lugol's solution
- Beakers/flasks
- Peristaltic or similar water pump
- Filtering apparatus
- Filters - nitrocellulose (0.45 micron)
- Sample bottles with labels
- Clear tape
- Aluminum foil
- Coolers with ice
- De-ionized water
- Phosphate-free detergent
- Nitrile gloves
- Data Sheets
- Field Notebooks with water resistant paper
- Handheld GPS Unit, with extra batteries
- Digital camera with extra batteries
- Tools for minor equipment repairs
- Anchors with ropes/chains

3.0 Sampling Procedures

Due to the relatively large number of analyses that will be conducted throughout the course of the sampling, different numbers and types of samples must be collected during each event. Therefore, the procedures for collecting water samples from Tenkiller Ferry Reservoir will vary based on parameters to be analyzed at each location during a specific sampling event. All samples should be collected and preserved in accordance with both the OWRB guidelines and the specific analytical laboratory sampling requirements.

Coordinates for sample locations LK01, LK02, LK03, and LK04 were recorded using a handheld GPS unit during the first sampling event. For subsequent events, field crews used a handheld GPS unit to navigate to the sample coordinates. It is very difficult to anchor on the exact coordinates for every sampling event, especially if sampling is conducted during high wind conditions. As a result, field crews will navigate to as close as possible to the original sample coordinates, but not greater than 100 feet away. After the boat is anchored and stable, gps coordinates will be recorded on field sheets or in the log book. If the boat drifts outside the 100 feet criteria during water quality measurements or sampling, field crews will pull anchor and navigate back to the recorded coordinates.

3.1 Sample Collection

Procedures for collecting the water samples are somewhat different depending on the type of sample desired. The following procedures should be employed.

3.1.1 Discrete Sample Collection

Discrete water samples are to be collected with Van Dorn sampler so that a sample from a specific depth may be collected and brought to the surface without being mixed with water from other depths. The steps required to take a discrete water sample are listed below:

- Calculate the number of samples you will be collecting from a particular depth at that location and prepare all the necessary bottles.
- If filtering is required, set up a decontaminated filtering apparatus and insert a new filter (wear nitrile gloves when handling the filters).
- Prepare the Van Dorn sampler for use by rinsing it and setting the spring-loaded stoppers. Make sure the messenger is ready to be deployed.

- Lower the sampler to the required depth by counting the number of 1 meter marks on the rope that pass through the surface of the water.
- Once at the correct depth, release the messenger and allow it to fall through the water until you can feel it make contact with the sampler. This should trigger the stoppers to close, thus sealing the water inside the device.
- Bring the sampler to the surface and slowly drain the contents through the attached hose into the churn splitter.
- Keep the hose under the surface of the water in the container or direct the stream at an angle against the inside wall of the container to limit the amount of oxygenation that may occur.
- If necessary, filter the sample by slowly pouring it into the filtering apparatus and allowing the pump to force it through the filter.
- Carefully fill the appropriate sample bottles with the sample water.
- Store the samples in a cooler with plenty of ice.

3.1.2 Plankton Sample Collection

At each of the four main sampling locations, phytoplankton and zooplankton sample will be collected. These samples will be collected using a Wisconsin-style tow net, a column sampler, and/or a Van Dorn sampler. Procedures for collecting plankton samples with the column or Van Dorn samplers are described in sections 3.1.1 and 3.1.2, respectively. The procedures for plankton sampling via tow net are as follows:

- Prepare the tow net by rinsing it and connecting a rope to the top end of the sampler. The rope should be clearly marked in 1 meter increments.
- Place the top end of the sampler in the water and allow it to begin sinking while holding on to the bottom of the net. Watch for pockets of air that might prevent the net from sinking properly.
- Once the tow net has straightened out, release the bottom end and allow the sampler to descend to the required depth.

- When the tow net reaches that depth, quickly pull up the sampler. This will invert the device and allow it to continue collecting plankton as it ascends.
- Remove the net from the water and, using a spray bottle and de-ionized water, rinse the inside of the cone so that any attached organisms will be flushed into the mesh cup at the bottom.
- Using a large-bore 100ml syringe, extract all water and organisms from the collection cup and place them in a sample bottle.
- For phytoplankton samples, add 5 drops of Lugol's solution to the sample container.
- For zooplankton samples, add 10ml of 10% formalin solution to the sample container.

3.2 Filtering Samples

Some parameters require that the sample be filtered in the field prior to being preserved. This is accomplished with the use of a filtering apparatus, nitrocellulose filters, and a peristaltic pump. Some types of filter pads must be prepared in advance of a sampling event. Generally, this involves rinsing them with laboratory-grade, de-ionized water two times and allowing them to soak for several hours. Refer to instructions supplied with the filters or in the analytical procedure for specific details. Procedures for filtering in the field are described below:

- Set up the filtering apparatus by attaching the pump to a filtering flask and inserting the appropriate filter.
- Turn the pump on and begin slowly adding the sample water to the filtering apparatus. Again, try not to aerate or disturb the water too much.
- Allow the water to drip through the filter and into the flask until the required amount of sample water has accumulated in the flask. In some cases, the filter may need to be changed before the necessary amount can be filtered for that sample.
- Pour the filtered water into the appropriate sample bottles, label, and store on ice.

3.3 Chlorophyll *a* Samples

The procedure for collecting chlorophyll *a* samples is somewhat different then most other sample types. This type of sampling involves preserving the sample prior to filtering. Instead of sending the filtered water to the laboratory, the filter is preserved and sent to the lab for analysis. Procedures for chlorophyll *a* sampling are outlined below:

- Collect a discrete water sample from the required depth as described in previous sections of this document.
- Set up the filtering apparatus and insert the glass filter pads distributed by the laboratory. These filters are different from the ones used for other parameters and should be inserted with the rough side of the pad facing up.
- Add 1ml of $MgCO_3$ preservative to a 100ml sample of water and stir gently.
- Pour the water with preservative into the filtering apparatus and allow the entire contents to be flushed through the filter.
- Remove filter pad and fold it in half so that the bottom of the pad is on the outside.
- Wrap the filter in a piece of aluminum foil and place in an opaque sample bottle. This will ensure that no light can reach the sample material.
- Label the bottles and store on ice.

3.4 Raw Water Intake Samples

In addition to the four main sampling stations on Tenkiller Ferry Reservoir, several raw drinking water intakes around the reservoir and nearby tributaries will be sampled. These raw water intakes are used for public and private water supplies and will be sampled primarily for one parameter: Trihalomethane Formation Potential (TFP).

Trihalomethanes (THMs) are a group of chemicals that can be formed as byproducts of a water chlorination process. The EPA regulates the amount of THMs that can be found in drinking water. The amount of THM found in a water supply is dependent on certain characteristics of the source water and of the disinfection process (Oklahoma DEQ, 2005). For this reason, the water immediately surrounding some of the raw water intakes in the reservoir will be sampled.

Procedures for collecting these samples are the same as any non-filtered, discrete water sample described in this document. However, the locations for sampling will differ from the main sampling stations.

4.0 Sample Containers, Preservation Techniques, Quality Control

Refer to **Table 1** for the specific analyses and analytical methods. Either at the end of the week or periodically throughout the sampling, samples will be packed and shipped in coolers to one of several different analytical laboratories (contact information below) depending on analyses required. A description of sample containers required for each parameter is also provided in **Table 2**.

Field control samples will be collected by the sampling team to determine whether data are of suitable quality. Control samples may include trip blanks, duplicates, decontamination (rinsate) blanks, or split samples. Duplicates (either co-located or split samples) should be collected at least once for every twenty samples.

4.1 Decontamination Procedures

To ensure that samples are not contaminated by equipment or containers, it is necessary to follow certain procedures for cleaning or decontaminating equipment. All sampling equipment which is in direct contact with the sample water must be cleaned between each sample collection. Equipment which must be decontaminated will include, but is not limited to: Van Dorn samplers, churn splitters, filtering apparatus, beakers or flasks, and volumetric measurement devices.

Procedures for decontamination are as follows:

- Rinse all surfaces with de-ionized or distilled water.
- Using a spray bottle, apply a layer of phosphate-free detergent to all surfaces.
- Rinse all surfaces again with de-ionized or distilled water until all detergent has been removed.
- If possible, rinse the container with water from the sample source.

4.2 Laboratory Contact Information

The following is a list of contact information and shipping addresses for all analytical laboratories used for water samples from Tenkiller Ferry Reservoir. Refer to Table 2 for a list of samples that should be sent to each laboratory.

General Engineering Laboratories, LLC
701 Pine Ridge Road,
Unit 5
Golden, CO 80401
Contact: Paul Winkler, 720-253-3093
E-mail: Paul.winkler@gel.com

Aquatic Research Inc
3927 Aurora Avenue North
Seattle, WA 98103
Contact: Steve Lazoff, 206-632-2715

A&L Analytical Laboratories, Inc.
2790 Whitten Rd.
Memphis, TN 38133
Contact: Jimmy Ferguson or Scott McKee, 800-264-4522
E-mail: smckee@allabs.com

Environmental Microbiology Laboratory
1150 Bayhill Drive, Suite 100
San Bruno, CA 94066
Contact: Cole Mackelprang, 858-268-2762
E-mail: cmackelprang@emlab.com
Contact: Megan S. Tatreau, 858-268-2770
E-mail: mtatreau@emlab.com

Idaho State University-Department of Biological Sciences
650 Memorial Drive
Pocatello, ID 83209-8007
Contact: Erin O'Leary-Jepsen (208) 282-4890

Aquatec Biological Sciences
273 Commerce St.
Williston, VT 05495
802-860-1638
Contact: Jennifer Gallant or Phil Downey
E-mail: jgallant@aquatecb.com

Jeff Janik, Ph.D.
2928 Boathouse Avenue
Davis, CA 95616
530-304-2286
E-mail: jjanik@omsoft.com

5.0 Documentation

Bound field logbooks should be used for the maintenance of field records. All aspects of sample collection and handling as well as visual observations shall be documented in the field logbooks. Supplemental information may be documented on the field data sheets provided. All entries in field logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.

6.0 Additional Information

Other contact information:
Ronald French
CDM
100 North Tucker Blvd., Suite 550
Saint Louis, MO 63101
Telephone: 314-241-8510

7.0 References

Oklahoma Water Resources Board (OWRB). 2001. *Standard Operating Procedures (SOP) for Field Sampling Efforts of the Oklahoma Water Resources Board's Beneficial Use Monitoring Program.* Oklahoma Water Resources Board; Water Quality Programs Division; Oklahoma City, Oklahoma.

Oklahoma Department of Environmental Quality (DEQ). 2005. *Water: Trihalomethane (THM) Fact Sheet.* Available online at:
http://www.deq.state.ok.us/factsheets/water/THMfactsheet.pdf

8.0 Revised Dates*

The following revision dates are applicable to this SOP:

Revision 1 – November 29, 2005

Table 1. An example of samples to be collected at each site during an expanded sampling event. Not all samples will be collected during each event.

	Number of Samples from Each Site (Depths distributed equally for each parameter)				
Parameters – Expanded Parameter Sampling	**Raw Water Intakes 5 locations**	**Lake #1 6 depths**	**Lake #2 6 depths**	**Lake #3 3 depths**	**Lake #4 3 depths**
Total dissolved solids, TDS		3	3	3	3
Total suspended Solids, TSS		3	3	3	3
Total organic carbon		3	3	3	3
Total Phosphorus (P-365.2), non-filtered		6	6	3	3
Total P, ortho/reactive, non-filtered		6	6	3	3
Dissolved P total (365.2), field filtered		6	6	3	3
Dissolved P, ortho/reactive, field filtered		6	6	3	3
Nitrogen, nitrate + nitrite		6	6	3	3
TKN, ammonium		6	6	3	3
Metals/6010/6020 – TAL+Mo+ P - dissolved (field filtered)		3	3	3	3
Metals/6010/6020 – TAL+Mo+P - total (non-filtered)		3	3	3	3
Trihalomethane Formation Potential (TFP)	Approximate intake depth				
anions: sulfate, chloride and alkalinity		3	3	3	3
Estrogen metabolites		2	2	2	2

P analyses (total P, total dissolved P and soluble reactive P - 4500PF)		6 (+1 at 3m)	6 (+1 at 3m)	3	3
Bacteria: six types bacteria		Surface	Surface	Surface	Surface
PCR		Surface	Surface	Surface	Surface
Chlorophyll *a*		3	3	3	3
Zooplankton		1 (15m tow)	1 (15m tow)	1 (5m tow)	1 (5m tow)
Phytoplankton		1 (15m tow)	1 (15m tow)	1 (5m tow)	1 (5m tow)
Note: Not all parameters will be sampled during each sampling event.					

Table 2. Summary of sample parameters, bottles used, preservatives, filtering required, and analytical laboratories used during Tenkiller Ferry Reservoir sampling.

Parameters	Bottles		Preservative	Filtered	Analytical Lab
TAL Metals +P +Mo (6010/6020)	500 mL	plastic	Nitric Acid	Yes	A&L
TAL Metals +P +Mo (6010/6020)	500 mL	plastic	Nitric Acid	No	A&L
TKN, Total P (365.2), ammonium	500 mL	plastic	Sulfuric Acid	No	A&L
Total P (365.2)	500 mL	plastic	Sulfuric Acid	Yes	A&L
Total Suspended Solids (TSS), Total Dissolved Solids (TDS), Total Ortho P, Nitrate+Nitrite, pH, Spec Cond.	1 liter	plastic	None	No	A&L
Dissolved Ortho P, Anions (Chloride Sulfate Alkalinity)	500 mL	plastic	None	Yes	A&L
Total Organic Carbon (TOC)	2 x 40mL	VOA vials	Hydrochloric Acid	No	A&L
Dissolved Organic Carbon (DOC)	2 x 40mL	VOA vials	Hydrochloric Acid	Yes	A&L
Total P /SRP/TDP	60 mL	plastic	None	No	Aquatic Research
Chlorophyll *a*	250 mL	brown plastic	$MgCO_3$	Yes- send filters	Aquatec

Zooplankton	125 mL	plastic	10% Formalin	No	Aquatec
Phytoplankton	125 mL	plastic	Lugol's Solution	No	Aquatec or Jeff Janik
Trihalomethane Formation Potential (TFP)	1 liter	glass	None	No	A&L or Alpha Labs
Estrogen Metabolites	1 liter	amber glass	H2SO4	No	GEL
Bacteria (6 types)	500 mL liter	sterile plastic	None	No	EML
Polymerase Chain Reaction (PCR)	1 liter	sterile plastic	None	No	Idaho State University

Prepared: Andrew Santini Review: Jeremy Berblinger

Approved: Roger L Olsen Approved Date: 2/06/07

1.0 Overview and Application

This standard operating procedure (SOP) describes field procedures used for collection of public drinking water samples from water supply distribution systems in the Illinois River Watershed for analysis of trihalomethanes (THM) and haloacetic acids (HAA). The SOP also includes procedures used for collection of surface water samples at the raw water intake locations for the water supply systems.

THM and HAA are a group of chemicals that are produced by a reaction between inorganic and organic compounds in water (e.g., algae and organic matter) with chlorine used for disinfection to control microbial contaminants. The EPA regulates the amount of THM and HAA that can be found in drinking water because of the potential risk of increased cancer rates associated with exposure to these chemicals. The amount of THM and HAA found in a water supply is dependent on certain characteristics of the source water and of the disinfection process (Oklahoma DEQ, 2005). Samples will also be collected in order to assess the THM formation potential, and the actual presence and concentrations of THM and HAA within a water system at Gore PWA, Cherokee RWD #2, and Tahlequah water systems.

2.0 Sampling Methods Summary

Several days before sampling, field crew members will contact each water plant manager identified in Section 6 to schedule a date and time for sampling. This gives each manager enough time to contact the owners or residents where the sampling will occur. Plant managers will accompany field crew members to each sampling location. Sampling will consist of four residential or commercial locations within each of the three water supply systems, in addition to the incoming raw and finished water at the plant, for a total of eighteen locations. The raw water intakes for the three systems will also be sampled in a manner consistent with the procedures outlined in SOP 1-1 Tenkiller Ferry Reservoir Sampling. Raw water intakes will be sampled for THM formation potential, chlorophyll *a*, TOC, and alkalinity. Public water sampling locations will be sampled for THMs and HAAs. Sampling procedures are detailed in Section 3.

THM and HAA samples must be preserved immediately after collection. Bottles provided by the laboratory already contain the necessary types and amounts of preservatives. For THM analysis, each sample will include two 40-ml VOA vials preserved with ascorbic acid and field crews will add 2 drops of 1:1 HCl per vial. Cap and invert vials to mix, making sure there is no headspace or air bubbles present. For the HAA analysis, each sample will include four 40-ml VOA vials preserved with NH_4Cl and filled with no headspace or air bubbles. THM formation potential samples do not require a preservative, but the water should be put in a one-liter amber glass container with minimal headspace. A trip blank will be provided by the laboratory.

2.1 Sampling Locations

Sample locations have been identified in three water supply systems: Gore PWA, Cherokee RWD #2, and Tahlequah. Sampling locations were determined based on the information such as historic sample results, population, number of connections, water use, etc. **Table 1** provides a list of each sample location within a specific water system, along with the parameters to be analyzed. Public water samples will be collected from taps inside or outside a residential or commercial property and from sinks found in kitchens or bathrooms. These locations correspond to permitted locations regularly sampled by plant personnel. Sample locations are not to have any aerators, water purification, or water softening units as these may interfere with the analytical results. Raw water intakes samples will be collected at the same locations as those collected during the Tenkiller Ferry Reservoir sampling.

2.2 Sample Types

Water samples will be grab samples. A grab sample is defined as a single aliquot from a specific location or depth at a given point in time. This type of sample represents a single value and can, in certain instances, be used as an alternative to analyzing a number of individual discrete samples and calculating an average value.

2.3 Sample Nomenclature

In general, samples taken during the public water sampling event will be labeled according to the identification scheme: AAA-xxx-DD.

AAA is defined by the location type. The public water supply system locations will start with "PWA", while raw water intake locations will begin with "RWI".

xxx is defined by a truncated location ID.

DD is defined by a two digit number which identifies the type of sample i.e. the original sample (01), field blank (02), or a field split sample (03).

2.4 General Supplies

The following is a list of the minimum supplies needed for public water sampling:

- Beakers/flasks
- Sample bottles with labels
- Clear tape
- Coolers with ice
- De-ionized water
- Phosphate-free detergent
- Nitrile gloves
- Data Sheets
- Field Notebooks with water resistant paper
- Handheld GPS Unit, with extra batteries
- Digital camera with extra batteries

3.0 Sampling Procedures

3.1 Public Water Sampling

In general, the following procedures should be employed when collecting public water samples.

- Contact each plant manager prior to sampling. The plant managers will guide and accompany the field crews to each sampling location within their respective distribution system. A list of the locations is included in Table 1.
- Each water supply system will have six total sampling locations. Four of the locations are from residential or commercial properties, while the other two are of the raw and finishing/treated water at the plant.
- At sampling locations within Gore PWA and Tahlequah, allow tap or faucet to run one to two minutes, and up to five minutes for Cherokee RWD#2 locations. These run times are consistent with how each plant manager samples the locations on a regular basis and will provide a comparable dataset to historic and future results.
- After the tap or faucet has been allowed to flush for several minutes, either collect the water in a one-liter glass beaker or fill vials directly underneath the tap or faucet. Be careful not to overflow the vial as each one contains a specific amount of preservative. Two 40-ml VOA vials preserved with ascorbic acid will be filled and 2 drops of 1:1 HCl will be added to each vial for THM analysis. Cap each vial and invert to mix, making sure there is no headspace or air bubbles present. If air bubbles are present, uncap the vials and refill so that a meniscus forms over the vial opening. Recap and check again for air bubbles. It is very important that there are no air bubbles in the vials, as these will interfere with the analytical results.
- Similarly, four 40-ml VOA vials preserved with NH_4Cl will be filled for HAA analysis. Again, cap and invert each vial to mix, making sure there is no headspace or air bubbles present.
- Since most of the sample locations are in public places, it is best to collect the water in a one liter beaker and fill vials at the field vehicle. This will help minimize disturbance to properties owners and the public.
- Store samples in a cooler with plenty of ice until shipment to appropriate laboratories. Package sample vials carefully with bubble wrap and secure inside the cooler to minimize chance of breakage during shipment.
- Record sample information in a bound field book.

- Fill out appropriate COC forms.
- Record coordinates using a handheld GPS unit if the location varies from the original sample location.

3.2 Raw Water Intake Sampling

In addition to the public water supply sampling, the raw water intakes for each system will be sampled consistent with SOP 1-1 Tenkiller Ferry Reservoir Sampling. This includes collecting Gore PWA and Cherokee RWD#2 samples by boat, at a depth of 3 meters below the surface using a Van Dorn sampler, and Tahlequah at the surface. These raw water intakes will be sampled for THM formation potential. In general, the following procedures should be employed when collecting raw water Intake samples.

- Discrete water samples will be collected with the use of a Van Dorn sampler so that a sample from a specific depth will collected and brought to the surface without being mixed with water from other depths.
- Prepare the Van Dorn sampler for use by rinsing it and setting the spring-loaded stoppers. Make sure the messenger is ready to be deployed.
- Lower the sampler to the required depth by counting the number of 1 meter marks on the rope that pass through the surface of the water.
- Once at the correct depth, release the messenger and allow it to fall through the water until you can feel it make contact with the sampler. This should trigger the stoppers to close, thus sealing the water inside the device.
- Bring the sampler to the surface and slowly drain the contents through the attached hose into a churn splitter.
- Keep the hose under the surface of the water in the container or direct the stream at an angle against the inside wall of the container to limit the amount of oxygenation that may occur.
- Carefully fill the appropriate sample bottles with the sample water.
  - For THM formation potential, fill 1 one-liter amber glass bottle with no preservative.
  - For Chlorophyll a, fill 1 250-ml brown plastic bottle. (If storing the chlorophyll a sample for more than one day before shipping, it will be important to filter 100 ml of water with 1 ml of $MgCO_3$ onto a glass filter pad as outlined in SOP 1-1).

- For TOC, fill 2 40-ml VOA vials pre-preserved with HCl.
- For Alkalinity, filter at least 250 ml into a clear plastic bottle with no preservative as outlined in SOP 1-1.
- Store the samples in a cooler with plenty of ice until shipment.

4.0 Sample Containers, Preservation Techniques, Quality Control

Refer to **Table 1** for the specific analyses for each sample location. At the end of the sampling day, samples will be packed and shipped in coolers to the analytical laboratories (see contact information below) depending on analyses required in Table 1. VOA vials and one-liter amber glass bottles will be bubble wrapped to minimized breakage during overnight shipment to the laboratories.

Field control samples will be collected by the sampling team to determine whether data are of suitable quality. Control samples may include trip blanks, duplicates, decontamination (rinsate) blanks, or split samples. Duplicates (either co-located or split samples) should be collected at least once for every twenty samples.

4.1 Decontamination Procedures

To ensure that samples are not contaminated by equipment or containers, it is necessary to follow certain procedures for cleaning or decontaminating equipment. All sampling equipment which is in direct contact with the sample water will be cleaned between each sample collection. Equipment which will be decontaminated include, but is not limited to: Van Dorn sampler, filtering apparatus, beakers or flasks, and volumetric measurement devices.

Procedures for decontamination are as follows:

- Rinse all surfaces with de-ionized or distilled water.
- Using a spray bottle, apply a layer of phosphate-free detergent to all surfaces.
- Rinse all surfaces again with de-ionized or distilled water until all detergent has been removed.
- If possible, rinse the container with water from the sample source.

4.2 Laboratory Contact Information

The following is a list of contact information and shipping addresses for analytical laboratories used for the public water and raw water samples collected under this SOP. Refer to Table 1 for a list of samples that should be sent to each laboratory.

Alpha Woods Hole Analytical Labs
Eight Walkup Drive
Westborough, MA 01581-1019
Contact: Denise McLaughlin (508) 898-9220
E-mail: dmclaughlin@alphalab.com

A&L Analytical Laboratories, Inc.
2790 Whitten Rd.
Memphis, TN 38133
Contact: Jimmy Ferguson or Scott McKee, 800-264-4522
E-mail: smckee@allabs.com

Aquatec Biological Sciences
273 Commerce St.
Williston, VT 05495
802-860-1638
Contact: Jennifer Gallant or Phil Downey
E-mail: jgallant@aquatecb.com

5.0 Documentation

Bound field logbooks should be used for the maintenance of field records. All aspects of sample collection and handling as well as visual observations will be documented in the field logbooks. All entries in field logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.

6.0 Additional Information

Other contact information:

Gore PWA
Dallas Sitton (918) 489-2636 (water plant)
(918) 774-6425 (cell)

Cherokee RWD#2
David Pulliam (918) 822-0123 (water plant)

Tahlequah
Ken Johnson (918) 456-2123 (water plant)

7.0 References

Oklahoma Water Resources Board (OWRB). 2001. *Standard Operating Procedures (SOP) for Field Sampling Efforts of the Oklahoma Water Resources Board's Beneficial Use Monitoring Program.* Oklahoma Water Resources Board; Water Quality Programs Division; Oklahoma City, Oklahoma.

Oklahoma Department of Environmental Quality (DEQ). 2005. *Water: Trihalomethane (THM) Fact Sheet.* Available online at:
http://www.deq.state.ok.us/factsheets/water/THMfactsheet.pdf

8.0 Revised Dates*

There are no revisions applicable to this SOP at this time.

Table 1. An example of samples to be collected at each location in the water system. "X" denotes the parameter(s) to be analyzed for the location.

System	Location	THM[1]	HAA[1]	THMFP[1]	TOC/Alkalinity[2]	Chlorophyll a[3]
Gore PWA	Gore High School (teachers lounge bathroom sink)	X	X	-	-	-
	Marval Trout Resort (men's bathroom sink)	X	X	-	-	-
	City Complex (kitchen sink)	X	X	-	-	-
	Corps of Engineers Office (sink in lounge area)	X	X	-	-	-
	Treatment Plant - Raw Water (tap inside)	X	X	-	-	-
	Treatment Plant - Finished Water (sink in lab)	X	X	-	-	-
	Raw Water Intake	-	-	X	X	X
Cherokee RWD #2	Maurice Johnson (kitchen sink)	X	X	-	-	-
	Keyes Fire Dept. (sink in lounge area)	X	X	-	-	-
	John Bates (bathroom sink)	X	X	-	-	-
	Jimmie Houston Store (usually from men's bathroom sink, but both bathrooms closed – took from sink behind counter)	X	X	-	-	-
	Treatment Plant - Raw Water (outside tap)	X	X	-	-	-
	Treatment Plant - Finished Water (sink in lab)	X	X	-	-	-
	Raw Water Intake	-	-	X	X	X
Tahlequah	Dano's (tap on side of store)	X	X	-	-	-
	E-Z Mart (sink inside store)	X	X	-	-	-
	WPC Plant (bathroom sink)	X	X	-	-	-
	Welling Store (tap on back of store)	X	X	-	-	-
	Treatment Plant - Raw Water (tap outside)	X	X	-	-	-
	Treatment Plant - Finished Water (sink in lab)	X	X	-	-	-
	Raw Water Intake**	-	-	X	X	X

Notes:

1 – Alpha Analytical

2 – A&L Analtyical

3 – Aquatec Biological Sciences

Prepared: Jeremy Berblinger Review: Tim Cox

Approved: Roger L. Olsen Date Approved: 2/06/07

1.0 Background and Scope

Instructions presented in this standard Operating Procedure (SOP) are for collecting representative high flow samples from streams during runoff events using ISCO automated samplers. This SOP describes in detail the protocol for the sampling proposed for the Illinois River Watershed Sampling Program.

The Illinois River watershed lies in eastern Oklahoma and western Arkansas. Water quality in this watershed is impaired, likely due primarily to agricultural runoff. The dominant industry in the watershed is poultry, an industry where farmers routinely spread their waste on open fields. The SOP outlined here supports a sampling program aiming to: 1) directly measure and summarize flow-weighted concentrations of key pollutants of concern associated with runoff events in small watershed tributaries, and; 2.) investigate potential correlations between these concentrations and land use characteristics, poultry operations, and storm event hydrographs characteristics.

2.0 Equipment Specifications

ISCO automated in-stream samplers, model 6712, are recommended for this sampling program. These samplers draw samples from a given stream according to pre-programmed specifications and can be triggered by increases in stream stage or flow rate. They have been widely used for this type of application.

Model 6712 samplers are housed in drum-like containers approximately 20 inches in diameter by 30 inches high. The 6712 sampler has the capacity to collect 24 liters of water in 24 one-liter polyethylene bottles or in a single composite bottle. The bottles are secured to the base of the unit with a retainer ring. Stream water is pumped through a suction line with a peristaltic pump housed in the top of the drum, and is distributed to the bottles via a rotating arm. The sampler is programmable and allows for collection of samples at predetermined times, or after a threshold stage or flow is reached in the river. Each sampler should be equipped with an ISCO 730 or 750 Flow Meter that monitors the stage and/or flow rate of the river. Sampler programming is described in Section 5.

3.0 Sampling Locations and Schedule

Prior to sampler deployment, potential sampling locations should be evaluated using available information. For the objectives described here, the following information should be used to select locations: stream size and order, sub-basin location and size, land use, sub-basin chicken house density, locations of suspected litter application fields, and locations of active USGS flow gaging stations. The number of sampling locations identified and the number of samplers deployed should be sufficient to: a.) achieve statistical significance in the correlation analysis; b.) cover a wide range in values and basin characteristics with respect to the criteria defined above; c.) provide an adequate number of alternative sites should any of the initial preferences be deemed unsuitable in the field; and d.) fit in with the given time, budget, and personnel constraints. For the program described here, it is recommended that a minimum of ten (10) and a maximum of fifteen (15) samplers be deployed simultaneously.

Samplers should be deployed in early spring (March) and, if possible, remain in place until late fall (October) in order to capture a range of seasonal storm events and field conditions.

4.0 Sampler Installation

Installation of the samplers will be guided by manufacturer recommendations and past experience.

At each location, the sampler unit should be positioned so as not to exceed the elevation head limitations (see manufacturer guidelines) while still being out of the main channel flow path. Typically, this is along the margins or banks of the stream. It is recommended that the units be secured to a tree or permanent structure (such as a bridge abutment) to safeguard against both vandalism and high flow disturbance. Past experience shows that an effective way of protecting and securing the sampler units is to contain them in a 55 gallon poly drum with a secured lid. Holes can be drilled in the side to accommodate drainage, cable connections and the sampling suction tube. Additionally, a chain or cable should be threaded through the holes and padlocked to any available permanent structure.

The suction tubes from the river to samplers should be positioned according to recommendations provided by the manufacturer and based on professional experience with installations at other sites. Power to the samplers should be supplied by 12 volt deep cycle marine batteries. Batteries should also be protected and secured. If 55 gallon drums are being used, the battery should fit either in the bottom of the drum or on top of the sampler (but still

inside the drum). The batteries will need to be replaced or recharged periodically for long deployment periods.

The sampler intake and flow meter probe (Model 750 or 730) must be secured to the stream bed. Past experience suggests that an effective way to do this for gravel and cobble bed streams is to attach the intake and probe to a piece of sheet metal (approximately 2 x 2 feet) which can then be staked to the stream bottom using rebar. Cable ties and drilled holes in the sheet metal are a good way of securing the devices to the sheet metal.

Stream flow gaging should be performed with each sampler installation and periodically throughout the sampling period, following standard USGS protocol. The initial gaging should include detailed channel cross-section mapping up to the estimated high flow line. These data can be used to establish an approximate relationship between stage, velocity, and flow. Alternatively, ISCO now offers heavy metal plates that can be bolted to the bottom substrate to serve this purpose.

5.0 Sampler Programming

Samplers should be programmed to capture the full duration of significant high flow events. The typical duration and targeted minimum magnitude of captured events must be determined apriori using available stream hydrograph and area precipitation data. Sampling intervals within a given event must also be determined apriori based on typical storm durations, sample bottle constraints (24), and desired hydrograph resolution.

Each of the samplers will be programmed to trigger following an increase in river stage that correlates to rainfall. The goal is to obtain samples during storm events that result in high flow conditions in the Illinois River Watershed.

The ISCO Model 6712 sampling program is setup using a keypad and electronic screen. In the programming mode (Extended 6712.2), the display steps the user through the sampler programming process by indicating various parameters to be used. The example described below assumes a 24-bottle setup, with a 2-part program: part 'A" = bottles 1 – 10 (hourly) and part 'B' = bottles 11 – 24 (every 3 hours). The reader is referred to the ISCO 6712 operators manual for more details.

The first step in the programming is to enter extended programming mode. Power up the machine, and if the words "Standard Programming" appear on the second line of the start-up message then the sampler is in standard programming mode. To enter extended programming

mode, type 6712.2 at the main menu and hit enter (arrow key). This will take you to the extended program screen which is where you will begin programming. An example list of settings for programming the ISCO model 6712 is shown below:

- Program type: **Extended.**
- Program Name: e.g., **"Springtown"** this is the name of the town or stream where the sampler is located.
- Site Description: e.g. **"02"** this will be the number that particular site was assigned.
- Units Selected for Length: **Feet; Flow Rate: Cubic Feet per Second (CFS), Flow Volume: Cubic Feet (CF), and Velocity: Feet per Second (FPS).**
- Area-Vel Module: **Level and Velocity.**
- **Data Interval: 10 minute.**
- Bottle kit: **24, 1000-milliliter bottles.**
- Suction-line length: This is in feet, the maximum is **25 feet** and smaller streams might be less.
- Suction Head: **Auto Suction Head.**
- Next step is **Line Rinses: None** or **0** is what should be selected.
- Sampling retries: **None** or **0** should be selected.
- Part 'A' and 'B' bottle assignments: **Bottles 1-10 to part 'A', bottles 11-24 to part 'B'.**
- Part 'A' pacing: **Time pacing, sampling every 1 hours, 0 minutes.**
- Part 'A' distribution: **Sequential, 1 sample per bottle.**
- Part 'A' sample volume: **1000 milliliters.**
- Part 'A' enable: Enable varies for each location, e.g. **0.2** feet added to current level of **0.425 feet**. The enable screen would be programmed to read **Level >0.625 feet (0.2 + 0.425 = 0.625)** and when water level reaches above this point, the sampler would trigger and begin sampling.

- Part 'A' enable: **Once enabled, stay enabled.**
- Part 'A' enable: **Sample at enable.**
- Part 'A' enable: **0 pause & resumes.**
- Part 'B' pacing: **Every 3 hours, 0 minutes.**
- Part 'B' distribution: **Sequential, 1 sample per bottle.**
- Part 'B' sample volume: **1000 milliliters.**
- Part 'B' enable: **When 'A' is done.**
- Part 'B' enable: **Once enabled, stay enabled.**
- Part 'B' enable: **No sample at enable.**
- Part 'B' enable: **0 minute delay to start of sampling.**
- Part 'B' enable: **0 pause & resumes.**
- **No delay to start.**
- Run this program now? YES

6.0 Maintenance and Sample Collection

Samplers should be checked every 7-10 days to make sure they are functioning properly, have not been vandalized, and have sufficient battery power. Battery voltage can be checked by testing the battery directly with a voltage meter (likely requiring removal of the sampler from the barrel). Alternatively, the power supply cord on the back of the sampler can be removed and voltage can be checked by placing the voltage meter probes in each of the holes on the power supply cord. The following "tips" are provided to assist personnel in performing maintenance checks and collecting samples from the ISCO units:

- The screen will read "done" if the sampling has already occurred and is complete and ready for bottles to be collected. (See Download Data section if screen reads done).

- If, for example, the screen reads "Part 'A' is done .... Bottle 21" : this means that the Part "A" program or bottles 1-10 are complete and now the sampler is on Part 'B' bottles 11-24. The screen will display the exact time the next bottle is set to sample. Sample collection will need to occur at a later time when the program is complete.
- If the screen reads "Part 'A' is active and disabled and Part 'B' is active and disabled": this means that the sampler has not triggered (the trigger depth was not reached).
- If the screen reads "Power Failure": this means the battery is dead or below the voltage required to power the sampler. (Needs to be above 12 volts)
- If sampler is done:
  - Remove lid by pulling top portion off to access bottles.
  - Individual bottle lids should be labeled with date, bottle number and site numbers and placed on bottles prior to removing from sampler. Bottle positions are provided on the edge of the sampler base. Lids should be secured before being removed from sampler base to avoid spilling.
  - Bottles are secured to the base by a retainer ring and elastic cords. Remove cords and ring after lids are secured on bottles.
  - Once the bottles are extracted from sampler, remove base from barrel and drain any excess water from sampler base and barrel to avoid moisture build up inside barrel.
  - Refill sampler base with new or cleaned and sterilized empty bottles (without lids) and secure ring and cords in base of sampler. It is important to make sure bottles are aligned properly with numbers around outside of sampler base.
  - Place sampler base back inside barrel on top of battery (if that is the preferred configuration) and place top portion of sampler onto the base. Make sure sampler is level inside barrel and not tilting to either side.
  - The lid to the ISCO sampler must be secure each time it is put on and aligned correctly to help prevent the distributor arm from being jammed.
- Always write down in the field log book the current depth, velocity, programmed enable depth, and current and replacement battery voltage.

- Check and make sure tubing is not crimped inside barrel and place lid on barrel and secure with bungee ring.
- Place zip tie on bungee lid clasp to ensure no tampering has taken place the next time samplers are serviced.
- Before leaving the site, check probe and intake port to make sure they are clear of debris.

7.0 Data Download

The following steps and tips are provide to assist personnel in downloading electronic data pertaining to the sampling event from the ISCO samplers:

- Downloading data will be performed using a Rapid Transfer Device (RTD).
- If sampler screen reads done, then data will need to be downloaded before samples can be removed from the sampler.
- The first step in downloading data using the RTD is to remove the barrel lid to gain access to sampler.
- Look on the back of the top portion of the sampler and there will be three ports.
- Unscrew the cap off the middle port. It should have a laptop icon above it.
- Plug RTD unit into port.
- On top of the RTD unit, two lights will be present, one orange and one green.
- The orange light should be on and constant; the green light will be blinking intermittently signifying data are being transferred.
- Downloads can last anywhere from 5-20 minutes depending on amount of data being downloaded.
- The download is complete when the green light stops blinking and remains lit and solid.
- Remove the RTD unit and screw the cap back on transfer port.

- Proceed with collecting samples as described in section 4.0.

8.0 Tips and Troubleshooting

The following are general operating and troubleshooting tips based on past experience with this sampling program:

- Establishing appropriate trigger (or "enable") depths is critical to a successful sampling program. If your enable depth is too high, you could miss portions, or all, of good representative events. Alternatively, if your enable depths are too low, you will create a lot of unnecessary work for the sampling team and may miss significant events because of previous triggering during a preceding insignificant event.

- Make sure there is a secure and robust connection to the power supply (battery). We have found the screw clamp connections much more reliable than alligator clip connections. Batteries need to be routinely checked.

- To conserve battery life, the backlight option for the display screen should be disabled. Data intervals need to be in synch when readings are taken and when readings are stored.

- To help avoid corrosion of battery connections, coat battery terminals with electrolyte grease after each battery change.

- It is not uncommon for electrical storms to cause shorts in your sampler. Samplers should be checked immediately following local electrical storms.

- The correct type of tubing (flexible rubber) must be used with the peristaltic pump (through the pump housing). The manufacturer or lease company should supply this tubing and it is generally identified by 2 or 3 blue bands. The intake/suction line and the distribution line can be any type of standard ½ inch poly tubing.

- Distributor arm jams are not uncommon and are often caused by improper alignment of the sample bottles in the sampler tray. Bottles must be all the way at the bottom of the tray with no single bottle extending higher than the others.

- Programming steps should be followed closely per the manual. If the instructions on the screen are not matching up with the description in the manual, then likely an error or a

missed step occurred previously in the programming. Starting over, from the beginning, is generally a good idea in these cases.

- It is important that the "once enabled, stay enabled" option is selected in programming for the application described here.
- Past experience has shown that the velocity probe associated with the 750 Flow Module is unreliable. Too much or too little suspended particles in the water can cause large oscillations and inaccurate readings. Velocities should be used as supplementary data only, rather than relied upon for flow estimates and/or sample compositing.
- Screen messages indicating "spectrum error" are indicative of poor velocity readings due to insufficient water turbidity. Per the above, these messages are not cause for alarm and will not effect sampling under depth-enabled programming.
- Careful consideration should be given to the legality of sampler placement with respect to private vs. public property. In the States of Oklahoma and Arkansas, the stream channel and banks up to the high flow line are considered public land, as are most bridges and roads.
- A test of the sampler system is recommended upon installation. There are a variety of ways to force the sampler to grab a test sample immediately, including setting the enable depth to the actual current water depth. This type of test would reveal any mechanical or structural problems associated with collectcing a sample.

9.0 Documentation

Bound field logbooks should be used for the maintenance of field records. All aspects of sample collection and handling as well as visual observations will be documented in the field logbooks. Supplemental information may be documented on the field data sheets provided. All entries in field logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.

10.0 Additional Information

Other contact information:

Bert Fisher, PhD
Lithochimeia, Inc.

222 South Kenosha Ave.
Tulsa, OK 74120
Telephone: 918-382-9784

11.0 Revised Dates*

The following are other revision dates applicable to this SOP:

Revision 2: 02/06/06
Revision 1: 01/18/06

SOP: 2-2
Standard Operating Procedure
High Flow Sample Compositing and Filtering

Revision: 1
Initial Date: 04/21/06
Last Revised: 02/05/07

Prepared: Todd Burgesser Review: Tim Cox

Approved: Roger L Olsen Date Approved: 2/06/07

1.0 Background and Scope

Instructions presented in this standard Operating Procedure (SOP) are for compositing and filtering high flow samples from streams during runoff collected using ISCO automated samplers. This SOP describes in detail the protocol for the compositing and filtering high flow samples as outlined for the Illinois River Watershed Sampling Program.

The Illinois River watershed lies in eastern Oklahoma and western Arkansas. Water quality in this watershed is impaired, likely due primarily to agricultural runoff. The dominant industry in the watershed is poultry, an industry where farmers routinely spread their waste on open fields. The SOP outlined here supports a sampling program aiming to: 1) directly measure and summarize flow-weighted concentrations of key pollutants of concern associated with runoff events in small watershed tributaries, and; 2.) investigate potential correlations between these concentrations and land use characteristics, poultry operations, and storm event hydrographs characteristics.

2.0 Equipment Specifications

For compositing the stormwater, 250-mL graduated cylinders (1-mL gradations, glass) are used for measuring the sample prior to compositing. A 3000-mL glass beaker is used for mixing and dispensing the composited water. Nucleopore or equivalent filtering apparatus (47mm) with 0.45 u nitrocellulose filter and a stand alone vacuum pump is recommended for filtering. This device will filter the composited water at a rate of approximately 50-mL/minute.

3.0 Sample Receipt and Login

Upon receipt of coolers from field personnel, all coolers are opened and the internal temperature of the cooler is recorded on the chain-of-custody (COC) form. The thermometer used for measuring the temperature will be previously calibrated against a NIST calibrated thermometer. The samples are then removed from the cooler and transferred to a refrigerator until compositing begins. Any discrepancies with the samples and the COC are noted and

resolved at this point through conversations with the original sampler and shipper. The COC will be signed and dated with the current date and time upon receipt.

4.0 Sample Compositing Scheme

Upon receipt of the downloaded data from the RTD device at each sampling station, a compositing scheme will be developed based on depth and/or velocity at the specific flow station. This scheme (a spreadsheet showing volumes to be used from each bottle) will be followed in the formation of the composite samples.

5.0 Sample Compositing

Upon receipt of the compositing scheme, sample bottles are removed from the refrigerator and placed on a lab bench. Each bottle is inverted to mix prior to opening and dispensing. The pre-determined volume of each sample bottle is poured into a pre-cleaned 250-mL graduated cylinder and transferred into the 3000-mL pre-cleaned beaker. After each sample bottle has been added to the 3000-mL beaker and mixed, bottles designated for specific analyses and different labs are filled and returned to the refrigerator. Any remaining sample is filtered as described in section 6.0. Section 9 presents sample bottle requirements, preservatives and minimum volumes for the analysis requested.

6.0 Composite Sample Filtering

The filtering apparatus is assembled by placing the fritted glass filter holder into a 250-mL Erlenmeyer filtering flask. A 47 mm nitrocellulose 0.45u filter is placed on the fritted section of the filter holder and a 250-mL funnel is clamped to the filter holder. A vacuum pump is attached to the Erlenmeyer flask and vacuum is applied while pouring sample into the funnel. When the Erlenmeyer flask is full, vacuum is released and the filtrate is transferred to the appropriate sample bottle. A new 0.45u filter is placed onto the filter holder and the process is repeated until all appropriate sample bottles are filled. Section 9 presents sample bottle requirements, preservatives and minimum volumes for the analysis requested.

7.0 Equipment Decontamination

All equipment including the filtering apparatus, Erlenmeyer flask, graduated cylinders, beakers and sample bottles received from the field should be thoroughly cleaned before use by washing vigorously with a brush, hot water and Alconox detergent. The equipment will then be rinsed with de-ionized water to remove any remaining soap followed by a rinse with 10% bleach and a last rinse with de-ionized water. The equipment is allowed to air dry before use. The cleaned sample bottles from the field will be packed into coolers with caps and shipped back to Tulsa for re-use.

8.0 Sample Shipping to Contracted Laboratories

After sample compositing and filtration the samples are packed into coolers with appropriate padding (bubble wrap, packing paper). Ice contained in re-sealable baggies is placed within the cooler followed by the completed chain-of-custody form. The cooler is closed and secured with packing tape followed by a signed custody seal. All coolers will be shipped to the respective laboratories vie Federal Express for overnight delivery.

9.0 Analyses, Bottle Requirements, Preservatives and Minimum Sample Volumes, Analytical Laboratory

The following table presents potential analysis, bottle requirements, preservatives, minimum sample volumes and analytical laboratory.

Analysis	Bottle	Preservative	Minimum Volume	Analytical Laboratory
Total Organic Carbon	2-40-mL glass	HCl	80 mL	A&L
Total Metals+Mo+P	500-mL poly	HNO_3	125-mL	A&L
Dissolved Metals+Mo+P (filtered)	500-mL poly	HNO_3	125-mL	A&L
TKN, Ammonia	500-mL poly	H_2SO_4	225-mL	A&L
Sulfate, Chloride, Alkalinity (filtered)	500-mL poly	None	350-mL	A&L
Nitrate+Nitrite, TSS, TDS, pH	500-mL poly	None	400-mL	A&L

Total and Dissolved P; soluble reactive P	150-mL poly	None	100-mL	Aquatic Research
Estrogens	1000-mL amber glass	H_2SO_4	1000-mL	GEL
PCR	1000-mL poly sterile	None	1000-mL	Northwind/Idaho State University
Bacteria	500-mL poly sterile	None	500-mL	EML

10.0 Documentation

Bound laboratory logbooks should be used for the maintenance of field records. All aspects of sample compositing and handling as well as visual observations will be documented in the field logbooks. Supplemental information may be documented on the field data sheets provided. All entries in laboratory logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.

Prepared: Pamela Rede Review: Jeremy Berblinger

Approved: Roger L. Olsen Date Approved: 2/06/07

1.0 Scope and Application

Instructions presented in this Standard Operating Procedure (SOP) are for collecting representative water samples from flowing spring water bodies or springs. In particular, this SOP describes in detail the spring sampling for the Illinois River Watershed sampling.

In the Illinois River Watershed, over 50 springs or seeps have been identified. These springs typically result from groundwater seepage or outflow at specific locations and durations. This task will evaluate the chemical composition of the springs. The information will be used to provide a preliminary evaluation of potential groundwater contamination.

Based on the locations of known springs in the United States Geological Survey (USGS) database and locations where various samples have been collected by Universities, approximately 20 springs will be selected for sampling. Samples will be collected and field parameters measured (pH, ORP, SC, DO, and temperature). Samples for dissolved constituents will be filtered in the field using a 0.45 micron filter. The preserved samples will be sent to the laboratories for analyses.

2.0 Sampling Methods Summary

Presented below are sampling instructions for collecting spring water samples. Prior to sample collection, water body characteristics (size, depth, flow) will be recorded in the field logbook. Sampling will proceed from downstream locations to upstream locations so that disturbance from sampling does not affect sampling quality. In collecting spring water samples from any source, care must be taken to minimize disturbance as it is retrieved.

The sampler should not place the sample bottle into the flowing spring water because some sample bottles contain chemical preservatives. Instead, the spring water should be collected in a container (bucket or gallon jug) that has been properly washed and rinsed at each sample location.

2.1 Spring Water Sampling Locations

Based on the locations of known springs in the United States Geological Survey (USGS) database and locations where various samples have been collected by Universities, approximately 20 springs in the Illinois River watershed have been selected for sampling. In order to collect a representative spring water sample, the sample location must be within 200-feet of the spring head (location where the water table outcrops). In the event the spring head is not accessible, the sample location should be hydrologically tied to the location where the water is flowing out of the ground surface.

2.2 Sample Types

Water samples can either be discrete or composite samples. A discrete sample is defined as a single aliquot from of a specific location at a given point in time. Composites are samples composed of two or more specific aliquots (discrete samples) collected from one or several sampling locations and/or different points in time. This type of sample represents an average value and can, in certain instances, be used as an alternative to analyzing a number of individual discrete samples and calculating an average value. It should be noted, however, that compositing can mask the presence of contaminants by diluting isolated concentrations of analytes that may be present in the environmental matrix.

2.3 Sampler Selection

The choice of samplers is dictated by sampling objectives (surficial versus subsurface samples) and site constraints based on water depth. Each sampling technique presents various advantages and disadvantages for its application. For example, sample disturbance, sample volume, chemical and physical reactivity between potential contaminants and sampling tool materials, and ease of decontamination vary from technique to technique.

Table 1. List of the minimum equipment required for spring sampling.

Equipment	Amount
Yellow Springs Incorporated (YSI) Meter	1
Peristaltic Pump	1
12 Volt Battery	1
Low Density Polyethylene tubing	Several Feet (disposable)
Pump Head tubing (pure silicon)	Several Feet (disposable)

0.45 micron filters	May use 2 to 3 filters per sampling site
Container (gallon jug or bucket)	2
Field Book	1
GPS with compass to show north	1
60 mL syringes	20
Labels	Enough for each samples bottle
Permanent markers and pens	3 each
Coolers for storing and shipping	Enough to carry and ship samples

2.4 Data to be Collected

At each spring sampling location, a suite of physical variables should be recorded. These variables are intended to locate each sampling point and to quantify factors likely to be associated with collecting a representative sample. Variables may include:

- Water depth.
- Approximate flow rate.
- Distance from spring head to sample location.
- Surrounding land use (i.e. farmland, residential, private vs. public property, etc).
- Sketch of the spring location.
- Photo of the sample location (minimum of two photos per site).
- Presence or absence of algae growth.
- Latitude and Longitude readings.
- Temperature.
- Dissolved Oxygen as a percentage and in milligrams per liter.
- Oxidation-Reduction Potential.
- Conductivity.
- pH.

2.5 Sample Nomenclature

Samples should be labeled according to the given names (i.e. Living Water Springs, July Springs, etc) followed by the date the sample was collected. These names were either issued by the United States Geological Survey (USGS) or by local residents. If additional springs are found (located in the field but was not from the USGS database) and the sampler determines it is a good location but has no associated name, the sampler should label the spring by either the name of the nearest resident or another unique name. As long as there are latitude and longitude readings, the location of the spring will be known. The date will be included along with the spring name in case there are multiple visits.

3.0 Spring Sampling Procedures

There are two ways the sample can be collected. By collecting the sample in a large container or if the flow is immediately inaccessible or low flow, the spring water can be pumped through a tube directly into the bottles for non-filtered samples or pumped through a filter for the field filtered samples.

When filtering the spring water sample, it is more convenient to use a battery-operated peristaltic pump. A minimum of 1-gallon of the spring water should be purged before taking any samples. If the flow from the spring is too shallow to submerge a gallon jug or bucket, then the water for both filtered and non-filtered samples must be collected using a tube and pump. Otherwise, the non-filtered samples can be collected directly from the stream.

3.1 Sampling Using the Container Method

1. Before approaching the spring, make an assessment as to whether it is located on private property. If the spring is on private property, the sampler will obtain the landowner's permission to enter the property for collecting spring samples.

2. Before sampling at each site, a meter reading (using a YSI meter) will be taken. Once the meter appears to have stabilized (i.e. the values are changing less than 10% between readings), document the time and then

the parameter (pH, DO%, DO mg/L, temperature, ORP and conductivity). Three meter readings will be taken at each sampling site.

3. Locate the proposed sample location with a GPS unit and take photographs (minimum of two photographs per site).

4. If flow is adequate, take a bucket or gallon jug and collect the spring water and rinse the bucket several times downstream of the sample location. A gallon jug with the top cut off works well because it is flexible and there is a handle.

5. Grab a homogenous, representative sample of spring water by emerging the bucket or jug into the middle stream of water preferably at the point where the groundwater surfaces or very close to that point. Keep sediment and debris from entering the sample. As stated before, the sample location should preferably be no more than 200-feet downstream of the point where the water reaches the ground surface.

6. Fill the appropriate bottles by pouring slowly without touching the collection jug to the bottles. Included in these bottles for non-filtered samples will be a one-liter amber glass bottle, four 250-mL sterile plastic bottles, one plastic bottle with nitric acid preservative, one plastic bottle with sulfuric acid preservative, A one-liter plastic bottle with no preservative, and two 40-mL amber glass with hydrochloric acid preservative. When filling the 40-mL amber glass bottles, allow no room for air bubbles. A complete listing of bottles for spring sampling is presented in this SOP.

7. The container method of collecting the spring water works well for collecting the non-filtered samples; however, for the filtered samples, pumping the spring water (using a peristaltic pump and tubing connected to a 0.45 micron filter) works best. In the event that the spring is located a distance away from the vehicle (far enough from the road that a hike is required), the spring water may be collected in a container, and then taken back to the vehicle and pumped from the container, through the filter and into the bottles.

8. To pump the spring water from the container and through the filter, extend Low Density Polyethylene (LDPE) tubing into the container.

9. Connect the LDPE tubing to a section of flexible pure silicon pump head tubing and then run the pump head tubing through the pump.

10. Connect the pump to a 12.0 Volt battery using the cable provided in the pump kit. The red clip connects to the positive side of the battery and the black clip connects to the negative side of the battery. The other end of the cable connects to the pump for power.

11. Secure the tubing and then connect the other end into the inlet side of a 0.45 micron filter.

12. Turn the pump on and allow the water to run through the tubing, through the filter and into the sample bottles (only after flushing a minimum of 1-gallon through the pump). The bottles for filtered samples include a one-liter plastic bottle with no preservative, a plastic bottle with nitric acid preservative, and a plastic bottle with sulfuric acid preservative. A complete listing of bottles for spring sampling is presented in this SOP.

13. Label each container by using the spring name as outlined in Section 2.5. Be sure to complete the label carefully and clearly, addressing all the categories and parameters. Include the date, time the sample was collected (military time) and the initials of the sampler.

14. Store the sample bottles on ice until transfer shipment to the analytical laboratories.

15. Complete all chain-of-custody documents, field sheets and record in the field logbook.

16. Decontaminate sampling equipment after use and between sample locations using Alconox and deionized water.

17. All other equipment (i.e. tubes for the pump, filters and medical gloves) are disposable.

18. Repeat procedure for each sample location.

3.2 Sampling Using the Pumping Method

1. Before approaching the spring, make an assessment as to whether it is located on private property. If the spring is on private property, the sampler will obtain the landowner's permission to enter the property for collecting spring samples.

2. Before sampling at each site, a meter reading (using a YSI meter) will be taken. Once the meter appears to have stabilized (i.e. the values are changing less than 10% between readings), document the time and then the parameter (pH, DO%, DO mg/L, temperature, ORP and conductivity). Three meter readings must be taken at each sampling site.

3. Locate the proposed sample location with a GPS unit and take photographs (minimum of two photographs per site).

4. For the non-filtered samples, if the spring flow is not readily accessible, it is best to use a pump because the sampler can extend the LDPE tubing into the spring water and use the pump to collect the sample in the appropriate bottles for collecting non-filtered samples (only after flushing a minimum of 1-gallon through the pump).

5. For the field filtered sample, it is more convenient to pump the water directly from the spring, through the filter and then into the appropriate bottles.

6. To pump the spring water, extend the LDPE tubing into the stream flow, approximately mid depth so not to collect debris or sediment.

7. Connect the LDPE tubing to a section of flexible pure silicon pump head tubing and then run the pump head tubing through the pump.

8. Connect the pump to a 12.0 Volt battery using the cable provided in the pump kit. The red clip connects to the positive side of the battery and the black clip connects to the negative side of the battery. The other end of the cable connects to the pump for power.

9. Secure the tubing and then connect the other end into the inlet side of a 0.45 micron filter.

10. Turn the pump on and allow the water to run through the tubing, through the filter and into the sample bottles (only after flushing a minimum of 1-gallon through the pump). The bottles for filtered samples include a one-liter plastic bottle with no preservative, a plastic bottle with nitric acid preservative, and a plastic bottle with sulfuric acid preservative. A complete listing of bottles for spring sampling is presented in this SOP.

11. Label each container by using the spring name as outlined in Section 2.5. Be sure to complete the label carefully and clearly, addressing all the categories and parameters. Include the date, time the sample was collected (military time) and the initials of the sampler.

12. Store the sample bottles on ice or in a refrigerator until transfer shipment to the analytical laboratories.

13. Complete all chain-of-custody documents, field sheets and record in the field logbook.

14. Decontaminate sampling equipment after use and between sample locations using Alconox and deionized water.

15. All other equipment (i.e. tubes for the pump, filters and medical gloves) are disposable.

16. Repeat procedure for each sample location.

3.3 Sampling Using a Syringe

In some cases, the spring location may be quite a distance from the road. In this case, it is not convenient to carry the battery and pump (along with all other supplies) the entire distance. Also, the container method may not work if the flow is low. The following is a procedure to collect spring water samples using a syringe.

1. Before approaching the spring, make an assessment as to whether it is located on private property. If the spring is on private property, the sampler will obtain the landowner's permission to enter the property for collecting spring samples.

2. Before sampling at each site, a meter reading will be taken. Once the meter appears to have stabilized (i.e. the values are changing less than 10% between readings), document the time and then the parameter (pH, DO%, DO mg/L, temperature, ORP and conductivity). Three meter readings must be taken at each sampling site.

3. Locate the proposed sampling location with a GPS Unit and take photographs (minimum of two per site).

4. Withdraw the spring water using a 60-mL or larger capacity syringe. Push the water back out and do this several times to rinse the inside of the syringe.

5. For the non-filtered samples, place the spring water directly into the appropriate bottle being careful not to touch the tip of the syringe to the bottle.

6. For the field filtered samples, withdraw the spring water, connect the inlet side of a 0.45 micron filter to the tip of the syringe and then gently push the water through and into the appropriate bottles for filtered samples. Do not force the water through too hard because this may puncture a hole in the filter.

7. Label the containers by using the spring name and date system as outlined in Section 2.5. Be sure to complete the label carefully and clearly, addressing all the categories and parameters. Include the date, time the sample was collected (military time) and the initials of the sampler.

8. Store the samples on ice until shipment to analytical laboratories.

9. Complete all chain-of-custody documents, field sheets and record in the field logbook.

10. Decontaminate sampling equipment after use and between sample locations using Alconox and deionized water.

11. All other equipment (i.e. tubes for the pump, filters, medical gloves and syringe) are disposable.

12. Repeat procedure for each sample location.

4.0 Laboratory Analysis, Sample Containers, Preservation Techniques

Table 1 presents a list of all parameters that may be analyzed and a reference to the standard method for that analysis.

Table 1. Parameters and methods summary for water samples collected at springs within the Illinois River watershed.

Parameter	Method
Total dissolved solids, TDS	160.1
Total suspended Solids, TSS	160.2
Total organic carbon	415.2
pH	150.0
Specific Conductivity	120.1
Total Phosphorus (P), nonfiltered	365.2, persulfate digestion
Total P, ortho/reactive, nonfiltered	365.2, direct
dissolved P total, filtered	365.2, persulfate digestion
Dissolved P, ortho/reactive, filtered	365.2, direct
Nitrogen, nitrate + nitrite	353.3
Total Nitrogen	TKN, 351.3
Nitrogen, ammonium	350.2
TAL Metals, dissolved (filtered)	EPA SW-3050/6010/6020
Dissolved Molybdenum (Mo)	EPA SW-3050/6010/6020
Dissolved Phosphorus (P)	EPA SW-3050/6010/6020
TAL Metals, total (nonfiltered)	EPA SW-3050/6010/6020
Total Molybdenum (Mo)	EPA SW-3050/6010/6020
Total Phosphorus (P)	EPA SW-3050/6010/6020
Sulfate, filtered	375.1
Alkalinity, filtered	310.2
Chloride, filtered	325.1
Bacteria	
Total coliform	SM-9221B
enterococcus	SM-9230B
Fecal coliform	SM-9221E
e-coli	SM-9221F
staphylococcus	BAM 12
campylobacter	BAM, CHAP. 7
salmonella	BAM 5
17α-estradiol,17β-estradiol, estrone, estriol	LC-MS-MS

The following is a list of sample bottles required during collection for all parameters:

- 1- 500 mL plastic with nitric acid, non filtered for total metals and P
- 1- 500 mL plastic with nitric acid, filtered in field for dissolved metals and P
- 1- one L plastic with no preservative, non filtered for TDS, TSS, pH, nitrite+nitrate, ortho-P by 365.2
- 1- one L plastic with no preservative, filtered in field for dissolved ortho-P by 365.2, sulfate, chloride, alkalinity
- 1- 500 mL plastic with sulfuric acid, non filtered for total P by 365.2, TKN and ammonium
- 1- 500 mL plastic with sulfuric acid, filtered for dissolved total P by 365.2
- 2- 40 mL VOA vial with hydrochloric acid, non filtered for TOC
- 1- one L glass, non filtered for estrogen metabolites
- 1- 250 plastic sterile, non filtered for bacteria

4.1 Quality Control

Periodically throughout the sampling, samples will be packed and shipped in coolers to one of several different analytical laboratories (contact information below) depending on analyses required. Field control samples will be collected by the sampling team to determine whether data are of suitable quality. Control samples may include trip blank, duplicates, decontamination (rinsate) blanks or split samples. Duplicates collected as co-located or split samples should be collected at least once for every twenty samples.

4.2 Decontamination Procedures

To ensure that samples are not contaminated by equipment or containers, it is necessary to follow certain procedures for cleaning or decontaminating equipment. All sampling equipment

which is in direct contact with the sample water must be cleaned between each sample collection.

Procedures for decontamination are as follows:

- Rinse all surfaces with de-ionized or distilled water.
- Using a spray bottle, apply a layer of phosphate-free detergent to all surfaces.
- Rinse all surfaces again with de-ionized or distilled water until all detergent has been removed.

4.3 Laboratory Contact Information

The following is a list of contact information and shipping addresses for all analytical laboratories used for water samples.

Bottles for estrogen metabolites (one liter glass) will be shipped to:
General Engineering Laboratories, LLC
701 Pine Ridge Rd, Unit 5
Golden, CO 80403
Contact: Paul Winkler, 720-253-3093
Paul.winkler@gel.com

Bottles for nutrients, metals, P, etc (8 bottles: two 500 ml with nitric, two 500 mL with sulfuric, two 1-L no preservative, two 40 mL VOA with hydrochloric) will be shipped to:
A&L Analytical Laboratories, Inc.
2790 Whitten Rd.
Memphis, TN 38133
Contact: Scott McKee, 800-264-4522
smckee@allabs.com

Bottles for bacteria analyses (one 250 mL plastic sterile) will be shipped to:
Environmental Microbiology Laboratory
1150 Bayhill Drive, Suite 100
San Bruno, CA 94066
Contact: Meagan S. Tatreau 858-268-2762
E-mail: mtatreau@emlab.com

5.0 Documentation

Bound field logbooks will be used for the maintenance of field records. All aspects of sample collection and handling as well as visual observations will be documented in the field logbooks. Supplemental information may be documented on the field data sheets provided. All entries in field logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.

6.0 Additional Information

Other contact information:

Bert Fisher, PhD
Lithochimeia, Inc.
222 South Kenosha Ave.
Tulsa, OK 74120
Telephone: 918-382-9784

7.0 References

Oklahoma Water Resources Board (OWRB). 2001. *Standard Operating Procedures (SOP) for Field Sampling Efforts of the Oklahoma Water Resources Board's Beneficial Use Monitoring Program.* Oklahoma Water Resources Board; Water Quality Programs Division; Oklahoma City, Oklahoma.

Herlihy, A.T. 1998. Water chemistry. pp. 57-65 IN: J.M. Lazorchak, D.J. Klemm, and D.V. Peck (Eds.). Unpublished draft. *Environmental Monitoring and Assessment Program - Surface Waters: Western Pilot Study Field Operations Manual for Wadeable Streams.* EPA/620/R-94/004F. U.S. Environmental Protection Agency, Washington, D.C.

8.0 Revised Dates*

Revision 2 – January 19, 2006

Revision 1 – September 30, 2005

Prepared: Andrew Santini Review: Jeremy Berblinger

Approved: Roger L. Olsen Date Approved: 2/06/07

1.0 Scope and Application

Instructions presented in this Standard Operating Procedure (SOP) are for collecting representative sediment samples from surface water bodies. Sediment can be considered as solid material that is submerged/saturated (at least temporarily) or suspended in any surface water body. This includes sludges, lake bottom sediments, perennial and intermittent stream sediments, and marine sediments. This SOP describes in detail the sampling proposed for the Illinois River Watershed Sampling.

The Illinois River watershed in eastern Oklahoma and western Arkansas has been receiving inputs from agricultural runoff, including from fields where poultry waste has been applied. These wastes, along with the chemical constituents that make up the poultry waste, have apparently impacted the water quality and sediments in the streams and lakes in the Illinois River Watershed. The purposes of the work being performed are to evaluate and document 1) the linkage and relationship, if any, between the disposal of poultry wastes and environmental (primarily aquatic system) contamination within the Illinois River Watershed, and; 2) the resulting harm/injury to natural resources that may have resulted from the disposal of poultry wastes within the Illinois River Watershed.

2.0 Sampling Methods

Presented below are sampling instructions for the most common tube sampling technique for collecting sediment samples. For additional information, see Plumb, 1981 (00-0231), Spigolon, 1993 (00-0232), and Shelton (94-458). Prior to sample collection, water body characteristics (size, depth, flow) should be recorded in the field logbook. Sampling should proceed from downstream locations to upstream locations so that disturbance from sampling does not affect sampling quality. In collecting sediment samples from any source, care must be taken to minimize disturbance and sample washing as it is retrieved through the liquid column. Sediment fines may be carried out of the sample during collection if the liquid above is flowing or deep. This may result in collection of a non-representative sample due to the loss of contaminants associated with these fines.

While a sediment sample is usually expected to be a solid matrix, the sampler should not place the sample in the bottle and then decant the excess liquid. If the sample is collected properly, any liquid in the bottle is representative of sediment conditions. If the liquid above the sediment collection point is either flowing or greater than 6 inches in depth, a corer or other device that eliminates sample washing may be used to collect the sample to minimize washing the sediment as it is retrieved. It may be necessary to decant standing water from the top of the core. This should be done carefully and prior to transfer to the sample bottle.

2.1 Sediment Sampling Locations

In-stream sediment core samples will be collected from over fifty locations in streams, rivers, and lakes within the Illinois River Watershed.

2.2 Sample Types

Sediment samples can either be discrete or composite samples. A discrete sample is defined as a single aliquot from of a specific location at a given point in time. Composites are samples composed of two or more specific aliquots (discrete samples) collected from one or several sampling locations and/or different points in time. This type of sample represents an average value and can, in certain instances, be used as an alternative to analyzing a number of individual discrete samples and calculating an average value. It should be noted, however, that compositing can mask the presence of contaminants by diluting isolated concentrations of analytes that may be present in the environmental matrix.

2.3 Sampler Selection

The choice of samplers is dictated by sampling objectives and site constraints based on water depth. Each sampling technique presents various advantages and disadvantages for its application. For example, sample disturbance, sample volume, chemical and physical reactivity between potential contaminants and sampling tool materials, and ease of decontamination vary from technique to technique.

Discrete sediment samples from shallow to moderately deep water can be collected efficiently using polycarbonate (or Lexane) tube. Polycarbonate tube samplers are easy to use, portable, and are a direct method for obtaining sediment samples. The tube is forced into the sediment

and then withdrawn using a vacuum/suction technique. Additional sample methods may be utilized if the tube sampling method is not effective in collecting fine grained sediments. Alternative methods that may be applied are the use of a ponar dredge according to procedures provided by the Oklahoma Water Resources Board's Beneficial Use Monitoring Program (BUMP) or stainless steel scoop. These methods will be used as contingencies.

2.4 Data to be Collected

At each sediment sampling location, a suite of physical variables should be recorded. These variables are intended to locate each probing point and to quantify factors likely to be associated with deposition and accumulation of soft sediments. Variables may include:

- Water depth.
- Qualitative rating surface sediment type—silt, sand, gravel, cobble.
- Qualitative assessment of presence or absence of cohesive soft sediment.
- Sample location river segment type—straight, inside bend, outside bend, depositional zones, etc.
- Presence or absence of benthic organisms (i.e. observation of larval forms present or encrusted on river bottom cobble and rock).

2.5 Sample Nomenclature

Samples should be labeled according to the identification scheme: AA-BBB-CC-DD

AA is defined by:

- SD = Sediment
- SL = Soil
- FW = Fish
- SW = Surface Water

- BN = Benthics
- ZP = Zooplankton/Phytoplankton

BBB is defined by a three digit station identification number.

CC is defined by a two digit number which identifies the sample location along a transect. If the transect is identifier is not applicable, 00 should be used.

DD is defined by a two digit number which identifies the type of sample; i.e.; the original sample (01), field blank (02), or a duplicate sample (03).

3.0 Sediment Sampling Procedures

It is anticipated that the sampling locations are composed of sand/gravel/cobble mix and may require the use a sieves to separate out the fine sediment. Three Hubbard plastic sieves, with stainless steel US mesh sizes (5, 10, 35), are to be used. The sieves should be shaken aggressively and the small amounts of sediment should be worked through the sieves (while wearing medical nitrile gloves). The sediment that is retained on and passes through the no. 35 sieve will be kept for the sample. The sediment on the no. 5 and 10 sieves will be discarded.

3.1 Tube Sampling Procedure

The following is a procedure to collect sediment samples using polycarbonate tubes.

1. Locate the proposed sample location with a GPS unit. If there are no fine grained sediments at the location, the sample crew should go upstream and/or downstream of the planned coordinate until fine grained sediments are encountered.

2. Once at the location of fine grained sediment, record the water depth from the top of surface water to the top of sediment.

3. Lower the polycarbonate tube until the tube make contacts with the top of the sediment.

4. Gradually force tube into sediment (use a hammer or slambar if necessary to obtain the desired sediment depth of 6 inches).

5. Fill the remainder of the tube with water and cap to create a vacuum between the top of the sediment and the tubing. Tape cap to tube using either electrical or duct tape to ensure an airtight seal.

6. Pull the tube from the sediment using proper lifting techniques. If the tube becomes stuck in the sediment, two people may be needed to pull the tube. If the desired sediment recovery (6 inches) is not achieved, remove the sediment within the tube and flush using river water. Move the location over a foot and repeat procedure.

7. When desired sediment retrieval is achieved and tube is removed from the sediment, place the cap on the bottom of the tubing and secure with tape. Note that more than one core may be needed in order to obtain at 500 mL of fine sediment for the parameters. In that case, one or more samples will be collected in the same general area as the original core.

8. With tube held vertically, remove the top cap and cut a hole in the tube with a hack saw just above the top of the sediment to drain off water.

9. Cut off the tube just above the sediment surface and recap the upper end.

10. Label the top cap with the sample location ID, date, time, and "top" description using a permanent marker. Record the date, time, location of the sample, and other salient observations in the field book. Tubes must be kept vertical at an angel greater than 45 degrees.

11. Photograph each tube at the sampling location with a sign that contains the sample information detailed above.

12. Re-GPS the location if substantially different than planned coordinate or mark for survey.

13. Transport cores to the site work area, keeping them on ice.

14. The sediment core will be processed at the site work area in order to describe its structure and create samples for chemical analysis.

15. Place the polycarbonate tube on a decontaminated worktable and secure. Aluminum foil or plastic sheeting can be used to cover the worktable. For large recoveries, cut the core liner (filled with sediment) lengthwise along opposite sides. (Note: cut through the liner wall without cutting significantly into the sediment core itself. Disturbed

sediment adjacent to the liner wall should not be sampled. Also, it is important not to contaminate the undisturbed interior of the core with plastic chips or other debris from the cutting process. The best hand tool available for cutting hard plastic liners is a jig saw being careful to eliminate any of the sample with which the blade has come into contact. For shorter recoveries i.e. less than one foot of sediment, either push the sediment out or allow the sediment to fall out of the tube while holding vertically.

16. Extend a tape measure along beside the sediment, starting at the original top end of the core.

17. Photograph the core with a digital camera. Photograph the core section in overlapping frames using a small label with core field ID number so that it appears in each frame. Advance the tape measure appropriately for any additional sections of the same core.

18. While the core section is still intact, record a description of the core structure, noting zones of different color, classification, layering, sorting, and sediment type (silt, sand, clay, gravel, etc.).

19. Cores will be sectioned into one sample from the 0-6 inch interval.

20. Using disposable scoops or by hand with medical gloves, place soil on aluminum foil or stainless steel bowl and homogenize. (Note that if more than one sample was collected for a location, the sediment from all samples will be combined and homogenized.) Some locations may be more gravelly than others and may require the use a sieves as mentioned in Section 3.0. After sieving, put sediment into the appropriate sample containers.

21. Label each container with a unique sample identification number as outlined in Section 2.5. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

22. Store the sample bottles on ice or in a refrigerator until transfer shipment to the analytical laboratories.

23. Complete all chain-of-custody documents and field sheets and record in the field logbook.

24. Decontaminate sampling equipment after use and between sample locations using phosphate-free soap and deionized water. Sample equipment may include hacksaw or jigsaw blades. All other equipment i.e. polycarbonate tubing, medical gloves, etc. are

disposable. Sample material not sent to the lab for analysis will be disposed in a municipal landfill.

25. Repeat the procedure for each sample location.

3.2 Stainless Steel Scoop Sampling

The following is a procedure to collect sediment samples using a stainless steel scoop or shovel.

1. Locate the proposed sample location with a GPS unit. If there are no fine grained sediments at the location, the sample crew should go upstream and/or downstream of the planned coordinate until fine grained sediments are encountered.

2. Once at location of fine grained sediment, record the water depth from the top of surface water to the top of sediment.

3. Lower the scoop into the sediment being careful not to stirrup any sediment. Slowly bring the scoop and sample to the surface, again being careful not the let the fine sediment be washed downstream.

4. Place sediment on aluminum foil or in a stainless steel bowl and homogenize. (Note that if more than one sample was collected for a location, the sediment from all samples will be combined and homogenized.) Some locations may be more gravelly than others and may require the use a sieves as mentioned in Section 3.0. After sieving, put sediment into the appropriate sample containers.

5. Label each container with a unique sample identification number as outlined in Section 2.5. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

6. Store the sample bottles on ice or in a refrigerator until transfer shipment to the analytical laboratories.

7. Complete all chain-of-custody documents and field sheets and record in the field logbook.

8. Decontaminate sampling equipment after use and between sample locations using phosphate-free soap and de-ionized water. Sample equipment may include stainless

steel scoop, sieves, bowl etc. All other equipment i.e. polycarbonate tubing, medical gloves, etc. are disposable.

9. Repeat the procedure for each sample location.

3.3 Ponar Dredge Sampling

The following is a procedure to collect sediment samples using a ponar dredge.

1. Locate the proposed sample location with a GPS unit. If there are no fine grained sediments at the location, the sample crew should go upstream and/or downstream of the planned coordinate until fine grained sediments are encountered.

2. Once at location of fine grained sediment, record the water depth from the top of surface water to the top of sediment.

3. Insert the tension spring and slowly lower the dredge until it reaches the sediment bed. Slowly raise the dredge about two feet and let it drop into the sediment. The force of the dredge in contact with the sediment will release the tension spring and close the dredge on the top 4 to 6 inches of sediment. Pull the dredge out of the water.

4. Open the dredge and place the sediment on aluminum foil or in a stainless steel bowl and homogenize. (Note that if more than one sample was collected for a location, the sediment from all samples will be combined and homogenized.) Some locations may be more gravelly than others and may require the use a sieves as mentioned in Section 3.0. After sieving, if needed, put sediment into the appropriate sample containers.

5. Label each container with a unique sample identification number as outlined in Section 2.5. Be sure to complete the label carefully and clearly, addressing all the categories or parameters.

6. Store the sample bottles on ice or in a refrigerator until transfer shipment to the analytical laboratories.

7. Complete all chain-of-custody documents and field sheets and record in the field logbook.

8. Decontaminate sampling equipment after use and between sample locations using phosphate-free soap and de-ionized water. Sample equipment may include stainless

steel scoop, sieves, bowl etc. All other equipment i.e. polycarbonate tubing, medical gloves, etc are disposable.

9. Repeat the procedure for each sample location.

4.0 Sample Containers, Preservation Techniques, Quality Control

For sediment samples, there is no need for sample preservation beyond storing and shipping the samples on ice. Periodically throughout the sampling, samples will be packed and shipped in coolers to the appropriate laboratories. Shipping addresses and contact information are listed below:

A&L Analytical Laboratories, Inc.
2790 Whitten Rd.
Memphis, TN 38133
Contact: Jimmy Ferguson or Scott McKee, (800) 264-4522
E-mail: smckee@allabs.com

General Engineering Laboratories, LLC (GEL)
701 Pine Ridge Rd, Unit 5
Golden, CO 80403
Contact: Paul Winkler, (720) 253-3093
E-mail: Paul.winkler@gel.com

Great Lakes Environmental Center (GLEC)
739 Hastings Street
Traverse City, MI 49686
Contact: Mailee W. Garton, (231) 941-2230
E-mail: mgarton@glec-tc.com

Environmental Microbiology Laboratory
1150 Bayhill Drive, Suite 100
San Bruno, CA 94066
Contact: Meagan S. Tatreau 858-268-2762
E-mail: mtatreau@emlab.com

Field control samples will be collected by the sampling team to determine whether data are of suitable quality. Control samples may include trip blanks, duplicates, decontamination (rinsate) blanks, or split sample. Duplicates collected as either co-located or split samples should be collected one of every twenty samples.

5.0 Documentation

Bound field logbooks should be used for the maintenance of field records. All aspects of sample collection and handling as well as visual observations shall be documented in the field logbooks. All entries in field logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.

6.0 Additional Information

Other contact information:

Bert Fisher, PhD
Lithochimeia, Inc.
222 South Kenosha Ave.
Tulsa, OK 74120
Telephone: 918-382-9784

Ronald French
CDM
100 North Tucker Blvd.
Suite 550
Saint Louis, MO 63101
314-241-8510

7.0 References

Plumb, R.H., Jr. 1981. Procedures for Handling and Chemical Analysis of Sediment and Water Samples. USACE WES/EPA.

Spigolon, S.J. 1993. Geotechnical Factors in the Dredgeability of Sediments: Report 2, Geotechnical Site Investigation Strategy for Dredging Projects. Contract Report DRP-93-3. U.S. Army Engineer Waterways Experiment Station, Vicksburg, MS.

American Society for Testing and Materials. 1995. ASTM Standard D4823-95, "Guide for Core-Sampling Submerged, Unconsolidated Sediments," pp 282-295. In: American Society for Testing and Materials (ASTM). 1997. ASTM Standards on Environmental Sampling. Second Edition. ASTM Publication: 03-418097-38.

American Society for Testing and Materials. 1992 (1996). ASTM Standard D3676-92, "Practice for Preparation of Sediment Samples for Chemical Analysis," pp 301-303. In: American Society for Testing and Materials (ASTM). 1997. ASTM Standards on Environmental Sampling. Second Edition. ASTM Publication: 03-418097-38.

U.S. Environmental Protection Agency (EPA) and U.S. Army Corps of Engineers (USACE). 1998. Great Lakes Dredged Material Testing and Evaluation Manual. Final Draft. Prepared by U.S. Environmental Protection Agency Regions 2, 3, 5 and Great Lakes National Program Office and U.S. Army Corps of Engineers Great Lakes & Ohio Division.

Shelton, Larry R. and Capel, Paul D. Guidelines for Collecting and Processing Samples of Stream Bed Sediment for Analysis of Trace Elements and Organic Contaminants for the National Water-Quality Assessment Program. United States Geological Survey (USGS). 1994. Open-File Report 94-458.

9.0 Revised Dates*

The following are other revision dates applicable to this SOP.

Revision 2 – January 18, 2006

Revision 1 – July 22, 2005

Prepared: Roger Olsen Review: Darren Brown

Approved: _____ Date Approved: _____

1.0 Overview and Application

This standard operating procedure (SOP) describes field procedures used for collection of soil and litter/manure samples during the Illinois River watershed project.

2.0 Selection of Soil and Litter/Manure Sampling Locations

Sample locations will be selected from either contract growers' farms or company-owned facilities. At each of these farms/facilities, litter from the poultry houses will be collected. Fields where documentation of litter application from a specific farm and Integrator is available from the Oklahoma Department of Agriculture, Food and Forestry will also be selected for sample collection. Field locations selected will be within the Illinois River Watershed. Considerations for sample collection from farms/facilities and fields include:

1. Poultry litter/manure has been consistently generated,
2. Poultry litter/manure is currently being generated,
3. Poultry litter/manure has been consistently (every year for the at least the past 3 years) applied to land (Litter Application Locations, "LALs") associated with the Farm/Facility,
4. Availability of land upon which poultry litter/manure or other fertilizers have not been applied (Control Locations, "CLs").

To the extent possible, the following information should be collected for each associated Farm/Facility:

1. Name of Farm/Facility owner and Farm/Facility contact person,
2. Physical address and location (section-township-range) of Facility,
3. Contact address of Farm/Facility owner or Farm/Facility contact person,
4. Contact phone number of Farm/Facility owner or Farm/Facility contact person,
5. Whether or not one or more LALs can be accessed at the Farm/Facility,

6. The physical location of each LAL.
7. Whether or not one or more CLs can be accessed at the Farm/Facility.
8. The physical location of each CL.
9. Whether or not a litter/manure and/or nutrient management plan has been prepared for LALs at the Farm/Facility,
10. Estimates of the amounts, rates and dates of prior litter/manure applications to each LAL at the farm/facility,
11. Estimates of litter treatment or amendments added to each LAL (e.g., alum, etc), if any, and information as to amount, rate and dates of application
12. Number, type, dimensions, and capacity of poultry grower houses (or other poultry/egg production facilities, as appropriate) operated at Farm/Facility ("Poultry Houses").

Most of the above information may not be available to the field crews. These data may be acquired through the attorneys during deposition.

3.0 Sampling Documentation

3.1 Sampling Log Book and Sampling Forms

1. Sampling Log Books and/or Sampling Forms will be maintained by the field crews.
2. Pages in the Sampling Log Book will reference specific Sampling Forms by use of the Facility Identification.
3. The Sampling Log Book shall be bound and will be constructed of waterproof paper.
4. Entries in the Sampling Log Book or on the Sampling Form will be made in permanent ink, preferably black ink.
5. Each page of the Sampling Log Book will be dated.
6. The preparer will initial each page of the Sampling Log book.
7. If available, and to the extent possible, for each Farm/Facility sampled, the following information will be recorded in the Sampling Log Book or on the Sampling Forms:
    a. Name, address and phone number of the Farm/Facility owner,

b. Identification of the Farm/Facility, FAC1 – FAC8,

c. Name, address and phone number of the Farm/Facility operator,

d. Name, address and phone number of the Integrator responsible for the Farm/Facility,

e. Names, addresses and phone numbers of persons who have spread litter/manure on LALs associated with the Farm/Facility,

f. The amounts, rates and dates of prior litter/manure applications to specific LALs at the Farm/Facility (confirm State Reports),

g. The existence of prior soil sampling data for LALs or CLs at the facility (yes or no),

h. The water supply for the Farm/Facility,

i. The legal description (qtr-qtr-qtr-sec-twp-rng) of the property related to the Farm/Facility,

j. The legal description (qtr-qtr-qtr-sec-twp-rng) of the CLs at the Farm/Facility,

k. Type of animals generating litter (broilers, layers, pullets, turkeys, etc.),

l. Number of flocks of birds that have used the litter that is sampled,

m. The number of birds in each flock that have used the litter that is sampled,

n. The time since birds last used the litter,

o. Litter treatment (e.g. alum amendment), if any, and information as to amount, rate and date or dates of treatment,

p. Information as to any other fertilizers, chemicals or soil amendments added during the last five years,

q. Use of each LAL by cattle (yes or no) and typical number of cattle,

r. Specific information listed within this protocol,

s. Sketch map of each LAL with approximate dimensions; indicate local features on the sketch (vegetation, water bodies, adjacent fields, location of poultry houses, roads, old fence rows, livestock feeding areas, livestock grazing areas, etc); dimensions and features can also be placed on the aerial photographs,

t. Additional information such as identified springs, wells, seeps, or sinkholes should be indicated on the sketch map or aerial photograph

u. Land slope of each LAL (or LAL sub-area),

v. Distance to nearest water body,

w. Notes on weather (temperature, wind, last precipitation event, etc),

x. Type of vegetation currently on the LAL, if any, and any known vegetation grown in past 5 years,

y. Use of adjacent fields, and;

z. Other information as appropriate or relevant.

3.2 Photographic Record

A photographic record shall be made and maintained for all sampling activities on the LAL. Pictures of the LALs, CLs and the outsides of the poultry house will be taken. No pictures of sampling activities inside the poultry houses will be taken. A video recording will be made, to the extent possible, from a vantage point immediately outside the poultry house.

All photographs made shall be time and date stamped.

3.3 Chain-of-Custody

A Chain-of-Custody will be prepared for each set of samples transferred to the soil and litter processing lab (CDM Support Laboratory in Denver, Colorado). A second chain-of-custody will be prepared at the processing lab for the analytical laboratory.

The Chain-of-Custody to the soil processing lab shall, at a minimum, contain the following information:

1. The project name, ***Illinois River Watershed Soil and Litter/Manure Sampling,***
2. Name of person or entity relinquishing the sample and was part of the field crew,

3. Signature blocks with dates and times for all persons having custody (sampler, shipper, processing laboratory, etc),
4. For each sample related to a Chain-of-Custody:
   a. The unique numeric identifier on the submitted sample container/bag,
   b. The date and time interval the samples were collected,
   c. The sample "matrix" (i.e. SOIL or LITTER or WATER).

4.0 Soil Sampling

4.1 Litter Application Locations (LALs) and Control Locations (CLs)

4.1.1 Permissible Soil and Weather Conditions

1. Soils are not to be sampled if water saturated.
2. Soils are not to be sampled during precipitation events.

4.1.2 Division into Sampling Areas

A Sampling Area is an area within a LAL or CL that is reasonably homogenous with respect to soil types, soil properties, topography, landscapes, management history (to the extent known), and other relevant factors, as appropriate.

1. For each LAL or CL sampled, the LAL or CL shall be divided into a maximum of four Sampling Areas, identified as A, B, C and D.
2. Sampling Areas identified within the LAL or CL shall be a minimum of approximately one acre and shall not exceed approximately 10 acres.
3. In making determinations concerning the division of the LAL or CL into Sampling Areas, the person or persons making those determinations shall consult the USGS topographic map, aerial photograph, and/or other data including relevant USDA/NRCS soil survey. The data consulted shall be identified by reference in the Sampling Log Book.
4. The person or persons who make the determinations concerning the division of the LAL or CL into Sampling Areas shall prepare a sketch map of the LAL or CL and its constituent Sampling Areas. This sketch map shall show the approximate boundaries of each Sampling Area and the estimated area of each Sampling Area. This information can also be recorded on the aerial photographs.

4.2 Identification of Sub-Sampling Locations

A Sub-Sampling Location is a one to ten acre area within a Sampling Area at which individual soil samples will be collected. A total of 20 sub-sampling locations shall be sampled for each Sampling Area. The selection of Sub-Sampling Locations shall avoid:

1. Old fence rows,
2. Livestock feeding areas,
3. Livestock loafing areas, and;
4. Localized conditions atypical of the Sampling Area.

The geographic coordinates (Latitude and Longitude) of the first Sub-Sampling Location in each Sampling Area or a corner of the Sampling Area shall be determined using a Global Positioning System (GPS) receiver accurate to at least five (5) meters. These geographic coordinates will be recorded in the Sampling Log Book.

Representative Sub-Sampling locations will be documented with a time and date stamped photograph.

The following procedure will be used, when possible, to establish a grid system for each Sampling Area (Subareas A, B, C, or D).

1. On the map/aerial photo, select the general area to establish a grid pattern of twenty sampling locations. If the field configuration permits, the Subarea should be either a square or rectangle in shape.
2. The grid setup in either a square or rectangle shape will have 4 evenly spaced sample points within a width and 5 evenly spaced sample points within a length. In other words, the grid system will typically be a 4 by 5 grid, with sample points at the nodes.
3. If the selected grid location is near a fence line or tree line, the corner should be established by insetting a distance of Width (W) of Subarea grid divided by 8 (W/8).
4. Once the corner is established, determine the spacing of the remaining width (W) grid points by dividing the remaining width (RW) by 3. RW equals W – W/4. In summary, width grid points will be established at W/8 and then at distances of (W-W/4)/3.
5. For the length of the Subarea grid, the spacing of the length (L) grid points will be the remaining length (RL) divided by 4. Keeping in mind that the inset distance was W/8, RL equals L – W/4. In summary, length grid points will be established at W/8 and then at distances of (L-W/4)/4.

6. If the Subarea grid is more than 50 feet from a fence or tree line within a field, the grid spacing is simply W/3 by L/4.
7. If the field configuration does not permit the use of rectangular or square Subarea field configurations, try to establish a grid that provides for relatively uniform spacing within the field shape.
8. Record the grid spacing in the field book and, if possible, on the aerial photograph or map.
9. Once the grid spacing has been determined on the aerial photograph or map, the field crew shall use the maps to establish grid layout in the field. Conditions permitting, the grid points will be marked with pin flags, which will be removed after the grid point is sampled.

4.3 Soil Samples to be collected at each Sub-Sampling Location

For purposes of this Protocol, a Sub-Sampling Location shall be an area defined by a triangle with three-foot sides with the middle placed on the Sub-Sampling Location. When possible, one point of the triangle will be oriented in the north direction.

At each Sub-Sampling Location, core samples with a length of at least six inches will be collected at the corners of the triangle. The samples will be divided into three separate soil samples as follows:

1. Four (4) to Six (6) Inch Sample depth. This two inch sample will be collected by measuring the length of the core from the top of the sample. The two inch section of core will be placed in a plastic bag with the appropriate identification.
2. Two (2) to Four (4) Inch Sample depth. This two inch sample will be collected by measuring the length of the core from the top of the sample. The two inch section of core will be placed in a plastic bag with the appropriate identification.
3. Zero (0) to Two (2) Inch Sample depth. This two inch sample will be collected by measuring the length of the core from the top of the sample. The two inch section of core will be placed in a plastic bag with the appropriate identification.
4. One core sample will be collected at each corner of the triangle until enough sample is collected (approximately 100 to 200 grams, depending upon QA/QC needs). The first core will be at the triangle corner oriented to

the north. Additional cores, if necessary, will be collected at the remaining triangle corners.

5. The soil samples collected at each sub-sampling location will be collected with soil probe coring devices either marked for 6-inch, 4-inch and 2-inch depths, or with a vertical slot so that the core measurements can be made with a ruler. The diameters of all soil corers used should be the same, and should be of a diameter consistent with general practice for agricultural soil sampling.
6. Whenever a soil sample is to be collected, thatch and other plant residue shall be moved aside or lightly scuffed aside without removing the surface soil prior to pushing the soil probe core into the soil.
7. Coring devices will be manually driven to at least six inches in depth if possible. If coring devices are being driven with a post hole driver and the coring device shows no or very limited advancement after ten consecutive blows, the coring device will be considered to have reached refusal. The corer shall be extracted and the available core collected. Attempts to collect soil samples from the missing core depths can be made at the remaining triangle corners.
8. Core recovery will be noted for each 2-inch interval. Recovery will be qualified as good, poor, or no recovery. Poor recovery will note that an incomplete two-inch sample was recovered.
9. In the event that soil conditions do not permit the use of a soil probe coring device, samples may be collected with a shovel.
10. Thatch and other plant residue shall be removed prior to collecting a sample with a shovel.
11. When a shovel is used for collection the following procedure shall be followed:
    a. At each sub-sampling location, dig a hole at least 6 inches deep.
    b. During excavation, material from zero to 2 inches should be placed in a bag appropriately labeled for the depth. Then the material from 2 to 4 inches should be placed in a separate bag. And finally the material from 4 to 6 inches should be placed in a separate bag.
    c. Material from each depth interval may be placed on a plastic sheet to facility sample collection.

When possible, representative soil samples collected from the field will be documented with a time and date stamped photograph.

4.4 Handling of Samples

All individual samples from each sub-location will be placed in individual plastic bags. The sample number will be placed on the outside of the sample bag. Each sampling area (up to four sampling areas per LAL) will have 60 individual samples (20 sub-sample locations x 3 sample depths = 60 samples). For each subarea, the sample bags from each depth will be segregated and placed in a larger resealable reinforced plastic bag (typically one-gallon freezer bags). For example, all zero to 2-inch samples within sub-location "A" will be placed in the same one gallon resealable plastic bag. Soil samples from one LAL or CL will then be placed within one large plastic bag which will be sealed before it is placed in an insulated container (cooler). All samples will be shipped to the soil/litter processing laboratory for compositing.

Compositing of samples will be performed at the soil processing laboratory.

4.5 Field QA/QC Samples (Soils)

1. Field Duplicate Samples may be created at the soil processing lab.
2. Blind Standard: A blind standard of a certified reference soil may be sent to the analytical lab for every 50 samples send to the analytical laboratory. The blind standard will be sent by the soil processing lab.
3. Decontamination Blank: a sample of the final decontamination rinsate may be collected and forwarded to the soil processing laboratory for analysis at a frequency of one decon rinsate collected after sampling is completed at a facility or at a rate of one per 20 decontamination events . The decon blank will be generated in the field using laboratory grade distilled water.

4.6 Decontamination Procedures

Full decontamination will occur between every LAL property, or upon exit of a growers' field onto a public right-of-way. A decontamination station will be established and maintained at the boundary of the grower's property and the public right-of-way, unless a location has otherwise been designated by the grower or integrator.

Full decontamination steps will be as follows:

1. Bagged samples will be placed into a receiving bag held by the members of the decon team.
2. All electronic equipment will be transferred from the resealable plastic bags carrying the electronic equipment into the field into a plastic bag held by a member of the decon team.
3. All reusable tools will be decontaminated by removing all soil or other material by brushing/scraping the equipment. The equipment will then be washed with a phosphate free soap solution. This will be followed by a rinse a 6 or 10 percent bleach solution and then with distilled water.
4. All disposable PPE equipment such as gloves, coveralls, boot covers, etc. will be removed and disposed into a plastic trash bag held by the decon team. The trash bag will be placed into a second trash bag and tied shut.
5. The rubber boots worn by the field crews will then be decontaminated using the same procedures used to decontaminate the reusable tools. Upon decontamination of the rubber footwear, the field crew members may leave the field.
6. Any vehicles driven onto the LAL fields will be driven through the decon line with the front tires brushed to remove soil and other material, sprayed and brushed with a phosphate free detergent solution, and then sprayed with a bleach solution. Once the front tires and wheel wells have been decontaminated, the rear tires will be addressed using the same procedure before the vehicle enters the public right-of-way.

Decontamination between subareas within an LAL and not requiring Full Decontamination procedures will consist of removing soil material from the corer barrel and the knife or implement used to cut the soil samples prior to collection of the first soil sample from the next LAL subarea.

After discussion with Oklahoma Department of Agriculture, Food, and Forestry personnel, it was determined that the decontamination water and solutions will be considered *de minimus* material and will be disposed of on the ground on the right-of-way leading into the facility.

5.0 Litter/Manure Sampling

5.1 General Conditions

1. All litter/manure samples will be collected with litter/manure in place within Poultry Houses.

2. Litter/manure may be sampled at any time regardless of weather conditions.
3. More than one Poultry House may be sampled at a Farm/Facility. The litter/manure from each house will be maintained as a separate sample.
4. Bio-Security Protocol dictated by the Oklahoma Department of Agriculture, Food, and Forestry, and as supplemented by individual integrators and/or growers will be followed at all times.
5. The sampling team will consist of three individuals. One individual will enter the Poultry House and collect the samples. A second individual will accompany the first individual onto the property but will only video tape the first individual from a vantage point generally outside of the Poultry House. The third individual will maintain their position at a decontamination station anticipated to be at the public right-of-way entrance to the grower's property.
6. The individual responsible for the video taping will relay house entry times, house exit times, start of compositing times, and completion of sample compositing times to a third individual located at the public right of way entry to the grower's property via radio communications. The third individual will enter those times into the field book.
7. Prior to entry onto the grower's property, a decontamination/ sample handling station will be established on the public right-of-way adjacent to the grower's property, or on the grower's property if an adequate location is identified by the grower.

5.2 Location and Distribution of Poultry House Sub-Sample Collection Points

1. Broiler or Pullet Houses
   a. Sub-samples are collected from approximately 1/3 house-width zones.
   b. Approximately six samples are collected from each zone.
   c. Sub-samples should be located so as to obtain two samples from around the waters, feeders and walls on each side of the house.
   d. Depending upon the size of the poultry house, sub-samples are estimated to be spaced at 20 to 25 pace intervals within each 1/3 zone.

e. Sub-sampling locations alternate between the "sides" of each zone (i.e. a "zig-zag" pattern is traversed between sampling locations within a zone).

f. Sub-samples collected from adjacent zones should not be immediately adjacent.

2. Breeder Houses (partially slatted)

   a. Sub-samples will be collected from both slatted and litter areas.

   b. Twenty (20) sub-samples will be collected

   c. Sub-samples will proportionally represent the relative aerial proportion of slatted and litter areas; for example if 2/3 of the house is under slats, and 1/3 is litter area, 14 litter/manure samples should be collected from under the slats and 7 litter/manure samples should be collected from the litter area.

   d. Sub-samples taken beneath slats will be as fully penetrating of the manure as possible and will be distributed so as to obtain a representative sample of the entire slatted area.

   e. Sub-samples from litter areas will be collected in the same manner (i.e. "zig-zag" pattern) as used for broiler or pullet houses.

3. Other Circumstances

   a. Sampling of litter/manure within a Poultry House for circumstances and conditions other than those described for Broiler, Pullet or Breeder Houses will be conducted so as to obtain a representative sample of the litter/manure within that Poultry House.

   b. The circumstances or conditions requiring a variation from the sampling protocol described for Broiler, Pullet or Breeder Houses will be documented in the Sampling Log Book.

   c. A description of the method(s) and procedures used to collect a representative sample of the litter/manure within a Poultry House in which the sampling protocol for Broiler, Pullet or Breeder Houses cannot be followed will be documented in the Sampling Log Book.

   d. The method(s) and procedures used to collect a representative sample of the litter/manure within a Poultry House in which the sampling protocol for Broiler, Pullet or Breeder Houses cannot be

followed will follow the principles embodied in the reference materials.

e. All sub-samples will be collected with an appropriate solid manure sampling device.

f. All samples from litter areas will be collected through the full thickness (surface to base) of the litter/manure.

g. All samples from slatted areas will, to the extent possible, be collected through the full thickness (surface to base) of the litter/manure.

h. Immediately after collection, all sub-samples will be placed in a plastic bag contained inside a 5-gallon plastic bucket.

i. For partially slatted houses, sub-samples from slatted and litter areas will be composited together.

4. Container

a. During sample collection, all samples will be placed into a 5-gallon bucket double-lined with plastic bags.

b. After sample collection, the material within the 5-gallon bucket will be manually mixed using either a clean hand trowel and/or the shovel used to collect the samples inside the poultry house.

c. The rough mixing/compositing will be accomplished by breaking the cake material and turning over, to the extent possible, the entire contents of the bucket without damaging the plastic bag liners.

d. The rough mixing/compositing will be conducted immediately outside the poultry house and immediately after sample collection.

e. After mixing, a small subsample (500 ml in volume) will be removed via hand trowel and placed into a sterile plastic bottle or whirl pack, which will be immediately sealed and labeled with the Sample ID.

f. The remaining material within the plastic bags lining the 5-gallon bucket will be tied shut. At the decon station, the plastic bags will be placed into another appropriately sized plastic bag which will be tied shut, sealed with duct tape, and the sample ID written on the duct tape with an indelible marker.

g. The small subsample container will also be placed into a resealable plastic container at the decon station. This second container will also be labeled with the sample ID and date using an indelible ink marker.

h. The samples will then be placed in an appropriately-sized cooler with ice that will also be double bagged in plastic bags and sealed shut.

i. The appropriate chain-of-custody will be placed inside each cooler and the cooler sealed with tape and a chain of custody label.

j. The cooler with the 500 ml volume sample will be shipped overnight directly to the EML Lab for bacteria analyses.

k. The cooler with the remaining litter sample will be shipped overnight to the soil/litter processing lab.

5. Exiting the Property

a. Once the field team has sampled a Poultry House, the team will approach the decontamination station maintained at the boundary of the grower's property and the public right-of-way, unless a location has otherwise been designated by the grower or integrator.

b. Samples will be handed across the decon station line into clean receiving bags as noted above.

c. All electronic equipment will be passed from the sampling team into a resealable plastic bag held by the decon team member. The electronic equipment will be wiped down with an antibacterial wipe followed by a cloth moistened with dionized water.

d. Sample trowels, shovels, and empty collection bucket will be offered to the grower. If the grower does not want these tools, they will be included with the protective coveralls and gloves to be discarded to a sanitary landfill or a municipal incinerator.

e. All disposable Personal Protective Equipment (PPE) to be disposed will placed into double bagged plastic bags held by the decon team member. These bags will be disposed at a dumpster serviced by a municipality that either disposes of the trash at a sanitary landfill or by incineration.

f. The rubber boots of the sampling team will be subject to decontamination by a phosphate free detergent rinse, followed by a bleach solution rinse, followed by a tap water rinse.

g. Once the boots have been decontaminated, the sampling team may cross the decontamination line onto the public right-of-way.

5.5 Field QA/QC Samples (Manure/Litter)

1. Field Duplicate Samples may be created in the soil/litter processing lab.
2. Decontamination Blank (created in the field): a sample of the final decontamination rinsate may be collected and forwarded to the processing lab to send to the analytical lab for analysis at a frequency of one decon rinsate for every facility. A decon rinsate would only be generated in the event that sampling equipment were to be reused. Currently, the plan is that all sampling equipment for manure/litter sampling is disposed after a single use.

6.0 Identification of Samples

Identifying information to be recorded on the sample label for soil samples:

1. Alphanumeric identification of the LAL or CL: LAL1 – LAL24, CL1 – CL8. The log book will be used to record the farm and location of each LAL or CL.
2. Alphanumeric identification of the Sampling Area: A – D
3. Alphanumeric identification of the Sub-sample location: 1 - 20
4. Alphanumeric identification of the depth of collection (i.e. –2, -4, -6)
5. The following sample number is an example of the soil sample taken from LAL field number 5, sampling area B, sub-sample location 18, and a depth of 2 inches:

   LAL5-B-18-2

6. For samples submitted to the analytical lab, additional alphanumeric identification of the type of sample will be added to the end of the identification number:

   a. A= laboratory sample

   b. B = laboratory duplicate

   c. C = reference soil (standard)

d. D = decontamination blank (added to field samples)

e. E = laboratory QA/QC (extra volume)

7. Date of sample collection (only on chain-of-custody),
8. Time of sample collection (only on chain-of-custody),
9. Initials of the person collecting the sample (only on chain-of-custody).

6.1 Identifying information to be recorded on the sample label for litter/manure samples:

1. Alphanumeric identification of the Facility: FAC1 – FAC8.
2. Alphanumeric identification of the Poultry House: A – C
3. The following sample number is an example of the litter sample taken from facility number 5 and poultry house B:

   FAC5-B

4. Samples sent to the analytical laboratory will have alphanumeric identification of the type of sample added to the end of the number:

   a. A= laboratory sample

   b. B = laboratory duplicate

   c. C = reference soil (standard)

   d. D = decontamination blank (added in the field)

   e. E = laboratory QA/QC (extra volume)

5. Date of sample collection (only on chain-of-custody),
6. Time of sample collection (only on chain-of-custody),
7. Initials of the person collecting the sample (only on chain-of-custody).

7.0 Shipment of Samples to the soil/litter processing laboratory and to the analytical laboratory

1. Once placed in sampling containers (plastic bags or jars), samples will be placed on ice (double bagged and sealed in plastic bags) within insulated protective containers.
2. If possible, samples will be shipped immediately via overnight shipment to the analytical laboratory.

3. In no event, will FAC samples be held more than 24 hours before shipment. Depending upon circumstances, LAL samples may be held as much as 48 hours before shipment.
4. Samples will be sent to the laboratory under a Chain-of -Custody.
5. A custody seal will be placed on the outside of the container across the area between the lid and the container. The custody seal will be signed.
6. The Chain-of-Custody will be sealed in a plastic bag and placed within the insulated protective container holding those samples to which it refers.
7. Samples shipped to the EML laboratory will be shipped to the following address:

   Environmental Microbiology Laboratory
   1150 Bayhill Drive, Suite 100
   San Bruno, CA 94066
   Contact: Cole Mackelprang, 858-268-2762
   e-mail: cmackelprang@emlab.com
   Contact: Megan S. Tatreau, 858-268-2770

8. Samples shipped to the CDM Prep laboratory will be shipped to the following address:

   CDM
   2714 Walnut Street
   Denver, CO 80205
   Contact: Todd Burgesser, 303-298-1311
   e-mail: burgesserte@cdm.com

8.0 Analytical

8.1 Laboratory

The laboratory conducting the analyses will be experienced in conducting the specified analyses and will have certifications to conduct the specified analyses.

All analyses and sample preparation will be conducted using accepted and published protocols and/or methods.

8.2 Analytical Protocols

Soil samples will be analyzed for one of two sets of parameters (short list or long list). Table 1 provides the parameters and analytical methods for the short and Table 2 provides the parameters and analytical methods for the long list.

Litter samples will be analyzed for Table 2 parameters.

Table 1: Short List Parameters – Soil

Parameter	Method
Moisture content (%)	Gravimetric (105C)
Organic matter	Walkley-Black (Modified)
Soil pH	Water 1:1
Soil Conductivity	Water 1:2
Total Nitrogen	Kjeldahl, modified
Total Aluminum (Al)	EPA SW-3050/6020
Total Phosphorus (P)	EPA SW-3050/6020
Total Arsenic (As)	EPA SW-3050/6020
Total Copper (Cu)	EPA SW-3050/6020
Total Zinc (Zn)	EPA SW-3050/6020

Table 2: Long List Parameters – Manure and Soil

Parameter	Method
Moisture content (%)	Gravimetric (105C)
Organic matter	Walkley-Black (Modified)
Texture (% sand, silt and clay)	Hydrometer ASTM-D422
Soil pH	Water 1:1
Soil Conductivity	Water 1:2
Total Phosphorus (P)	EPA SW-3050/6020
Mehlich-III Phosphorus (Mehlich-III P)	Mehlich III (ICP)
Soluble Phosphorus	Water 1:10, Bull.396, pg 17
Soluble nitrate	Water 1:10
Total Nitrogen	Kjeldahl, modified
Soluble ammonium	Water 1:10
Soluble sulfate	Water 1:10
Soluble chloride	Water 1:10
TAL Metals	EPA SW-3050/6020
Total Molybdenum (Mo)	EPA SW-3050/6020

Bacteria:	
Total coliform	SM-9221B
Enterococcus	SM-9230B
Fecal coliform	SM-9221E
c-coli	SM-9221F
Staphylococcus	BAM 12
Campylobacter	BAM-Chap. 7
Salmonella	BAM 5
17β-estradiol, estrone, estriol	LC-MS-MS

8.3 Data Reporting

1. Data from the laboratory shall be reported in both electronic and paper reports.
2. Data reports shall include all quality control data generated, including results for duplicates, blanks and spikes, as applicable. If applicable, a level 3 data quality report will be provided by the laboratory.
3. Data reports shall include a copy of the Chain of Custody accompanying each set of samples submitted

9.0 Bio-security, Decontamination of Equipment and Personal Protective Equipment

All persons engaged in sampling, observing sampling or documenting sampling under this protocol shall follow appropriate bio-security precautions. All persons doing sampling will receive bio-security training from the State of Oklahoma.

9.1 Soils

To the extent possible, disposable sampling equipment should be used.

All reusable sampling equipment shall be decontaminated using a non-phosphate detergent, a 6% (minimum) bleach solution, and three de-ionized water rinses between Sampling Areas.

9.2 Litter/Manure

To the extent possible, disposable sampling equipment should be used.

All reusable sampling equipment shall be decontaminated using a non-phosphate detergent, a 6% (minimum) bleach solution, and three de-ionized water rinses between poultry houses.

9.3 Health and Safety Plan:

A health and safety plan that is specific to this sampling protocol will be prepared and reviewed by all samplers.

10.0 References

Zhang, H. and Johnson, G. 2003. How to get a good soil sample. Oklahoma State University Cooperative Extension Service Fact Sheet F-2207. Available at http://osuextra.okstate.edu/pdfs/F-2207web.pdf

Zhang, H., Hamilton, D. W. and Britton, J. G. 2002. Sampling Animal Manure. Oklahoma State University Cooperative Extension Service Fact Sheet F-2248. Available at http://osuextra.okstate.edu/pdfs/F-2248web.pdf

Eucha/Spavinaw Watershed Management Team. Undated. Soil Sampling Protocol.

Eucha/Spavinaw Watershed Management Team. Undated. Steps for Pulling Litter Samples.

11.0 Revised Dates*

The following are other revision dates applicable to this SOP.

Revision 8 – February 5, 2007
Revision 7 – April 24, 2006
Revision 6 – May 11, 2005
Revision 5 – April 20, 2005
Revision 4 – March 25, 2005
Revision 2, 3 – March 16, 2005
Revision 1 – January 25, 2005

Prepared: Todd Burgesser Review: Kim Zilis

Approved: Roger L. Olsen Date Approved: 2/06/07

1.0 Overview and Application

This standard operating procedure (SOP) describes field procedures used for compositing of soil and poultry litter samples from the Illinois River watershed of eastern Oklahoma and western Arkansas. This will include handling, mixing, and shipment of soil and litter samples.

2.0 Handling and Compositing of Soil and Litter Samples

All individual soil samples from each sub-location will be placed in individual plastic bags (double bagged), packed in a cooler with blue ice and shipped over night under chain-of-custody to the CDM processing laboratory in Denver, Colorado. The sample number will be located between the inner and outer plastic bag. Each sampling area (up to four sampling areas per LAL) will have 60 individual samples (20 sub-sample locations x 3 sample depths = 60 samples). All samples will be received by the CDM processing laboratory for compositing. Each of the 20 sub-samples will be composited into one homogeneous sample using the protocol described below.

Litter samples will be received by the CDM processing laboratory under chain-of custody in a 5-gallon bucket. The litter sample will be contained in a plastic bag inside of the 5-gallon bucket will be closed with a tie. A unique sample number will be written on the outside of the bucket.

Upon receipt of the samples, the cooler/bucket temperature will be measured using a NIST traceable thermometer. The samples soil will then be removed from the cooler and checked against the chain-of-custody to ensure that all samples have been received.

The twenty sub-samples associated with the individual sample depths or the entire litter sample will be poured into a stainless steel bowl or 2.5-gallon bucket ready for mixing. All equipment will be decontaminated/sterilized with laboratory grade distilled water and 10 percent bleach (see procedure below).

2.1 Mixing of Soil Samples

- All health and safety protocol will be followed as described in the Health and Safety Plan for the Illinois River Basin Project. This is includes wearing nitrile gloves and processing soil in the hood.
- All feathers, rocks, twigs, debris and vegetation will be removed before sieving and mixing.
- Mixing will be accomplished using a disposable, plastic sampling scoop or a decontaminated stainless steel spoon.
- All clods over 0.5 inches in diameter will be disaggregated into smaller particles by hand or the use of a decontaminated stainless steel spoon or mortar.
- If the moisture content is too high to allow homogenization or disaggregation of the particles, the sample will be placed in steel drying pan and air dried over night.
- The sample will be hand mixed using the plastic scoop or stainless steel spoon for at least five minutes or until particles are uniform in size.
- If a plastic bucket is used, the bucket will then be sealed and inverted or rotated at least 10 times.
- After mixing, the sample will be sieved to remove particles sizes of greater than 2 mm using a decontaminated US Sieve no. 10 (gravel size particles will be removed).
- Each fraction (greater than 2 mm and less than 2 mm) will be weighted. The less than 2 mm fraction will be placed in a plate grinder and reduced in size to 0.074 mm (US sieve no. 200, very fine sand).
- The ground sample will be split using a riffle splitter and sent to the various laboratories (see splitting procedure in section 1.3.1, Duplicate Samples).

2.2 Mixing of Litter Samples

The same procedure as described above for the soil will be used for the litter. However, grinding may not be necessary if the litter can be sieved directly through a US sieve no. 200.

2.3 Laboratory QA/QC Samples (Soil)

Laboratory QA/QC samples may consist of duplicate samples, decontamination blanks, and blind standards. The following describes each type of QA/QC sample.

2.3.1 Duplicate Samples (created at the soil processing lab)

After sample mixing, sieving and grinding, two split samples will be collected. The sub-sample splits should be collected using a nonbiased riffle splitter. The sample is poured through the riffle splitter and into the decontaminated collection pans. The amount of soil or litter contained by the sample container shall be sufficient for the chemical and physical analyses to be conducted.

2.3.2 Blind Standards

A blind standard of a certified reference soil will be sent to the analytical laboratory for approximately every 50 samples send to the laboratory. The blind standard will be sent by the CDM soil processing lab. Blind standards will be for metals, arsenic, and phosphorus.

2.3.3 Decontamination Blanks

A sample of the final decontamination rinsate will be collected and forwarded to the analytical laboratory for analysis. The decontamination rinsate blank will be generated in the CDM processing laboratory using a final rinse of laboratory grade distilled water. All parameters will be analyzed.

3.0 Shipment of Samples to the Analytical Laboratory

- Once placed in sampling containers (plastic bags or jars), samples will be held at 4° C on blue ice (sealed in plastic bags) within insulated protective containers.
- If possible, samples will be shipped immediately after compositing via overnight shipment to the analytical laboratory.
- After compositing, samples should not be held more than 24 hours before shipment.
- Samples will be sent to the laboratory under a Chain-of -Custody.
- A custody seal will be place on the outside of the cooler between the lid and the body of the cooler. The custody seal will be signed.
- The Chain-of-Custody will be sealed in a plastic bag and placed within the insulated protective container holding those samples to which it refers.

4.0 Decontamination of Processing Equipment

All nondisposable equipment (bowls, sieves, spoons, and grinders) will be decontaminated/sterilized after each composite sample is created. Decontamination will include washing with phosphate free water followed by rinsing with laboratory grade distilled water. A final rinse of 10 percent bleach will be performed. The equipment will be air dried.

5.0 List of Analytes and Bottle Requirements

5.1 Analytical Parameters

Soil samples will be analyzed for one of two sets of parameters (short list or long list). Table 1 provides the parameters and analytical methods for the short list and Table 2 provides the parameters and analytical methods for the long list.

Litter samples will be analyzed for Table 2 parameters.

Table 1: Short List Parameters - Soil

Parameter	Method
Moisture content (%)	Gravimetric (105C)
Organic matter	Walkley-Black (Modified)
Soil pH	Water 1:1
Soil Conductivity	Water 1:2
Total Nitrogen	Kjeldahl, modified
Total Aluminum (Al)	EPA SW-3050/6010/6020
Total Phosphorous (P)	EPA SW-3050/6010/6020
Total Arsenic (As)	EPA SW-3050/6010/6020
Total Copper (Cu)	EPA SW-3050/6010/6020
Total Zinc (Zn)	EPA SW-3050/6010/6020

Table 2: Long List Parameters – Manure and Soil

Parameter	Method
Moisture content (%)	Gravimetric (105C)
Organic matter	Walkley-Black (Modified)
Texture (% sand, silt and clay)*	Hydrometer ASTM-D422
Soil pH	Water 1:1
Soil Conductivity	Water 1:2
Total Phosphorous (P)	EPA SW-3050/6020
Mehlich-III Phosphorous (Mehlich-III P)	Mehlich III (ICP)

Soluble Phosphorous	Water 1:10, Bull.396, pg 17
Soluble nitrate	Water 1:10
Total Nitrogen	Kjeldahl, modified
Soluble ammonium	Water 1:10
Soluble sulfate	Water 1:10
Soluble chloride	Water 1:10
TAL Metals	EPA SW-3050/6020
Total Molybdenum (Mo)	EPA SW-3050/6020
Bacteria:	
Total coliform	SM-9221B
enterococcus	SM-9230B
Fecal coliform	SM-9221E
e-coli	SM-9221F
staphylococcus	BAM12
campylobacter	BAM7
salmonella	BAM5
17β-estradiol, estrone, estriol	LC-MS-MS

*split before sieving and grinding

5.2 Bottle Requirements

Soil samples will be analyzed for one of two sets of parameters (short list or long list). Table 3 provides the parameters, bottle requirement and laboratory for the short list and Table 4 provides the parameters, bottle requirement and laboratory for the long list.

Litter samples will be analyzed for Table 4 parameters.

Table 3: Short List Parameters - Soil

Parameter	Bottle	Laboratory
Moisture content (%)	1 quart glass	A&L
Organic matter	1 quart glass	A&L
Soil pH	1 quart glass	A&L
Soil Conductivity	1 quart glass	A&L
Total Nitrogen	1 quart glass	A&L
Total Aluminum (Al)	1 quart glass	A&L
Total Phosphorous (P)	1 quart glass	A&L
Total Arsenic (As)	1 quart glass	A&L
Total Copper (Cu)	1 quart glass	A&L
Total Zinc (Zn)	1 quart glass	A&L

Note: 1 bottle for all of the above analysis

Table 4: Long List Parameters – Manure and Soil

Parameter	Bottle	Laboratory
Moisture content (%)	1 quart glass	A&L
Organic matter	1 quart glass	A&L
Texture (% sand, silt and clay)*	1 quart glass (separate from the other bottles)	A&L
Soil pH	1 quart glass	A&L
Soil Conductivity	1 quart glass	A&L
Total Phosphorous (P)	1 quart glass	A&L
Mehlich-III Phosphorous (Mehlich-III P)	1 quart glass	A&L
Soluble Phosphorous	1 quart glass	A&L
Soluble nitrate	1 quart glass	A&L
Total Nitrogen	1 quart glass	A&L
Soluble ammonium	1 quart glass	A&L
Soluble sulfate	1 quart glass	A&L
Soluble chloride	1 quart glass	A&L
TAL Metals	1 quart glass	A&L
Total Molybdenum (Mo)	1 quart glass	A&L
Bacteria	1 - 250 mL plastic (sterilized) or 1-8 oz. Whirl bag	EML
PCR	1-8 oz. Whirl bag	ISU
17β-estradiol, estrone, estriol	1 – 4oz. glass	GEL

*split before sieving and grinding

6.0 Analytical Laboratories

Bottles for estrogen metabolites (all samples) will be shipped to:

General Engineering Laboratories, LLC
201 Pine Ridge Road, Unit 5
Golden, CO 80403
Contact: Paul Winkler, 720-253-3093
Paul.winkler@gel.com

Bottles for nutrients, metals, etc (all samples) will be shipped to:

A&L Analytical Laboratories, Inc.
2790 Whitten Rd.
Memphis, TN 38133
Contact: Scott McKee, 800-264-4522
smckee@allabs.com

Bottles for bacteria analyses from soil and litter will be shipped to:

Environmental Microbiology Laboratory
1150 Bayhill Drive, Suite 100
San Bruno, CA 94066
Contact: Megan S. Tatreau, 858-268-2770
mtatreau@emlab.com

Bottles for PCR will be shipped to:

Idaho State University
Department of Biological Sciences-MRCF
Attn: Erin O'Leary-Jepsen
640 Memorial Drive
Pocatello, ID 83209-8007
Contact: Erin O'Leary-Jepsen, 208-282-4890

7.0 Documentation

Bound laboratory logbooks should be used for the maintenance of field records. All aspects of sample compositing and handling as well as visual observations will be documented in the field logbooks. Supplemental information may be documented on the field data sheets provided. All entries in laboratory logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.

8.0 Revised Dates*

The following are other revision dates applicable to this SOP.

Revision 2: 02/09/06

Revision 1: 05/10/05

Prepared: Darren L. Brown Review: Roger Olsen

Approved: [signature] Date Approved: 2-06-07

1.0 Overview and Application

This standard operating procedure (SOP) describes field procedures used for collection of fecal matter for identifying the types and abundance of bacterial DNA. The bacterial DNA is first amplified by polymerase chain reaction (PCR), then digested with a restriction enzyme. The enzyme cuts DNA strands into different size fragments whose length is dependent upon the DNA sequence, and the last (terminal) fragment is labeled for detection. Each terminal fragment length is represents approximately one bacterial species. This program is designed to identify DNA fragments from bacteria that reside in fecal material from various animals, including cattle, swine, ducks, geese and humans.

2.0 Selection of Sampling Locations

Sample locations will be selected from farms, wildlife areas, septic clean-out trucks, or wastewater treatment plants as appropriate. The following sources of fecal matter will be targeted for collection.

1. A total of 10 fields where beef cattle are actively grazing; preferably five fields within the basin and five fields outside the basin,
2. A total of 2 dairy cattle milking barns; preferably in the basin, but could be outside of the basin (close to the basin as possible),
3. A total of 2 swine facilities; preferably in the basin, but could be outside of the basin (close to the basin as possible),
4. A total of five active geese landing areas; preferably in the basin, but could be outside of the basin (close to the basin as possible),
5. A total of five active duck landing areas; preferably in the basin, but could be outside of the basin (close to the basin as possible),
6. A total of three septic clean out trucks; preferably all in the basin, but at a minimum at least one sample in the basin,
7. A total of three small wastewater treatment plan influent locations; preferably all in the basin, but at a minimum at least one in the basin.

The locations should contain the following information for each associated Farm/Facility:

1. Name of Farm/Facility owner and Farm/Facility contact person,
2. Physical address and location (section-township-range) of Facility,
3. Contact address of Farm/Facility owner or Farm/Facility contact person,
4. Contact phone number of Farm/Facility owner or Farm/Facility contact person,
5. Whether or not one or more samples can be accessed at the Farm/Facility,
6. The physical location of each sample collection site(s) - record coordinates (latitude and longitude) of documented location (eg, corner of a field),
7. Estimate of number of animals at sample collection site or number of facilities serviced by wastewater treatment plant or septic clean out truck,
8. Estimate of the amount of feces available at the sampling site,
9. Estimate of when the feces was deposited; e.g., was the animal observed while it was defecating,
10. Observation as to whether any chicken litter application has occurred at the sampling field/site,
11. Estimates of amount, rate, and date of litter treatment applied to the site, if applicable, and information as to amount, rate and dates of application.

Site selections will be made based upon availability.

3.0 Sampling Documentation

3.1 Sampling Log Book and Sampling Forms

1. A Sampling Log Book and Sampling Forms shall be maintained.
2. Pages in the Sampling Log Book will reference specific sampling forms by use of the Sample Identification.
3. The Sampling Log Book shall be bound and shall be constructed of waterproof paper.
4. Entries in the Sampling Log Book or on the sampling form shall be made in black permanent ink.
5. Each page of the Sampling Log Book shall be dated.
6. The preparer shall initial each page of the Sampling Log book.

7. For each location sampled, the following information shall be recorded in the Sampling Log Book or on the sampling forms:
    a. Name, address and phone number of the Property/Facility owner,
    b. Identification of the Property/Facility (MAN),
    c. Name, address and phone number of the Property/Facility operator,
    d. If applicable, name, address and phone number of the Integrator responsible for the Property/Facility,
    e. If applicable, the amounts, rates and dates of prior litter/manure applications to specific fields at the Property/Facility (confirm State Reports),
    f. If applicable, the existence of prior soil sampling data for the property (yes or no),
    g. The water supply for the Property/Facility,
    h. The legal description (qtr-qtr-qtr-sec-twp-rng) of the property related to the Property/Facility,
    i. Information as to any fertilizers, chemicals or soil amendments added during the last five years,
    j. Specific information listed within this protocol,
    k. Sketch map of each property/facility with approximate dimensions; indicate local features on the sketch (vegetation, water bodies, adjacent fields, location of poultry houses, roads, old fence rows, livestock feeding areas, livestock grazing areas, etc); dimensions and features can also be placed on the aerial photographs,
    l. Land slope of property/facility,
    m. Distance to nearest water body,
    n. Notes on weather (temperature, wind, last precipitation event, etc),
    o. Type of vegetation currently on the LAL, if any, and any known vegetation grown in past 5 years,

p. Use of adjacent fields, and;

q. Other information as appropriate or relevant.

3.2 Photographic Record

A photographic record shall be made and maintained for all sampling activities on the MAN. All photographs made shall be time and date stamped.

3.3 Chain-of-Custody

A Chain-of-Custody shall be prepared for each set of samples transferred to the analytical laboratory, North Wind, Inc. in Idaho Falls, ID (see section 7).

The Chain-of-Custody shall, at a minimum, contain the following information:

1. The project name, ***Illinois River Watershed Manure DNA Sampling,***
2. Name of person or entity collecting samples,
3. Signature blocks with dates and times for all persons having custody (sampler, shipper, processing laboratory, etc),
4. For each sample related to a Chain-of-Custody:
    a. The unique numeric identifier on the submitted sample container/bag (see subsequent section 6)
    b. The date and time the sample was collected,
    c. The sample "matrix" (Manure).

4.0 Manure Sampling

4.1 Manure Locations (MAN)

4.1.1 Permissible Manure and Weather Conditions

1. Manure must be fresh. Sample should be from the interior of manure piles.
2. Manure should not be sampled during precipitation events.

4.1.2 Beef Cattle Sampling Areas

Manure samples will be collected from a total of ten fields actively grazed by cattle. Five locations will be from fields within the IRW. If available, both fields with and without litter application will be sampled. Five locations will be from fields outside the IRW and, if

possible, from fields with no litter application. Two composite samples will be collected from each field. Each composite sample will consist of samples from ten fresh manure piles. In all, twenty composite samples will be collected.

4.1.3 Dairy Cattle Sampling Areas

Manure samples will be collected from the clean out slurry of four milking barns. If possible, two barns handling cattle fed by grazing and two barns handling grain-fed cattle will be sampled. The clean out slurry must consist of that day's droppings. The samples must be collected from waste stream before the collection ponds. In all, four samples will be collected.

4.1.4 Swine Sampling Areas

Manure samples will be collected from the clean out slurry from two swine facilities. The clean out slurry must consist of that day's droppings. The samples must be collected from waste stream before the collection ponds. In all, two samples will be collected.

4.1.5 Duck Sampling Areas

Manure samples will be collected from up to five landing or residence areas. Sampling locations will be from wildlife areas, golf courses, or local ponds. Two composites will be collected from each landing/residence area. Composites will consist of ten swabs or direct fecal samples each, if possible. In all, ten samples will be collected.

4.1.6 Geese Sampling Areas

Manure samples will be collected from up to five landing or residence areas. Sampling locations will be from wildlife areas, golf courses, or local ponds. Two composites will be collected from each landing/residence area. The locations may be co-located with the duck locations; however, the samples have to be distinctly separate between species. Composites will consist of ten fecal samples each, if possible. In all, ten samples will be collected.

4.1.7 Human Waste Samples

Human sewage samples will be collected at two sources: septic clean out trucks and influent to wastewater treatment plants. Sewage samples will be collected from three separate septic clean out trucks. The samples should be collected at the pump out facility after at least several homes have been visited. The sample should be collected after the pumping has been in progress and the waste in probably mixed.

Sewage samples will be collected from the plant influent at three different wastewater treatment plants. The plant operator will determine the best way to collect a representative influent sample which has not been subject to treatment. Wastewater treatment plants will be selected that do not have contribution from industries which could contribute poultry or other animal waste products (i.e. processing plants).

In all, six human waste samples will be collected.

4.2 Collection and Handling of Samples

Sampling personnel will wear disposable, sterile gloves at all times when collecting fecal samples and will change gloves before they collect each new fecal sample. Samples will either be pre-composited samples (i.e. dairy cattle, swine, and human samples) or will be composited in the field (beef cattle, duck, and geese). All samples will be collected into 20 milliliter, sterilized, polystyrene, round bottom tubes. Each tube will contain 10 mL of 20 % glycerol solution (added to the tube by the laboratory). Pre-composited samples will be collected directly into the tubes (approximately 2 - 10 grams). For the samples to be composited in the field, ten aliquots will be sampled using a sterilized, disposable, polystyrene spatula. A similar sized sample (1 -2 grams) from each individual stool will be placed into one tube. The contents will then be mixed in the field by shaking the tube containing the glycerol/waste mixture. If swabs (sterile, cotton-tipped applicators) are used to collect duck feces, all the swab tips (ten) will be placed into the same round bottom tube. The tips will be cut from the attached plastic tube (or stick) using scissors (sterilized by cleaning with an alcohol wipe before use). Labels will be placed on the tubes and secured with transparent tape. The tubes will be placed inside individual resealable plastic bags. The bags will be placed in a cooler containing dry ice before leaving the property/facility where the sample was collected. The samples must be frozen prior to being shipped to the analytical laboratory. If the samples have not been frozen by exposure to the dry ice, they shall be placed in a freezer until freezing is complete. Samples will remain frozen until immediately prior to shipping. Samples shall be placed in a cooler with standard ice and shipped priority overnight to the analytical laboratory.

4.3 Field QA/QC Samples (Manure)

1. Duplicates: no field duplicate samples will be created since samples will be composite samples.
2. Blind Standard: no blind standards will be submitted for this particular program.
3. Decontamination Blank: no decontamination blanks will be generated for this particular program as all collection equipment will not be reused between samples.
4. Field Blanks: field blanks will be collected at a rate of one per twenty or per sample shipment. Field blanks will be collected by one of three methods.
    a. Dairy Cattle, Swine, and Humans - one field blank associated with one of these locations will be collected by opening the screw top cap and immediately replacing the cap. The tube will contain the glycerol from the laboratory.

b. Beef Cattle and Geese – one field blank associated with one of these locations will be collected by opening a packet containing a sterilized collection spatula and placing it directly into the screw cap tube containing the glycerol.

c. Duck – one field blank associated with one of these locations will be collected by placing a swab tip directly into the screw cap tube containing the glycerol.

4.4 Decontamination Procedures

Sampling equipment will be one time use. No equipment decontamination is anticipated. Only the scissors will be reused and these will be cleaned with an alcohol wipe between sampling sites.

If appropriate, bio-security decontamination measures will be implemented. All waste generated during the sampling procedure will be placed in disposable trash bag and placed in a container where the waste will be transported to a sanitary landfill.

5.0 Person(s) Collecting Samples and Observing Sampling

Personnel from CDM or Lithochimeia will conduct the manure sampling from each MAN. CDM personnel will process samples, chain-of-custody, coordinate shipping, etc.

6.0 Identification of Samples

Identifying information to be recorded on the sample label for DNA Manure samples:

1. Beef Cattle: Alphanumeric identification will consist of MAN-BC-1, MAN-BC-2 etc. The log book will be used to record the facility/property and location of each composite sample.
2. Dairy Cattle: Alphanumeric identification will consist of MAN-DC-1, MAN-DC-2 etc. The log book will be used to record the facility/property and location of each composite sample.
3. Swine: Alphanumeric identification will consist of MAN-SW-1, MAN-SW-2 etc. The log book will be used to record the facility/property and location of each composite sample.
4. Duck: Alphanumeric identification will consist of MAN-DK-1, MAN-DK-2 etc. The log book will be used to record the facility/property and location of each composite sample.
5. Geese: Alphanumeric identification will consist of MAN-GS-1, MAN-GS-2 etc. The log book will be used to record the facility/property and location of each composite sample.

6. Human: Alphanumeric identification will consist of MAN-HM-1, MAN-HM-2 etc. The log book will be used to record the facility/property and location of each composite sample.
7. If necessary, an alphanumeric identification will be assigned to a subarea if more than one sample is collected from the same facility/property: A, B, C, D etc.
8. The following sample number is an example of a manure sample taken from Beef Cattle field number 5, sampling area B:

   MAN-BC-5-B

9. For samples submitted to the analytical lab, additional alphanumeric identification of the type of sample will be added to the end of the identification number:
   a. F = Field Blank
10. Date of sample collection (only on chain-of-custody),
11. Time of sample collection (only on chain-of-custody),
12. Initials of the person collecting the sample (only on chain-of-custody).

7.0 Shipment of Samples to the analytical laboratory

1. Shipping coolers will be packed such that samples are stored with standard ice placed in double-bagged resealable plastic bags. The shipping coolers shall be insulated protective containers.
2. If possible, samples shall be shipped immediately via overnight shipment to the analytical laboratory. The laboratory address is:

   Idaho State University

   Department of Biological Sciences- MRCF
   Attn: Erin O'Leary-Jepsen

   650 Memorial Drive
   Pocatello ID 83209-8007
   208-282-4890

3. In no event, shall samples be held more than 24 hours before shipment unless they are frozen.
4. Samples shall be sent to the laboratory under a Chain-of -Custody.
5. A custody seal will be place on the outside of the container across the area between the lid and the container. The custody seal will be signed.

6. The Chain-of-Custody shall be sealed in a plastic bag and placed within the insulated protective container holding those samples to which it refers.

8.0 Analytical

8.1 Analytical Protocols

Analyses are being conducted by Tamzen W. Macbeth (208-528-8718), North Wind, Inc., 1425 Higham St., Idaho Falls, ID 83402. Analytical protocols are provided in a separate document.

8.3 Data Reporting

1. Data from the laboratory shall be reported in both electronic and paper reports.
2. Data reports shall include all quality control data generated, including results for duplicates, blanks and spikes, as applicable.
3. Data reports shall include a copy of the Chain of Custody accompanying each set of samples submitted

9.0 Bio-security, Decontamination of Equipment and Personal Protective Equipment

All persons engaged in sampling, observing sampling or documenting sampling under this protocol shall follow appropriate bio-security precautions.

9.1 Manure

To the extent possible, disposable sampling equipment should be used.

Any reusable sampling equipment shall be decontaminated using a non-phosphate detergent, bleach and three de-ionized water rinses between Sampling Areas. No reusable equipment is currently anticipated.

9.2 Health and Safety Plan:

The overall health and safety plan for the project will be used for this sampling protocol and will be reviewed by all samplers.

10.0 Revised Dates*

The following revision dates are applicable to this SOP:

Revision 1 -July 11, 2006

Prepared: Brian Bennett Review: Ronald French

Approved: Roger L Olsen Date Approved: 2/06/07

1.0 Overview and Application

This standard operating procedure (SOP) describes field procedures used for collection of surface water samples from rivers and streams in the Illinois River watershed of eastern Oklahoma and western Arkansas. This will include water chemistry, nutrients, bacteria, and other water sampling protocols. Procedures for on site measurement of various water quality parameters such as temperature, conductivity, turbidity, pH, and dissolved oxygen will also be discussed. This SOP will be used during field work and will follow the guidelines described in the *Standard Operating Procedures (SOP) for Field Sampling Efforts of the Oklahoma Water Resources Board's Beneficial Use Monitoring Program.* (ORWB, 2001).

The Illinois River watershed in eastern Oklahoma and western Arkansas has been receiving inputs from agricultural runoff, including inputs from fields where poultry waste has been applied. These wastes, along with the chemical constituents that make up the poultry wastes, have apparently impacted the water quality and sediments in the streams and lakes in the Illinois River Watershed. The purposes of the work being performed are to evaluate and document 1) the linkage and relationship, if any, between the disposal of poultry wastes and environmental contamination within the Illinois River Watershed, and; 2) the resulting harm/injury to natural resources that may have resulted from the disposal of poultry wastes within the Illinois River Watershed.

2.0 Sampling Methods Summary

Water samples of rivers and streams will be collected periodically at various locations throughout the Illinois River watershed and in several streams in surrounding watersheds. Multiple water chemistry parameters may be analyzed by qualified laboratories and on site water quality measurements may be taken. Due to the wide range of parameters being analyzed, multiple samples may be taken from each location and shipped to separate laboratories. **Table 1** describes the parameters, containers, and laboratories to be used.

For certain parameters, samples may be preserved immediately after collection. This is accomplished by placing the samples in bottles distributed by the analytical lab which already

contain the necessary types and amounts of preservatives (sulfuric acid, nitric acid, hydrochloric acid, etc.). In some cases, it may be necessary to add certain preservatives after sample collection (e.g. formalin in zooplankton samples). Some samples may be filtered prior to preservation in the appropriate sample bottles by using a peristaltic pump to force the sample water through a filter and into a beaker or flask.

2.1 Sampling Locations

Three large tributaries to Tenkiller Ferry Reservoir will be sampled on dates coinciding with reservoir sampling events. Additional water samples may be collected from each of 13 biological sampling stations during biological sampling events. Water sampling may also be conducted during base flow conditions at each of the 12 automated high flow sampling stations. Several other locations may also be sampled at various times throughout the course of this project.

2.2 Sample Types

Water samples can either be discrete or composite samples. A discrete sample is defined as a single aliquot from of a specific location or depth at a given point in time. Composites are samples composed of two or more specific discrete samples collected from one or several sampling location. This type of sample represents an average value and can, in certain instances, be used as an alternative to analyzing a number of individual discrete samples and calculating an average value. It should be noted, however, that compositing can mask the presence of contaminants by diluting isolated concentrations of an analyte that may be present in the environmental matrix.

2.3 Sampler Selection

The choice of samplers is dictated by sampling objectives, site accessibility, and parameters to be examined. Each sampling technique presents various advantages and disadvantages for its application. For example, sample disturbance, sample volume, chemical and physical reactivity between potential contaminants and sampling tool materials, and ease of decontamination vary from technique to technique.

Discrete samples will be collected using a Van Dorn water sampler or similar device. These samplers consist of a PVC tube that can be lowered into the water and then closed to collect a discrete sample. In some cases, discrete samples may be collected by a surface dip method in which the sample containers or an intermediate container such as a churn splitter are submerged and filled directly from the source water. However, sample bottles containing preservative should not be filled by the direct surface dip method so that any preservative is not lost into the water source. Composite samples can be collected by combining several discrete samples into a churn splitter and mixing prior to filling the sample containers.

2.4 Data to be Collected

At each sampling location, a variety of physical and water quality parameters may be assessed and recorded. The exact location of the sampling should be measured with a handheld GPS unit and recorded. Water quality parameters may be measured using a YSI model 650 multi-meter which should be calibrated and verified before and after each sampling event as described in Standard Operating Procedure 9.1: *Water Quality Meters*. At each sampling location the YSI meter should be lowered into the water and a reading should be recorded for each of the following parameters:

- Temperature in degrees Celsius
- pH
- Dissolved Oxygen (DO) in milligrams per liter
- Specific Conductance in micro-Siemens per cm
- Turbidity in Nephelometric Turbidity Units (NTU)

When an YSI-650 meter is not available, other electronic measurement devices may be used. Consult the devices' operation manuals for instructions on maintenance, calibration, and use.

2.5 Sample Nomenclature

In general, water samples collected from rivers and streams should be labeled according to the identification scheme: **AA-BB-CC-DD.**

AA is defined by the location type. Biological stations should use BS and high-flow stations should be denoted by HFS.

BB is defined by a two digit station identification number. In some cases, additional digits may be required.

CC is defined by a two digits which identify the sample medium; typically SW for surface water.

DD is defined by a two digit number which identifies the type of sample i.e. the original sample (01), blank (02), or a duplicate sample (03).

2.6 General Supplies

The following is a list of the minimum supplies needed for surface water sampling in streams and rivers:

- YSI 650 Multi-meters for collecting water quality parameters
- Cable for YSI 650 meter
- Van Dorn water sampler
- Churn splitter or similar mixing container
- Beakers/flasks
- Peristaltic or similar water pump
- Battery to power pump
- Filtering apparatus
- Filters (0.45 micron)
- Sample bottles with labels
- Clear tape
- Aluminum foil

- Coolers with ice
- De-ionized water
- Phosphate-free detergent
- Nitrile gloves
- Data Sheets
- Field Notebooks with water resistant paper
- Handheld GPS Unit, with extra batteries
- Writing utensils (waterproof)
- Digital camera with extra batteries
- Sunscreen
- Drinking water/snacks/lunches for the crew

3.0 Sampling Procedures

Due to the relatively large number of analyses that may be conducted throughout the course of the sampling, different numbers and types of samples may be collected during each event. Therefore, the procedures for collecting water samples may vary based on parameters to be analyzed at each location during a specific sampling event. All samples should be collected and preserved in accordance with both the OWRB guidelines and the specific analytical laboratory sampling requirements.

3.1 Sample Collection

Procedures for collecting the water samples are somewhat different depending on the type of sample desired. The following procedures should be employed.

3.1.1 Discrete Sample Collection

Discrete samples may be collected by the dip method wherever feasible, provided that the sample bottle does not contain preservatives and no filtering is necessary. This can be

accomplished by submerging the sample container below the surface and allowing it to fill with water. Care should be taken to avoid disturbing the sample by allowing air bubbles to form inside the bottle while filling. Nitrile gloves should also be worn to prevent contamination.

Discrete water samples may also be collected with the use of a Van Dorn sampler so that a sample may be collected and transferred to the appropriate sample container with a minimum of disturbance and aeration. The steps required to take a discrete water sample using a Van Dorn sampler are listed below:

- Calculate the number of samples you will be collecting at that location and prepare all the necessary bottles.
- If filtering is required, set up a decontaminated filtering apparatus and insert a new filter (wear nitrile gloves when handling the filters).
- Prepare the Van Dorn sampler for use by rinsing it and setting the spring-loaded stoppers. Make sure the messenger is ready to be deployed.
- Lower the sampler into the water and then release the messenger. This should trigger the stoppers to close, thus sealing the water inside the device.
- Bring the sampler to the surface and slowly drain the contents through the attached hose into the churn splitter.
- Keep the hose under the surface of the water in the container or direct the stream at an angle against the inside wall of the container to limit the amount of aeration that may occur.
- If necessary, filter the sample by slowly pouring it into the filtering apparatus and allowing the pump to force it through the filter.
- Carefully fill the appropriate sample bottles with the sample water.
- Store the samples in a cooler with plenty of ice.

3.1.2 Composite Samples Collection

During the main river sampling, three points equally distributed along a transect across the water body should be sampled and mixed into one composite sample. This can be done by combining a number of discrete samples collected with the Van Dorn device into a churn

splitter. Mix the samples thoroughly and pour into the sample bottles. Composite sampling may be conducted at other locations as well.

3.2 Filtering Samples

Some parameters require that the sample be filtered in the field prior to being preserved. This is accomplished with the use of a filtering apparatus, filters, and a peristaltic pump. Some types of filters need to be prepared in advance of a sampling event. Generally, this involves rinsing them with laboratory-grade, de-ionized water two times and allowing them to soak for several hours. Refer to instructions supplied with the filters or in the analytical procedure for specific details. Procedures for filtering in the field are described below:

- Set up the filtering apparatus by attaching the pump to a filtering flask and inserting the appropriate filter.
- Turn the pump on and begin slowly adding the sample water to the filtering apparatus. Again, try not to aerate or disturb the water.
- Allow the water to move through the filter and into the flask until the required amount of sample water has accumulated in the flask. In some cases, the filter may need to be changed before the necessary amount can be filtered for that sample.
- Pour the filtered water into the appropriate sample bottles, label, and store on ice.

3.3 Chlorophyll *a* Samples

The procedure for collecting chlorophyll *a* samples is somewhat different than most other sample types. This type of sampling involves preserving the sample prior to filtering. Instead of sending the filtered water to the laboratory, the filter pad is preserved and sent to the lab for analysis. Procedures for chlorophyll *a* sampling are outlined below:

- Collect a discrete water sample from the required depth as described in previous sections of this document.

- Set up the filtering apparatus and insert the glass filter pads distributed by the laboratory. These filters are different from the ones used for other parameters and should be inserted with the rough side of the pad facing up.
- Add 1ml of $MgCO_3$ preservative to a 100ml sample of water and stir gently.
- Pour the water with preservative into the filtering apparatus and allow the entire contents to be flushed through the filter.
- Remove the filter and fold it in half so that the bottom of the pad is on the outside.
- Wrap the filter in a piece of aluminum foil and place in an opaque sample bottle. This will ensure that no light can reach the sample material.
- Label the bottles and store on ice.

3.4 Base Flow Sampling

During periods of seasonal base flow, water samples may be collected from each of the 12 automated high flow sampling locations. Such samples may be collected with the standard collection methods or by pumping the water from the stream with the ISCO automatic sampler. Prior to beginning the grab sample, the input hose should be disconnected from the ISCO sampler at the connection between the peristaltic pump and the internal distribution arm. Start the pump and allow the water to run through the hose for several seconds before filling bottles. When filling a container, hold the hose at an angle against the side or below the surface of the water to minimize aeration and mixing. Filter samples as needed.

Once all samples have been collected, power down the ISCO sampler. Reattach the hose to the internal distributing arm connection. Reprogram the ISCO sampler as required to initiate monitoring for high flow sampling as described in CDM Standard Operating Procedure 2-1: *High Flow Sampling.*

4.0 Sample Containers, Preservation Techniques, Quality Control

Refer to **Table 1** for the specific analyses and analytical methods. Periodically throughout the sampling, samples should be packed and shipped in coolers to one of several different analytical laboratories depending on analyses required (contact information below). A description of sample containers required for each parameter is also available in **Table 1.**

Field control samples may be collected by the sampling team and/or created by the CDM laboratory (Denver) to determine whether data are of suitable quality. Control samples may include trip blanks, duplicates, decontamination (rinsate) blanks, or split samples. Duplicates collected as co-located or split samples should be collected at least once for every twenty samples

4.1 Decontamination Procedures

To ensure that samples are not contaminated by equipment or containers, it is necessary to follow certain procedures for cleaning or decontaminating equipment. All sampling equipment which is in direct contact with the sample water should be cleaned between each sample collection. Equipment which should be decontaminated may include, but is not limited to: Van Dorn samplers, churn splitters, filtering apparatus, beakers or flasks, and volumetric measurement devices.

Procedures for decontamination are as follows:

- Rinse all surfaces with de-ionized or distilled water.
- Using a spray bottle, apply a layer of phosphate-free detergent to all surfaces.
- Rinse all surfaces again with de-ionized or distilled water until all detergent has been removed.
- If possible, rinse the container with water from the sample source.

4.2 Laboratory Contact Information

The following is a list of contact information and shipping addresses for all analytical laboratories used for water samples. Refer to Table A-1 for a list of samples that should be sent to each laboratory.

General Engineering Laboratories, LLC
701 Pine Ridge Road
Unit 5
Golden, CO 80403
Contact: Paul Winkler, 720-253-3093
E-mail: Paul.winkler@gel.com

Aquatic Research Inc
3927 Aurora Avenue North
Seattle, WA 98103
Contact: Steve Lazoff, 206-632-2715

A&L Analytical Laboratories, Inc.
2790 Whitten Rd.
Memphis, TN 38133
Contact: Jimmy Ferguson or Scott McKee, 800-264-4522
E-mail: smckee@allabs.com

Environmental Microbiology Laboratory
1150 Bayhill Drive, Suite 100
San Bruno, CA 94066
Contact: Cole Mackelprang, 858-268-2762
E-mail: cmackelprang@emlab.com
Contact: Megan S. Tatreau, 858-268-2770
E-mail: mtatreau@emlab.com

Idaho State University-Department of Biological Sciences
650 Memorial Drive
Pocatello, ID 83209-8007
Contact: Erin O'Leary-Jepsen (208) 282-4890

Aquatec Biological Sciences
273 Commerce St.
Williston, VT 05495
802-860-1638
Contact: Jennifer Gallant or Phil Downey
E-mail: jgallant@aquatecb.com

5.0 Documentation

Bound field logbooks should be used for the maintenance of field records. All aspects of sample collection and handling as well as visual observations should be documented in the field logbooks. Supplemental information may be documented on the field data sheets provided. All entries in field logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.

6.0 Additional Information

Other contact information:

Bert Fisher, PhD
Lithochimeia, Inc.
222 South Kenosha Ave.
Tulsa, OK 74120
Telephone: 918-382-9784

Ronald French
CDM
100 North Tucker Blvd.
Suite 550
Saint Louis, MO 63101
314-241-8510

7.0 References

Oklahoma Water Resources Board (OWRB). 2001. *Standard Operating Procedures (SOP) for Field Sampling Efforts of the Oklahoma Water Resources Board's Beneficial Use Monitoring Program.* Oklahoma Water Resources Board; Water Quality Programs Division; Oklahoma City, Oklahoma.

Herlihy, A.T. 1998. Water chemistry. pp. 57-65 IN: J.M. Lazorchak, D.J. Klemm, and D.V. Peck (Eds.). Unpublished draft. Environmental Monitoring and Assessment Program - Surface Waters: Western Pilot Study Field Operations Manual for Wadeable Streams. EPA/620/R-94/004F. U.S. Environmental Protection Agency, Washington, D.C.

8.0 Revised Dates*

The following are other revision dates applicable to this SOP:

Revision 1 - April 14, 2006

Table 1. Summary of sample parameters, bottles used, preservatives, filtering required, and analytical laboratories used during water sampling of rivers and streams.

Parameters	Bottles		Preservative	Filtered	Analytical Lab
Cu, Zn, As, P (6010/6020)	500 mL	plastic	Nitric Acid	Yes	A&L
Cu, Zn, As, P (6120/6020)	500 mL	plastic	Nitric Acid	No	A&L
TKN, Total P (365.2)	500 mL	plastic	Sulfuric Acid	No	A&L
Total P (365.2)	500 mL	plastic	Sulfuric Acid	Yes	A&L
Total Suspended Solids (TSS), Total Dissolved Solids (TDS), Total Ortho P, Nitrate+Nitrite	1 liter	plastic	None	No	A&L
Dissolved Ortho P, Anions	500 mL	plastic	None	Yes	A&L
Total Organic Carbon (TOC)	2 x 40mL	VOA vials	Hydrochloric Acid	No	A&L
Total P (duplicate)	60 mL	plastic	None	No	Aquatic Research
Total P, Ortho P (365.1) (duplicate)	60 mL	plastic	None	Yes	Aquatic Research
Chlorophyll *a*	250 mL	brown plastic	$MgCO_3$	Yes- send filters	Aquatec
Trihalomethane Formation Potential (TFP)	1 liter	glass	None	No	A&L
Estrogen Metabolites	1 liter	amber glass	None	No	GEL
Bacteria (7 types)	1 liter	sterile plastic	None	No	EML
Polymerase Chain Reaction (PCR)	1 liter	sterile plastic	None	No	Idaho State University

Prepared: Brian Bennett Review: Tony Gendusa

Approved: [signature] Date Approved: 6-Feb-2007

1.0 Overview and Application

This standard operating procedure (SOP) describes field procedures used for collection of resident fish from rivers, lakes, and streams. This SOP will be used for collecting samples using electrofishing gear and by seining and will be based upon the USEPA Rapid Bio-assessment Protocols (RBP) for use in wadeable streams and rivers (Barbour, 1999).

The Illinois River watershed in eastern Oklahoma and western Arkansas has been receiving inputs from agricultural runoff, primarily from fields where poultry waste has been applied. These wastes, along with the chemical constituents that make up the poultry wastes, have impacted the water quality and sediments in the streams and lakes in the Illinois River Watershed. The purposes of the work being performed are to evaluate and document 1) the linkage and relationship, if any, between the disposal of poultry wastes and environmental contamination within the Illinois River Watershed, and; 2) the resulting harm/injury to resident fish populations that may have resulted from the disposal of poultry wastes within the Illinois River Watershed.

2.0 Sampling Methods Summary

The collection of fish will involve the use of both electrofishing and seining as capture techniques. Due to variations in water chemistry, stream type, and fish morphology, different sampling techniques must be employed to ensure that representative samples of the resident fish populations are collected. The relative advantages of each sampling method are discussed in detail in Fisheries Techniques (Murphy and Willis, 1983).

Electrofishing is the process of introducing a high voltage/low amperage electric charge into the water which stuns the fish and allows for easy collection, commonly with the use of dip nets. This sampling technique is selective for deep-bodied fish with large surface areas. Electrofishing is typically used in habitats where seining is not possible, such as around woody debris, in very shallow riffles, and near undercut banks. Electrofishers are only effective in water with a specific conductance of 40-1700uS with a maximum specific conductance of roughly 1000uS for the less powerful backpack units (OWRB, 2004).

Seines are nets of various dimensions and mesh sizes which are dragged through the water at a certain speed in order to encircle and trap the fish. The seines are pulled along the bottom from both ends of the net and are long enough to produce a pocket or bag behind the operators that will capture and hold fish. Seining is selective for collecting smaller fish then electrofishing and is only effective in areas without large quantities of woody debris or other obstacles that may become entangled in the net.

2.1 Sampling Locations

Fish sampling will be conducted at 10 impacted locations in various streams and tributaries within the Illinois River watershed. Additional fish sampling will be conducted at 3 predetermined reference locations within the USEPA Eco-region III. Sample areas will be 100 meters in length and consist of riffles, runs, and pools in proportions that are reflective of the overall characteristics of the stream.

2.2 Sampler Selection

In an effort to achieve a representative sample of the resident fish populations, both electrofishing and seining may be conducted at each sampling location. The amount and proportion of each method used will vary depending on site-specific factors that are limiting to the effectiveness of each sampling type.

2.3 Site Data to be Collected

At each fish sampling location, a variety of physical variables should be recorded in order to quantify factors that may have an influence on the resident fish populations and/or the efficacy of the sampling techniques employed. Variables may include, but are not limited to:

- Average stream width, depth, and velocity within the sampling reach.
- Water temperature, conductivity, pH, and dissolved oxygen (DO) content.

2.4 General Supplies

The following is a list of the minimum supplies needed for a resident fish sampling event:

- Backpack and bank electrofishing units
- Shocking wands (anodes) with kill switch
- Plastic five-gallon buckets with handles
- Block nets with ¼ inch mesh size
- Seines
- Dip nets of various sizes
- Fish cages for live fish storage
- Gas powered electric generators
- Variable voltage pulsator units (VVP)
- Tow boat to hold generator and VVP
- Waders for each crew member
- Non-conductive rubber gloves for each crew member
- Polarized glasses for each crew member
- Multi-meter for collecting water quality parameters
- Marsh-McBernie water velocity meter
- Tape measure (for measuring width and length of station)
- Magnifying glasses to aid in fish identification
- Fish identification keys

- Extra gasoline/oil mixture for generators
- Spark plugs and soldering iron for equipment repairs
- Voltmeter
- 100′ extension cord
- Handheld GPS Unit, with extra batteries
- One meter fish measuring board
- 1 pound capacity weight scale
- 5 pound capacity weigh scale
- Data Sheets on water resistant paper
- Field Notebooks with water resistant paper
- Writing utensils (waterproof)
- Digital camera with extra batteries
- Applicable scientific collection permits with team member who appears on permit
- Sunscreen
- Insect repellent
- Drinking water/snacks/lunches for the crew

3.0 Safety

Electrofishing involves running a high-powered electric current through water can be very dangerous if the proper safety procedures are not followed. Since electric shock can occur, crewmembers should be trained in first-aid and CPR prior to sampling. Some general safety guidelines include:

- While electrofishing, avoid contact with the water unless sufficiently insulated from electric shock. Only waders and watertight, non-conductive rubber gloves should ever touch the water.
- Do not reach into the water at anytime during electrofishing.
- Avoid contact with the anode.
- All electrofishing wands have kill switches that will stop the flow of electricity if released during sampling. Do not make any modifications to these switches.
- Be aware of other members of the crew. If another crewmember falls or makes contact with the water, release the kill switch to stop the electric current immediately.
- If waders or gloves develop leaks or become wet on the inside, they may no longer serve as effective insulators and should be replaced.
- Do not electrofish in heavy rain or around other people, pets, or livestock.
- If stream conditions are not fit for electrofishing (i.e., flow is too high, conductivity is <10uS or >1000uS, water is too deep or too turbid), the crew may choose not to sample the site at that time.
- Gasoline and preservatives should be handled and stored properly.

4.0 Sampling Procedures

Fish will be collected across a 100-meter stretch of stream with block nets (¼ inch mesh) placed on each end of the sampled reach to ensure that the sampling is limited to a closed population and the data collected is not influenced by emigrant or immigrant individuals. A multiple-pass depletion methodology based on a closed population will be used to provide the data necessary for a reasonable population estimate of all fish species present at each location (White et al. 1982).

4.1 State Notification

Prior to sampling the appropriate state agency must be contacted to inform them of the location of the sample site and when sampling will occur. A copy of the collecting permits must be present at all times. The contact information differs for each state.

For sites in Oklahoma contact:

Jim Burroughs
N.E. Region Fisheries Supervisor
Oklahoma Department of Wildlife Conservation
9097 N. 34th Street West
Porter, Oklahoma 74454
918-683-1031

For sites in Arkansas contact:

Capt. Luther Hungate
Arkansas Game and Fish Commission
Northwest Regional Office
455 Dam Site Road
Eureka Springs, AR 726314
866-253-2506
1-800-482-9262

4.2 Site Preparation

Once the location of each site has been established and verified by GPS, the site must be prepared prior to sampling. The 100m reach of stream to be sampled should be measured and block nets should be placed at both ends of the reach. The block nets should extend across the entire width of the stream and be tall enough to extend approximately 1 meter above the surface of the water while maintaining firm contact with the substrate. The block nets should be weighed down with a lead line or available rocks so that fishes cannot move past them. Fish cages should be placed at regular intervals along the stretch of stream to hold captured fish.

4.3 Procedures

Following the completion of necessary site preparations, the first sampling pass can begin. A sampling pass will consist of a thorough collection via electrofishing and/or seining in all feasible locations within the sampling reach. A minimum of two complete passes will be conducted at each location, and fish will not be returned to the site between passes. This will enable statistical calculations to be made so that a valid population estimate can be calculated.

4.3.1 Electrofishing

The backpack electrofisher is essentially a small gasoline powered generator attached to a variable -voltage pulsator (VVP) which has outputs for both an anode and a cathode. The cathode is a stainless steel cable that is trailed behind the operator. The anode consists of a horizontal metal ring or diamond shaped electrode attached at the end of a fiberglass pole with a safety kill switch mounted on the handle. In most cases, either electrode type may be used. However, in waters with extremely low conductivity (<40uS) or in deep water, the ring electrode is often more effective. In waters with a conductivity >500uS, the diamond-shaped electrode should be used (OWRB, 2004).

In larger streams a shore-based electrofishing unit may be used. This unit involves the use of a more powerful generator and VVP unit that can be placed on the shore or floated through the current on a small tow boat. The VVP is connected to the generator with a 100-foot waterproof extension cord to allow for greater mobility. The basic procedures remain the same as with a backpack unit.

Prior to shocking a safety overview of electrofishing should be conducted and crew members should read and understand the operating manuals for the backpack electrofishing units prior to use. To avoid the risk of electric shock, persons involved in sampling should wear rubber, non-conductive gloves and waders at all times. All safety precautions must be observed.

The following procedures should be followed:

- A minimum of two crew members are required per electrofishing unit, one to operate the unit and at least one person to net stunned fish. Additional netters may also be employed to increase the catch rate of stunned fishes. One crew member will be responsible for regularly

transporting collected fish in buckets to live wells to prevent the adverse effects of overcrowding in the collection buckets. Crew members must wear the appropriate safety gear at all times.

- Sampling begins at the downstream limit of the 100m site and will proceed in an upstream direction. All available habitat types should be sampled within the stream.
- Once the electrofishing unit is powered on, the operator should depress the safety kill switch, causing the electric field to be produced. The operator should gradually pass the anode back and forth across the stream width and around any areas that could provide cover. Special attention should be given to areas surrounding root wads, brush piles or undercut banks.
- As the electrode approaches fish, they will become stunned and will roll become visible to the netters. The netters can then collect the fish and move them to buckets or live wells until the collection is completed.
- In some cases, it may be more effective to insert the probe into an area containing fish prior to depressing the switch. This will allow the fish to become accustomed to the probe and will prevent them from fleeing. When the field is turned on, more fish will be stunned and will be easier to capture.
- Once the entire reach has been sampled, the crewmembers may power off the electrofishing units and return to the live wells to examine and enumerate the fish.

4.3.2 Seining

Seines consist of nets of various lengths attached to vertical poles on either end and are pulled through the water while maintaining contact with the bottom. The height of the net should be greater then the depth of the water to prevent fish from escaping. Obstructions within the stream will often make seining impossible, so only certain areas will be sampled by this method.

- Seining will typically be done by two crewmembers pulling the net through the water in a downstream direction. Enough slack should be maintained behind the operators in order to capture and hold fish. The lead-lines should maintain contact with the bottom and the float-lines should be above the surface at all times.

- The seine should be pulled through the water for at least 10 meters and then dragged up a gradually sloping bank. Then the fish captured can be collected and stored in live wells or buckets for identification and eventual release.
- If a gradually sloping bank is not available, the dip-method can be utilized. This involves keeping a wide bag in the net and then pulling it up a steeper bank by turning the poles horizontally while following the bottom as closely as possible.
- In some cases, a fast moving current will make seining in a downstream direction impossible. In this event, seining should be conducted perpendicular to the current with the downstream operator moving slightly ahead to form a "j" shape in the net.
- Other seining techniques may also prove effective and may be employed by the field crew in some cases.

5.0 Data Collection and Quality Control

After each pass, all fish captured will be counted and stored in a live well or bucket until the final pass at each site is completed. The specimen collected will then be keyed out and identified to the species level. Various fish identification manuals will be available including: *Fishes of Oklahoma* (Miller and Robinson, 2004), *Fishes of Arkansas* (Robinson and Buchanan, 1984), and the *Peterson Field Guide to Freshwater Fishes of North America.* Any specimen that cannot be positively identified in the field will be preserved and brought back to the lab for identification.

Where applicable, all captured fish will also be weighed and measured for total length and any physical abnormalities will be noted. All fish population data will be recorded on the supplied data sheets using the fish species codes (**Table 1**) and any additional information will be recorded in field notebooks. If a large number of fishes of the same species are collected at a site, they may be counted and grouped into size classes so that a representative specimen may be measured from each group.

6.0 Documentation

Bound field logbooks should be used for the maintenance of field records. All aspects of sample collection and handling as well as visual observations shall be documented in the field logbooks. Supplemental information may be documented on resident fish population sampling

field sheets provided. All entries in field logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.

7.0 Additional Information

Other contact information:

Bert Fisher, PhD
Lithochimeia, Inc.
222 South Kenosha Ave.
Tulsa, OK 74120
Telephone: 918-382-9784

Ronald French
CDM
100 North Tucker Blvd.
Suite 550
Saint Louis, MO 63101
314-241-8510

8.0 References

Barbour, M.T., J. Gerritsen, B.D. Snyder, and J.B. Stribling. 1999. Rapid Bioassessment Protocols for Use in Wadeable Streams and Rivers: Periphyton, Benthic Macroinvertebrates, and Fish, 2nd Edition. EPA 841-B-99-002. U.S. Environmental Protection Agency; Office of Water; Washington, D.C.

Murphy, B.R., and D.W. Willis, editors. 1996. Fisheries techniques, 2nd Edition. American Fisheries Society, Bethesda, Maryland.

Oklahoma Water Resources Board (OWRB). 2004. Water Quality Monitoring Program: Field Sampling Protocol for Water Quality Assessments of Streams and Rivers, Draft Copy. Oklahoma Water Resources Board; Water Quality Programs Division; Oklahoma City, Oklahoma.

White, G.C., D.R. Anderson, K.P.Burnham, D.L. Otis, 1982. Capture-Recapture and Removal Methods for Sampling Closed Populations. LA-8787-NERP. U.S. Department of Energy; Los Alamos National Laboratory; Los Alamos, New Mexico.

9.0 Revised Dates*

The following revision dates are applicable to this SOP:

Revision 1 – November 29, 2005

Revision 2- January 19, 2006

Table 1: List of 3-Letter Codes for Captured Fish Species

Common Name	Scientific Name	Species Code
Banded darter	*Etheostoma zonale*	BND
Banded Sculpin	*Cottus carolinae*	BDS
Bigeye chub	*Hybopsis amblops*	BCH
Bigeye shiner	*Notropis boops*	BES
Black Bullhead	*Ameiurus melas*	BBH
Black Redhorse	*Moxostoma duquesnei*	BRH
Blackstripe Topminnow	*Fundulus olivaceus*	BTM
Bluegill Sunfish	*Lepomis macrochirus*	BSF
Bluntnose minnow	*Pimephales notatus*	BNM
Brook Silverside	*Labidesthes sicculus*	BSS
Cardinal Shiner	*Luxilus cardinalis*	CDS
Central Stoneroller*	*Campostoma anomalum*	CSR
Channel Catfish	*Ictalurus punctatus*	CCF
Creek Chub	*Semotilus atromaculatus*	CCH
Fantail Darter	*Etheostoma flabellare*	FTD
Fathead Minnow	*Pimephales promelas*	FHM
Gizzard Shad	*Dorosoma cepedianum*	GSD
Golden Redhorse	*Moxostoma erythrurum*	GRH
Green Sunfish	*Lepomis cyanellus*	GSF
Greenside Darter	*Etheostoma blennioides*	GSD
Largemouth Bass	*Micropterus salmoides*	LMB
Largescale stoneroller*	*Campostoma oligolepis*	LSR
Logperch	*Percina caprodes*	LGP
Longear Sunfish	*Lepomis megalotis*	LES
Mosquito Fish	*Gambusia affinis*	MOF
Northern Hogsucker	*Hypentelium nigricans*	NHS
Northern Studfish	*Fundulus catenatus*	NSF
Orangethroat Darter	*Etheostoma spectabile*	OTD
Ozark Minnow	*Notropis nubilus*	OZM
Redspot Chub	*Nocomis asper*	RCH
Rock bass	*Ambloplites rupestris*	RKB
Shadow Bass	*Ambloplites ariommus*	SHB
Shorthead redhorse	*Moxostoma macrolepidotum*	SRH
Slender Madtom	*Noturus exilis*	SMT
Smallmouth Bass	*Micropterus dolomieu*	SMB
Southern Redbelly Dace	*Phoxinus erythrogaster*	SRD
Spotted Bass	*Micropterus punctulatus*	SPB
Stippled Darter	*Etheostoma punctulatum*	STD
Warmouth Sunfish	*Lepomis gulosus*	WSF
Yellow Bullhead	*Ameiurus natalis*	YBH

STOK0020886

Prepared: Brian Bennett Review: Tony Gendusa

Approved: _____ Issued: _____

1.0 Overview and Application

This standard operating procedure (SOP) describes field procedures used for collection of resident fish from rivers and streams during the 2007 sampling event. This SOP will be used for collecting samples using electrofishing gear and kick seining and is primarily based on the USEPA Rapid Bio-assessment Protocols (RBP) for use in wadeable streams and rivers (Barbour, 1999), with modifications based on (1) protocols summarized from various natural resource agencies (**Table 1**) and (2) intended data uses.

The Illinois River watershed in eastern Oklahoma and western Arkansas has been receiving inputs from agricultural runoff, primarily from fields where poultry waste has been applied. These wastes, along with the chemical constituents that make up the poultry wastes, have impacted the water quality and sediments in the streams and lakes in the Illinois River Watershed. The purposes of the work being performed are to evaluate and document 1) the linkage and relationship, if any, between the disposal of poultry wastes and environmental contamination/nutrient enrichment within the Illinois River Watershed, and; 2) the resulting harm/injury to resident fish populations that may have resulted from the disposal of poultry wastes within the Illinois River Watershed.

2.0 Sampling Methods Summary

The collection of fish from rivers and streams will involve the use of both electrofishing and kick seining as capture techniques. Due to variations in water chemistry, stream type, and fish life history, different sampling techniques must be employed to ensure that representative samples of the resident fish populations are collected. The relative advantages of each sampling method are discussed in detail in Fisheries Techniques (Murphy and Willis, 1983).

Electrofishing is the process of introducing a high voltage/low amperage electric charge into the water which stuns the fish and allows for easy collection, commonly with the use of dip nets. This sampling technique is selective for deep-bodied fish with large surface areas. Electrofishing is typically used in habitats where seining is not possible, such as around woody debris, in very shallow riffles, and near undercut banks. Electrofishers are only effective in water with a specific conductance of 40-1700uS with a maximum specific conductance of roughly 1000uS for the less powerful backpack units (OWRB, 2004).

Seines are nets of various dimensions and mesh sizes which are dragged through the water at a certain speed in order to encircle and trap the fish or, for kick seines, are placed at locations for a short time period to trap fish dislodged by kicking the substrates in which these species live. Kick seines are specifically employed for the collection of riffle-dwelling fish species that are not successfully collected using electroshocking techniques. These commonly include small bottom dwelling species such as madtoms and darters. The kick seines are placed more or less perpendicular to the flow of water and held in place by two persons. A third person stands immediately (i.e., one to two meters) upgradient of the seine and kicks the stream bottom substrates (sand, gravel, cobble) to dislodge bottom dwelling or sediment-associated fish. Dislodged fish are then carried by the current into the seine, where they are collected, identified, and counted.

2.1 Sampling Stations

Fish sampling will be conducted at approximately 30 selected locations in various streams and tributaries within the Illinois River watershed. Additional fish sampling may be conducted at predetermined reference locations within the USEPA Eco-region III. In wadeable streams, the sampling area will consist of a stream length equal to 30 times the mean wetted stream width at the time of sampling, but in any case not less than 100 meters in length. The mean wetted stream width will be measured by averaging the mean wetted width of two riffles and two pools. The mean wetted width of four representative transects will be used where habitat variability is low (i.e., if two pools and two riffles are not present). Stream length will be measured along the descending left bank (on the left, facing downstream). The minimum stream length to be sampled is 100 meters, and the maximum length is 800 meters. The maximum will be most applicable to larger non-wadeable rivers such as the mainstem Illinois River. Block nets will not be used but natural barriers or habitat type boundaries (e.g., beginning or end of a riffle) will be used to define the beginning and end of each sampling reach.

2.2 Sampling Procedures - Overview

In an effort to achieve a representative sample of the resident fish populations, both electrofishing and kick seining will be conducted at each sampling location. The amount and proportion of each method used will vary depending on site-specific factors that are limiting to the effectiveness of each sampling type. Electrofishing will be employed for sampling runs and pools, and kick seining will be used to sample all riffles. Pools and runs At least 3 pools and 3 runs within the selected reach of wadeable streams will first be sampled using backpack electroshocking. At least 3 riffles of the same reach will be additionally sampled using kick seines. Sampling using both methods will be based on a predetermined unit of time, initially set at 3 minutes for pools and runs and 0.5 minutes (30 seconds) for riffles, but subject to field modification. Each unit of effort will therefore initially be a 3 minute (pools and runs) or a 0.5

minute (riffles) period of time. Some habitats (e.g., a small riffle) may require only one unit of effort (30 seconds) to completely sample, while others (e.g., a deep pool with logs and vegetation) may require 3 or more units of effort (9 + minutes, in this example). At the end of each unit of effort, the sampling will pause and another sample will be collected. The "pause" may simply be switching buckets to keep each "unit's" catch separate or, in cases where a habitat type has been completed, may consist of identifying and counting the fish collected during that "unit". Fish data (e.g., numbers of each species collected) will be kept separate for each unit of effort, even if multiple units are required to sample a single habitat unit. Units of effort will apply to both electroshocking (actual "pedal on" time of 3 minutes or 180 seconds) and kick seining (30 seconds of substrate disturbance). Habitat units will be limited to one of three habitat types: pool, riffles, and runs/glides. Each will be defined using U.S. Forest Service (USFS) guidelines (McCain et al. 1990). For this sampling effort, runs and glides are assumed to be equal, and no distinction is made between these two habitat types. USFS graphical and textual descriptions for each of the three habitat types will be maintained by each field team.

Electroshocking in wadeable streams will be conducted using one pass, proceeding from downstream to upstream, moving from shore to shore in a zigzag pattern, until the minimum number of habitat types has been completely sampled. Kick seining of riffle habitats will also proceed from downstream to upstream, following completion of backpack electroshocking. Electroshocking by boat in non-wadeable streams will proceed from downstream to upstream along one bank, then in the same direction along the opposite bank, making one pass per bank. Section 4.3 provides detailed procedures for each sampling method.

2.3 Data Collection

At each fish sampling location, a variety of physical variables should be recorded in order to quantify factors that may have an influence on the resident fish populations and/or the efficacy of the sampling techniques employed. Variables may include, but are not limited to:

- Average stream width, depth, and velocity within the sampling reach.
- Amount and type of vegetation along each bank and instream (e.g., 60% vegetated, primarily with grasses and shrubs)
- Water temperature, conductivity, pH, and dissolved oxygen (DO) content.
- Dominant substrate type and size for each of the four habitat types (pool, riffle, run/glide)
- Numbers of each type of fish collected by unit of effort for each habitat unit (i.e., for each 3 minute unit of effort for a given pool or run and for each 30 second effort for each riffle).

2.4 Supplies

The following is a list of the minimum supplies needed for a resident fish sampling event:

- Smith-Root LR-24 Electrofishing Equipment
- Boat and boat-mounted electroshocking equipment
- Shocking wands (anodes) with kill switch
- BC-24ps Battery Charger
- Portable generator
- Plastic five-gallon buckets with handles
- Kick seines (1/8 inch mesh)
- Dip nets of various sizes (1/8 inch mesh)
- Waders for each crew member
- Non-conductive rubber gloves for each crew member
- Polarized glasses for each crew member
- Habitat identification keys
- Multi-meter for collecting water quality parameters
- Marsh-McBirney water velocity meter
- Tape measure (for measuring width and length of station)
- Magnifying glasses to aid in fish identification
- Fish identification keys
- Extra gasoline/oil mixture for generators (if applicable)
- Extra batteries for electrofishing units
- Spark plugs and soldering iron for equipment repairs

- Voltmeter
- 100′ extension cord
- Handheld GPS Unit, with extra batteries
- Data Sheets on water resistant paper
- Field Notebooks with water resistant paper
- Writing utensils (waterproof)
- Digital camera with extra batteries
- Applicable scientific collection permits with team member who appears on permit
- Sunscreen
- Insect repellent
- Drinking water/snacks/lunches for the crew

3.0 Safety

Electrofishing involves running a high-powered electric current through water can be very dangerous if the proper safety procedures are not followed. Since electric shock can occur, crewmembers should be trained in first-aid and CPR prior to sampling. Some general safety guidelines include:

- While electrofishing, avoid contact with the water unless sufficiently insulated from electric shock. Only waders and watertight, non-conductive rubber gloves should ever touch the water. Non-conductive rubber gloves will be worn by all team members while in the water.
- Do not reach into the water at anytime during electrofishing.
- Avoid contact with the anode.
- All electrofishing wands have kill switches that will stop the flow of electricity if released during sampling. Do not make any modifications to these switches.
- Be aware of other members of the crew. If another crewmember falls or makes contact with the water, release the kill switch to stop the electric current immediately.

- If waders or gloves develop leaks or become wet on the inside, they may no longer serve as effective insulators and should be replaced.
- Do not electrofish in heavy rain or around other people, pets, or livestock.
- If stream conditions are not fit for electrofishing (i.e., flow is too high, conductivity is <10uS or >1000uS, water is too deep or too turbid), the crew may choose not to sample the site at that time.
- All field personnel should read the User Manual for the Smith-Root LR-24 Electrofisher, Pages 45 through 49 cover safety features for this equipment.

4.0 Sampling Teams

Sampling teams will consist of 4 persons per team. For electroshocking in wadeable streams, each team will consist of 1 shocker, 2 netters, and 1 recorder (follows closely behind the electroshocking team). For kick seining, each team will consist of 1 "kicker" (disturbs the substrate), 2 persons stabilizing the seine, and 1 recorder. For boat electroshocking, each team will include 1 shocker/netter, 1 recorder and boat driver. The recorder may have to follow along the shore if the boat capacity does not allow for 4 people.

4.1 State Notification

Prior to sampling the appropriate state agency must be contacted to inform them of the location of the sample site and when sampling will occur. A copy of the collecting permits must be present at all times. The contact information differs for each state.

For sites in Oklahoma contact:

Jim Burroughs
N.E. Region Fisheries Supervisor
Oklahoma Department of Wildlife Conservation
9097 N. 34th Street West
Porter, Oklahoma 74454
918-683-1031

For sites in Arkansas contact:

Capt. Luther Hungate
Arkansas Game and Fish Commission
Northwest Regional Office
455 Dam Site Road
Eureka Springs, AR 726314
866-253-2506
1-800-482-9262

4.2 Initial Tasks / Site Preparation

Once the location of each site has been established and verified by GPS, several tasks must be completed prior to sampling. Water quality samples, if required, should be collected prior to disturbing the bottom substrates. One team member should begin filling out the data collection/field forms which generally describe the site, as discussed in Section 2.3. Other team members can begin determining the specific reach to be sampled. This begins by determining if at least 3 runs, 3 riffles, and 3 pools are present within the minimum reach length of 100m. If yes, then the upper boundary of reach is flagged at 100m and the reach to be sampled is 100 meters in length. If at least 3 of each habitat units does not occur within 100m of the starting location, then the upper boundary of the reach is flagged at the point determined by 30 times the mean wetted stream width, as discussed in Section 2.1, but not to exceed 800m in length. In this case, the reach to be sampled should be measured and recorded in the field data form. The upper or most upstream limit of the reach may be extended slightly beyond the marked limit to ensure complete sampling of a specific habitat unit. For example, do not end the sampling in the middle of a large pool but extend the sampling to ensure complete sampling of the pool. The final length sampled should be recorded on the field data sheets, with notification if it exceeds the length beyond that defined by either the 100m minimum or 30 times the mean wetted stream width.

4.3 Detailed Sampling Procedures

4.3.1 Electrofishing

Wadeable Streams

The LR-24 backpack electroshocker consists of a trailing stainless steel electrode cable and a ring electrode mounted on a yellow wand that has a red operating/kill switch. A minimum of three people should be used for each electroshocking event. A fourth person may be identified as a recorder, and this person remains on the bank during electroshocking. One person carries the shocking unit and wand, and two people each carry both a large dip net and a smaller dip net.

Team members should not switch tasks during the sampling effort at each site (i.e. the person carrying the anode will not switch with a netter and vice versa during the pass). A netter should be on each side of the shocking wand. Each netter will have two nets, one smaller one for difficult to reach fish and one larger net. Collection begins at the downstream end of the segment, moving upstream. The wand holder should move in a zigzag pattern across the habitat unit, with the goal of sampling all habitat types within the specified habitat unit. The amount of electroshocking time as shown on the backpack unit (actual "pedal-on" shocking time) should be recorded on the field sheet. Prior to shocking, a safety overview of electrofishing should be conducted and crew members should read and understand the operating manuals for the backpack electrofishing units prior to use. To avoid the risk of electric shock, persons involved in sampling should wear rubber, non-conductive gloves and waders at all times. All safety precautions must be observed. All fish will be returned to the stream except those that cannot be reliably identified in the field. These will be retained in formalin for laboratory identification.

The following procedures should be followed:

- A minimum of four crew members are required per electrofishing unit, one to operate the unit, two persons to net stunned fish, and a team member to record data as sampling proceeds—this person follows the shocking crew. Upon the completion of sampling a habitat type (e.g. riffle, pool, etc), sampling team members will identify the fish collected and record on the appropriate data sheets. Fish will be released back to the stream, downstream of the habitat type sampled. The amount of electroshocking time for that habitat type will be recorded. Electroshocking time can be obtained from the LR-24 unit. Electroshocking time in each habitat type will be for 3 minutes.

- Sampling begins at the downstream limit of the reach to be sampled and will proceed in an upstream direction. All available microhabitats will be sampled within the identified habitat unit.

- Once the electrofishing unit is powered on, the operator should depress the safety kill switch, causing the electric field to be produced. The operator should gradually pass the anode back and forth across the habitat unit (e.g., pool) and around any areas that could provide cover. Special attention should be given to areas surrounding root wads, brush piles or undercut banks. After 3 minutes have passed and the electrode has been turned off, do not turn it back on to re-shock fish that may have been missed during the netting process, unless they are actively swimming away. Every effort should be made to keep the pedal on time to exactly 3 minutes.

- As the electrode approaches fish, they will become stunned and will roll or float to the surface where they become visible to the netters. The netters can then collect the fish and move them to buckets or live wells until the collection is completed. In some cases, it may be more effective to insert the probe into an area containing fish prior to depressing the switch. This will allow the fish to become accustomed to the probe and will prevent them from fleeing. When the field is turned on, more fish will be stunned and will be easier to capture.

- A brief description of the physical characteristics (e.g. substrate type, pool depth) of the habitat will be recorded on the field data sheets.

Unwadeable Streams

In larger streams a shore-based or boat-mounted electrofishing unit may be used. For this study, the Smith-Root 2.5 GPP electroshocking unit is mounted to a 14' boat. The pulsating unit is connected to the generator with the electroshocking wands and cable extending from the boat. The boat crew shall consist of one person driving the boat, one of the net persons directing the boat driver, and a third person netting (as needed) and recording. One additional person can remain on shore to aid in fish identification.

As per wadeable streams, the length of river reach to be sampled is based on the minimum of 100m or 30 times the mean wetted stream width. If this value is calculated to exceed 800 meters, then the maximum length of stream to be sampled is set to 800 meters. Both banks will be shocked, and any available instream habitat will be sampled as well. A single pass will be made along each bank, proceeding from downstream to upstream. Netted fish will be kept in a live cage/well until identified and enumerated. All fish will be returned to the stream except those that cannot be reliably identified in the field. These will be retained in formalin for laboratory identification.

Sampling by boat will be used for all pools and runs within the predetermined sampling reach when that reach contains a mix of wadeable and unwadeable portions. In such reaches, kick seines will be used to sample all riffles, as described below.

4.3.2 Kick Seining

Kick seines consist of nets about 6 to 8 feet in length, 4 feet in height, with a mesh size of 1/8 inch. At each end of the seine is a vertical pole used to maintain the net in an upright position. The net is placed at the downstream end of a riffle perpendicular to the flow, and kept more or less upright by two persons (one at each pole). A third person stands about two meters immediately upgradient of the seine and kicks/disturbs the bottom substrate to dislodge small bottom dwelling fish. Kicking continues for 30 seconds, then the seine is removed and the fish are identified and counted. If the riffle has been completely sampled with this single 30 second

sampling event, the seining continues on to the next riffle upgradient. If the riffle is large or includes multiple microhabitat types, it may require additional 30-second collections. All microhabitat types within a riffle will be sampled regardless of how many "30-second time units" it takes to complete the sampling. For example, some riffles may be sampled using 3 separate "transects" to ensure that unique microhabitats within the riffle are sampled.

5.0 Data Collection and Quality Control

After each unit of time (initially set at 3 minutes), all fish captured will be either identified and counted or stored in a separate bucket or live well until the collection is complete for that habitat unit (e.g., a pool). The fish collected will be identified to the species level. Various fish identification manuals will be available including: *Fishes of Oklahoma* (Miller and Robinson 2004), *Fishes of Arkansas* (Robinson and Buchanan 1984), and the *Peterson Field Guide to Freshwater Fishes of North America* (Page and Burr 1991). Any specimen that cannot be positively identified in the field will be preserved and brought back to the lab for identification.

All captured fish will also be observed for any physical abnormalities, and any findings will be recorded on the field data sheets. All fish population data will be recorded on the supplied data sheets using the fish species codes (Table 2). Any additional information relevant to this study will be recorded in field notebooks.

6.0 Documentation

Bound field logbooks should be used for the maintenance of field records. All aspects of sample collection and handling as well as visual observations shall be documented in the field logbooks. Supplemental information may be documented on resident fish population sampling field sheets provided. All entries in field logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.

<u>Other contact information:</u>

Ronald French
CDM
100 North Tucker Blvd.
Suite 550
Saint Louis, MO 63101
314-241-8510

8.0 References

Barbour, M.T., J. Gerritsen, B.D. Snyder, and J.B. Stribling. 1999. Rapid Bioassessment Protocols for Use in Wadeable Streams and Rivers: Periphyton, Benthic Macroinvertebrates, and Fish, 2nd Edition. EPA 841-B-99-002. U.S. Environmental Protection Agency; Office of Water; Washington, D.C.

McCain, M., D.Fuller, L.Decker and K.Overton. 1990 . Stream habitat classification and inventory procedures for northern California. FHC Currents. No.1. U.S. Department of Agriculture. Forest Service, Pacific Southwest Region.

Murphy, B.R., and D.W. Willis, editors. 1996. Fisheries techniques, 2nd Edition. American Fisheries Society, Bethesda, Maryland.

Oklahoma Water Resources Board (OWRB). 2004. Water Quality Monitoring Program: Field Sampling Protocol for Water Quality Assessments of Streams and Rivers, Draft Copy. Oklahoma Water Resources Board; Water Quality Programs Division; Oklahoma City, Oklahoma.

U.S. Forest Service (USFS). July 2005. Stream Condition Inventory Technical Guide. United States Forest Service. Ecosystem Conservation Staff. Vallejo, CA 111p.

White, G.C., D.R. Anderson, K.P.Burnham, D.L. Otis, 1982. Capture-Recapture and Removal Methods for Sampling Closed Populations. LA-8787-NERP. U.S. Department of Energy; Los Alamos National Laboratory; Los Alamos, New Mexico.

Table 1. Summary of Protocols for Fish Sampling / Proposed Protocols

Item / Issue	ADEQ	ORWB	ODWC	OCC	NAWQA	USEPA	Proposed
Sampling reach length (ft)	700 - 1,500 ft (used in at least 1 study but not a specific protocol)	General = 400 m (sm streams = 200 m, rivers = 800 m)	not stated	30X mean stream width	not stated	Fixed distance and proportional distance (e.g., 40X mean width) acceptable. Max time acceptable for large rivers (e.g., 3 hrs).	**30X mean wetted stream width (measured along left descending bank), determined by mean of widths at two riffles and two pools, min = 100 m and max = 800 m (natural barriers, no block nets)**
Alternate (to electroshock) method employed	yes, seine	yes, seine	requires electroschock + seine	Requires electroschock + seine	requires electroschock + seine	electroshock single best method	**electroschock (backpack for wadeable streams; boat for non-wadeable streams or portions of streams) + kick net seine ; kick net seine follows electroshock-ing and covers range of microhabitats in all available riffles (e.g., 1-3 samples per riffle)**
Number of passes	not stated	not stated	min = 1, multiple at restoration sites, min = 3 for depletion estimates	Not stated	2	not stated	**1 (4 person team; two netters, 1 shocker, one recorder; each netter has both large and small nets)**
Battery powered backpack	yes	no, gas with Honda generator	not specific	no, gas with Honda generator	not specific	not specific	**Yes**
Habitat sampled	all available	all available	all available	All available	all available	all available	**all available**

Comparison Station	Upstream of stressor source preferred (comparison to another water body or to "least disturbed" sites often used)	not stated	not stated	Not stated	not stated	not specific but implied via use of various metrics (comparison to reference)	**Little Lee Cr for small streams;**
Species focus	all fish (for assessing water quality impacts)	not stated	sport fish	All	all	all, but fish <20 mm in length are not included	**all, but fish <20 mm in length are not included in ID or counts**
Fish released	large fish	large fish, species of concern if ID verified, easily identified taxa	all sport fish following wt and length	Only large with field ID (others preserved for lab ID)	all T&E species, all post ID except voucher specimens	all T&E species, all post ID except voucher specimens	**all T&E species, all post ID except those not positively identifiable in the field**
Dip net mesh size	Not stated	not stated	not stated	Not stated	not stated	not stated	**1/8" (for both large and small nets)**
Catch per unit effort / Recording of Effort	Not stated	not stated	not stated	Not stated	not stated	not specific	**record min. of shock time, total stream length; record fish data per discrete habitat unit (e.g., per each run, riffle, pool); target sampling time unit is 3 minutes of "pedal-on" time (180 seconds, for pools and runs) or 30 seconds of "kicking" for kick seine collections. In all cases, multiple units may be required for large or complex habitat units (each habitat unit = pool or or run or riffle, as defined by**

							USFS guidelines. Note that glides and runs are defined here to be equal, with no distinction between these two habitat types)
Summary Comment	Appears flexible	less specific but generally similar to EPA	focus on sport fish	Less specific but generally similar to EPA	less specific but generally similar to EPA	most specific and detailed, multiple options presented	**The methods/details shown in this column have been discussed and accepted by all parties (CDM, ODWC, June 2007)**

Table 1. References

ADEQ	Arkansas Department of Environmental Quality	**Pages 71-72 in***:* Illinois River Water Quality, Macroinvertebrate and Fish Community Survey, Benton and Washington Counties, Arkansas. ADEQ Water Division, 1997, 90 p. **AND Page 63 in***:* 2002 Integrated Water Quality Monitoring and Assessment Report*.* Prepared pursuant to section 305(b) and 303(d) of the Federal Water Pollution Control Act, ADEQ Water Division, 476 p.
ORWB	Oklahoma Water Resources Board (2004)	Oklahoma Water Resources Board (OWRB). 2004. Water Quality Monitoring Program: Field Sampling Protocol for Water Quality Assessments of Streams and Rivers, Draft Copy. Oklahoma Water Resources Board; Water Quality Programs Division; Oklahoma City, Oklahoma.
ODWC	Oklahoma Department of Wildlife Conservation	Oklahoma Department of Wildlife Conservation (ODWC). Date unknown. Protocols for Assessing Fish Population Response to Stream Restoration Projects. ODWC, 5 pages.
OCC	Oklahoma Conservation Commission	Oklahoma Conservation Commission (OCC). 1996. Standard Operating Procedure: Sampling Procedures used by the Oklahoma Conservation Commission for Fish Collection in Streams. OCC, 7 pages.
NAWQA	USGS- National Water Quality Assessment Program	Moulton, S.R., J.G. Kennon, R.M. Goldstein, J.A. Hambrock. 2002. Revised Protocols for Sampling Algal, Invertebrate, and Fish Communities as Part of the National Water-Quality Assessment Program. USGS Open-File Report 02-150. US Geological Survey; Reston, VA.
USEPA	U.S. Environmental Protection Agency, Rapid Bioassessment Protocols (RBP), second edition	Barbour, M.T., J. Gerritsen, B.D. Snyder, and J.B. Stribling. 1999. Rapid Bioassessment Protocols for Use in Streams and Wadeable Rivers: Periphyton, Benthic Macroinvertebrates and Fish, Second Edition. EPA 841-B-99-002. U.S. Environmental Protection Agency; Office of Water; Washington, D.C.

Table 2: Fish Species in Illinois River Watershed (OK/AR)

Common Name	Scientific Name	Species Code	Trophic Guild	Tolerance
Shadow Bass	*Ambloplites ariommus*	SHB	-	-
Rock bass	*Ambloplites rupestris*	RKB	P	M
Black Bullhead	*Ameiurus melas*	BBH	I	M
Yellow Bullhead	*Ameiurus natalis*	YBH	I	T
Freshwater Drum	*Aplodinotus grunniens*	FWD	V	M
Central Stoneroller	*Campostoma anomalum*	CSR	H	M
Largescale stoneroller	*Campostoma oligolepis*	LSR	H	M
White Sucker	*Catostomus commersoni*	WHS	O	T
Banded Sculpin	*Cottus carolinae*	BDS	I	M
Grass carp	*Ctenopharyngodon idella*	GCP	H	M
Red Shiner	*Cyprinella lutrensis*	RSH	O	T
Steelcolor shiner	*Cyprinella whipplei*	SCS	I	M
Common Carp	*Cyprinus carpio*	CCP	O	T
Gizzard Shad	*Dorosoma cepedianum*	GSD	O	M
Gravel chub	*Erimystax x-punctata*	GCH	I	M
Greenside Darter	*Etheostoma blennioides*	GSD	I	M
Fantail Darter	*Etheostoma flabellare*	FTD	I	M
Stippled Darter	*Etheostoma punctulatum*	STD	-	-
Orangethroat Darter	*Etheostoma spectabile*	OTD	I	M
Banded darter	*Etheostoma zonale*	BND	I	I
Northern Studfish	*Fundulus catenatus*	NSF	I	I
Blackstripe Topminnow	*Fundulus olivaceus*	BTM	I	M
Mosquito Fish	*Gambusia affinis*	MOF	I	M
Bigeye chub	*Hybopsis amblops*	BCH	-	-
Northern Hogsucker	*Hypentelium nigricans*	NHS	I	I
Channel Catfish	*Ictalurus punctatus*	CCF	P	M
Smallmouth Buffalo	*Ictiobus bubalus*	SBF	I	M
Black Buffalo	*Ictiobus niger*	BBF	I	M
Brook Silverside	*Labidesthes sicculus*	BSS	I	M
Longnose Gar	*Lepisosteus osseus*	LNG	P	M
Green Sunfish	*Lepomis cyanellus*	GSF	I	T
Warmouth Sunfish	*Lepomis gulosus*	WSF	P	M
Bluegill Sunfish	*Lepomis macrochirus*	BSF	I	M
Longear Sunfish	*Lepomis megalotis*	LES	I	I
Redear Sunfish	*Lepomis microlophus*	RES	I	M
Cardinal Shiner	*Luxilus cardinalis*	CDS	I, other *Luxilus*	M, other *Luxilus*
Duskystripe Shiner	*Luxilus pilsbryi*	DSS	I, other *Luxilus*	M, other *Luxilus*
Redfin Shiner	*Lythrurus umbratilis*	RFS	I	M
Smallmouth Bass	*Micropterus dolomieu*	SMB	P	M
Spotted Bass	*Micropterus punctulatus*	SPB	P	M
Largemouth Bass	*Micropterus salmoides*	LMB	P	M
Spotted Sucker	*Minytrema melanops*	SPS	I	M
Black Redhorse	*Moxostoma duquesnei*	BRH	I	I
Golden Redhorse	*Moxostoma erythrurum*	GRH	I	M
Shorthead redhorse	*Moxostoma macrolepidotum*	SRH	I	M
Redspot Chub	*Nocomis asper*	RCH	I, other *Nocomis*	I, other *Nocomis*
Bigeye shiner	*Notropis boops*	BES	I	I
Ozark Minnow	*Notropis nubilus*	OZM	H	I
Rosyface Shiner	*Notropis rubellus*	RYS	I	I
Slender Madtom	*Noturus exilis*	SMT	I	I

Logperch	*Percina caprodes*	LGP	I	M
Southern Redbelly Dace	*Phoxinus erythrogaster*	SRD	H	M
Bluntnose minnow	*Pimephales notatus*	BNM	O	T
Fathead Minnow	*Pimephales promelas*	FHM	O	T
Bullhead Minnow	*Pimephales vigilax*	BHM	O	M
White Crappie	*Pomoxis annularis*	WCR	P	M
Black Crappie	*Pomoxis nigromaculatus*	BCR	P	M
Flathead Catfish	*Pylodictis olivaris*	FCF	P	M
Creek Chub	*Semotilus atromaculatus*	CCH	G	T

Trophic Guild:
- P-piscivore
- H-herbivore
- O-omnivore
- I- insectivore
- V-intertivore

Tolerance Designation (non-specific stressors):
- I-intolerant
- M-intermediate
- T-tolerant

Reference for Trophic Guild and Tolerance Designations:

Barbour, M.T., J. Gerritsen, B.D. Snyder, and J.B. Stribling. 1999. Rapid Bioassessment Protocols for Use in Streams and Wadeable Rivers: Periphyton, Benthic Macroinvertebrates, and Fish. Second Edition. EPA 841-B-99-002. U.S. Environmental Protection Agency: Office of Water: Washington, D.C.

Prepared: Brian Bennett Review: Renee Mulcrone

Approved: [signature] Date Approved: 6-Feb-07

1.0 Overview and Application

This standard operating procedure (SOP) describes field procedures used for collection of benthic algae (periphyton) from streams in the Illinois River watershed in eastern Oklahoma and western Arkansas. Periphyton are a vital part of many stream communities and are often used as indicators of water quality conditions (Windell, date unknown). By comparing the composition, density, and growth rates of benthic algal assemblages between affected and reference streams at similar times of year, a valuable assessment of the environmental impact of various forms of pollution can be formulated. This SOP will be used during sampling and will follow the standard laboratory based approach for periphyton sampling as outlined in the USEPA's Rapid Bioassessment Protocols (RBP) for Use in Wadeable Streams and Rivers (Barbour et al., 1999).

The Illinois River watershed has been receiving inputs from agricultural runoff, primarily from fields where poultry waste has been applied. These wastes, along with the chemical constituents that make up poultry waste have impacted the water quality and sediments in the streams and lakes in the Illinois River Watershed. The purpose of this work is to evaluate and document 1) the linkage and relationship, if any, between the poultry waste disposal and environmental contamination within the Illinois River Watershed, and; 2) the resulting harm/injury to resident periphyton communities that may have resulted from the disposal of poultry wastes within the Illinois River Watershed.

2.0 Sampling Methods Summary

Observations of benthic algae will involve collecting samples from multiple habitat types within each stream. Three types of substrate will be sampled: natural substrates pre-existing at the site, manufactured artificial surfaces (microslides) placed at the site, and from clean stones placed at the site. All substrates introduced to the site will remain on location for 18-21 days before collection to allow for periphyton to colonize the surface. If a high flow (scouring) event occurs during this period, a longer sampling time will be required. Each substrate type will be collected separately, for a total of three individual sample types for each site.

Once collected, the samples will be preserved in Lugol's solution and stored in labeled 250 ml plastic jars. The samples must be kept in the dark until laboratory analysis is conducted. Each sample will be shipped to a qualified lab for analysis. A chain of custody (COC) document will be completed and shipped with each sample.

2.1 Sampling Locations

Seven periphyton samples will be collected from a 100 m reach of stream at each of 10 impacted locations in various streams and tributaries within the Illinois River watershed. Additional periphyton sampling will be conducted at three predetermined reference locations within the USEPA Eco-region III.

2.2 Data to be Collected

At each sampling location, a variety of physical variables should be recorded to quantify factors that may have an influence on the resident periphyton communities and/or the efficacy of the sampling techniques employed. Variables may include, but are not limited to:

- Average stream width, depth, and velocity.
- Water temperature, conductivity, pH, and dissolved oxygen (DO) content.

2.3 General Supplies

The following is a list of the minimum supplies needed for a periphyton sampling event:

- Scraping tools (e.g., stainless steel teaspoons, toothbrushes, razor blades)
- Two inch sections of PVC pipe (1.5" diameter)
- Artificial substrate (micro-slides)
- Clean natural substrate (rocks) to place at site
- Fencing to contain placed natural substrate
- Frame to hold artificial substrata (Periphytometer)

- Forceps, suction bulb, and disposable pipettes
- Preservative (Lugol's solution)
- Chain of Custody forms and labels
- Data Sheets on water resistant paper
- Field Notebooks with water resistant paper
- Multi-meter for collecting water parameters
- Tape measure (for measuring width and length of station)
- Handheld GPS Unit, with extra batteries
- Writing utensils (waterproof)
- Digital camera with extra batteries
- Applicable scientific collection permits with team member who appears on permit
- Sunscreen
- Insect repellent
- Drinking water/snacks/lunches for the crew

3.0 Sampling Procedures

Periphyton will be collected across a 100-meter stretch of stream in accordance with the USEPA Rapid Bio-assessment Protocols (RBP) multi-habitat approach. Three sample types will be collected from each location. Appropriate metrics that are relevant to the Illinois River watershed will be used to measure the ecological health of the periphyton community.

Prior to sampling, it is necessary to:

- Locate the predetermined site using a hand-held GPS unit.
- Measure out and mark the appropriate length of stream to be sampled (100 m).

- Use the multi-meter to measure the various in-stream parameters for the site.
- Identify substrates to be sampled and decide on appropriate sampler locations.

3.1 Collection from Existing Natural Surfaces

When sampling the natural surfaces existing at a site, three randomly selected rocks will be scraped. These samples will then be combined into a single, composite sample for each site (Barbour, 1999). The total sampled area should be equal for each site. Collection methods are outline below:

- Collect 10 various sizes of in-stream rocks that show demonstrated algae growth. Place rocks in a row and assign them numbers from 1-10, beginning on the left. Write the numbers 1 through ten on small pieces of paper and mix them up. Random number selection can then be made by blindly drawing three numbered pieces of paper out of the ten. These numbers will represent each rock that will be sampled.
- On the three rocks selected, mark a 1 ½ inch area using a plastic PVC pipe. Trace a circle around the pipe using a sharp nail. Gently remove the algae within the circle area with a stiff brush or razor blade and siphon off the contents with a syringe. Place the contents in a 250 ml amber bottle, that contains 100 ml of site water and preserve with five (5) drops of Lugol's solution.

After collection from all three surfaces is complete, label the bottle with station number, location, substrate type, date, collector's name, and type of preservative. Record any additional information in field notebook. Place the sample in a cooler and fill out a chain of custody (COC) form.

3.2 Collection from Introduced Surfaces

The collection of benthic algae from artificial surfaces will require two visits to the site. During the first visit, the artificial substrates (microslides) and cleaned stones will have to be placed on site. The microslides will be placed in a device known as a periphytometer which holds the slides in place within the water. Cleaned stones will be placed in a wire cage to keep them separate from the pre-existing substrate. The cage will then be placed in the stream and clearly

marked. The substrates will then be collected 18-21 days later during the return visit and the periphyton that has accumulated on the surfaces can be sampled.

3.2.1 Placement of Periphytometers

The periphytometers will consist of glass microslides that are placed in a stand which is placed in the stream at an appropriate location. The microslides must be thoroughly cleaned before being placed on location. Periphytometers should be placed in the water with the shield facing upstream. They should be secured to the bottom by attaching them to a brick or large stone and marked so that they can be recovered. Each periphytometer will contain 16 microslides and 1 periphytometers will be placed at each site. If a high-flow or scouring event occurs during the 18-21-day incubation period, additional time may be necessary.

3.2.2 Retrieval of Periphytometers

After an incubation period of 18-21 days, the microslides may be collected and analyzed. Upon returning to the site, record the relevant stream conditions and parameters (see Section 2.2) in the field notebook. To determine if an individual microslide is suitable for collection, the following criteria should be reviewed:

- The slide should still be completely immersed in water.
- The slide should be free of any floating debris (trash, leaves, etc.).
- The periphytometer should not have been subjected to a high-flow event.
- The slides should not have had >10% of the surface area cleaned by grazing, abrasion, or any other means (OCC, 2002)

The slides in each periphtyometer will be numbered and a computer program will be used to randomly select which slide numbers will be sampled. Collect suitable microslides and remove the randomly selected slides from each site. Each slide collected should be carefully removed from the periphytometer and placed in a 250ml sample container completely filled with water and an adequate amount of preservative. Label the bottle with station number, location, substrate type, date, collector's name, and type of preservative. Record any additional information in field notebook. Place the sample in a cooler with ice and fill out a chain of custody (COC) form.

3.2.3 Placement of Stone Surfaces

These introduced natural substrates will consist of locally gathered rocks that have been cleaned thoroughly to ensure that no periphyton is attached to the surface prior to placement. The stones will be placed in a small wire cage (approximately 1 ft on each side) with 1" mesh. This cage will be placed in shallow, lotic water so that the stones will remain submerged for 18-21 days to allow periphyton to accumulate on the surface of the stones. The cage should be marked and the location recorded so that retrieval will be possible. If a high-flow or scouring event occurs during the incubation period, additional time may be necessary.

3.2.4 Retrieval of Stone Surfaces

After an incubation period of 18-21 days, the stones will be collected and analyzed. Upon returning to the site, record the relevant stream conditions and parameters (cited in Section 2.2) in the field notebook. For the stones to be used as suitable samples the following criteria should be met:

- The stones should still be completely immersed in water.
- The stones should be free of any floating debris (trash, leaves, etc.).
- The location should not have been subjected to a high-flow event.

Once the stones and cages have been located, they can be removed from the stream and a sample of attached periphyton can be taken. A 1.5-inch diameter circle should be randomly marked on the stones. Scrape or brush the benthic algae attached to the surface of the stones within the circle and rinse accumulated algae into one 250 ml sample container and add preservative. Label the bottle with station number, location, substrate type, date, collector's name, and type of preservative. Record any additional information in field notebook. Place the sample in a cooler with ice and fill out a chain of custody (COC) form.

4.0 Preservation Techniques and Quality Control

To ensure that all samples are in sufficient condition for analysis of the periphyton when received by the laboratory, certain procedures must be followed.

- After collecting each sample, be sure to fill the jars with water and an adequate amount of preservative (Lugol's solution) immediately to prevent decay.

- Keep jars out of the sunlight until received by laboratory.
- Label all samples on the outside of the jar with sample ID, date, location, time, type of sample, and sampler's name.
- Complete a chain of custody form for each sample location and ship to laboratory for analysis.

5.0 Documentation

Bound field logbooks will be used for the maintenance of field records. All aspects of sample collection and handling as well as visual observations should be documented in the field logbooks. Supplemental information may be documented on periphyton sampling field sheets provided. All entries in field logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.

6.0 Additional Information

Laboratory contact information:

Debbie Niewenhuis
Water's Edge Scientific, LLC
S2756A County Tk. T
Baraboo, WI 53913
608-355-7930

Other contact information:

Ronald French
CDM
100 North Tucker Blvd.
Suite 550
Saint Louis, MO 63101
314-241-8510

STOK0020893

7.0 References

Barbour, M.T., J. Gerritsen, B.D. Snyder, and J.B. Stribling. 1999. Rapid Bioassessment Protocols for Use in Wadeable Streams and Rivers: Periphyton, Benthic Macroinvertebrates, and Fish, 2nd Edition. EPA 841-B-99-002. U.S. Environmental Protection Agency; Office of Water; Washington, D.C.

Kentucky Department of Environmental Protection (KDEP). 2002. Methods for Assessing Biological Integrity of Surface Waters. Kentucky Department of Environmental Protection, Division of Water, Frankfort, Kentucky.

Oklahoma Conservation Commission (OCC). 2002. Periphytometers & Processing for Chlorophyll-a Measurement. SOP No: IIA-06.1. Oklahoma Conservation Commission, Water Quality Division, Oklahoma City, Oklahoma.

8.0 Revised Dates*

The following are other revision dates applicable to this SOP:

Revision 1 – January 17, 2006

Prepared: Brian Bennett Review: Renee Mulcrone

Approved: [signature] Date approved: 6-Feb-2007

1.0 Overview and Application

This standard operating procedure (SOP) describes field procedures used for collection of benthic macroinvertebrates from streams in the Illinois River watershed in eastern Oklahoma and western Arkansas. Benthic macroinvertebrates are found in most flowing water bodies and are often used as indicators of water quality conditions. By comparing the composition and density of macroinvertebrate populations between affected and reference streams at similar times of year, a valuable assessment of the environmental impact of various forms of pollution can be formulated. This SOP will be used for collecting samples using fine-meshed dip nets and benthic seines (kick nets) and will follow the Oklahoma Water Resources Board's Beneficial Use Monitoring Program (BUMP) protocols. Procedures for collecting samples using standard Hester-Dendy samplers are also included.

The Illinois River watershed has been receiving inputs from agricultural runoff, primarily from fields where poultry waste has been applied. These wastes, along with the chemical constituents that make up the poultry waste, have impacted the water quality and sediments in the streams and lakes in the Illinois River Watershed. The purposes of this work is to evaluate and document 1) the linkage and relationship, if any, between the disposal of poultry wastes and environmental contamination within the Illinois River Watershed, and; 2) the resulting harm/injury to resident macroinvertebrate populations that may have resulted from the disposal of poultry wastes within the Illinois River Watershed.

2.0 Sampling Methods Summary

Streams often exhibit a large amount of seasonal variability in the structure and composition of benthic macroinvertebrate communities (Rabeni, 1996). Because of these seasonal variations in the composition, all sampling must be conducted during the Summer Index Period (July 1-Sept 15). Also, the collection of macroinvertebrates will only be conducted in flowing (lotic) water because of the following reasons:

- Invertebrate communities in lotic water are routinely exposed to the average water quality of each stream.

- Organisms most sensitive to changes in water quality are more commonly found in flowing water.
- The metrics developed to analyze macroinvertebrate communities have been designed for lotic habitats (OWRB, 2004).

Benthic macroinvertebrates in streams will be sampled using a 34 cm-wide fine mesh (size #30 or 590 micron) D-ring dip net and a 1 m^2 kick net with similar mesh size. Five benthic samples will be collected from each site in any of three possible lotic habitats: shallow riffles, around streamside vegetation in flowing water, and around woody debris in flowing water. In addition, three Hester-Dendy sampling devices (circular 14-plate models) will be placed at each site and will remain for 6 weeks to allow macroinvertebrates to colonize the samplers before collection. Samples can only be collected if it is determined that the stream is at no greater then 3 cm above seasonal base and no sooner than 5 days after a high flow event. This will insure that a representative sample of the macroinvertebrate community will be collected at each location.

Once collected, the samples will be preserved in a 100% ethanol solution and stored in labeled 1-liter plastic jars. Each sample will be shipped to a qualified lab for picking, sorting, identification and enumeration. A chain of custody (COC) document will be completed for each sample and shipped with each sample.

2.1 Sampling Locations

Eight benthic macroinvertebrate samples will be collected from a 100 m reach of stream at each of 10 impacted locations in various streams and tributaries within the Illinois River watershed. Additional macroinvertebrate sampling will be conducted at 2 predetermined reference locations; one reference location within the Illinois River watershed and one outside of the watershed but within the USEPA Eco-region III.

2.2 Sampler Selection

To achieve representative samples of the resident macroinvertebrate populations, both 1-m^2 kick nets and small mesh dip nets will be used at each sampling location. The amount and proportion of each method used will vary depending on site-specific factors that are limiting to the effectiveness of each sampling type. Three Hester-Dendy samples will also be collected at each site to assess macroinvertebrate communities in the stream.

2.3 Data to be Collected

At each sampling location, a variety of physical variables should be recorded in order to quantify factors that may have an influence on the resident macroinvertebrate populations and/or the efficacy of the sampling techniques employed. Variables may include, but are not limited to:

- Average stream width, depth, and velocity.
- Water temperature, conductivity, pH, dissolved oxygen (DO) content, and turbidity value (NTU).

2.4 General Supplies

The following is a list of the minimum supplies needed for a benthic macroinvertebrate sampling event:

- 1 m x 1 m benthic seines (kick nets) with a mesh size of 500 microns
- 34 cm wide, triangular shaped dip-nets (D-ring nets) with a #30 mesh size
- Hester-Dendy samplers (round 14-plate models)
- 1 liter polyethylene jars for each sample
- Tweezers for removing insects from the net
- Sieve bucket with 500 micron mesh
- Ethanol
- Nylon Rope
- Bricks or stones for weights
- Large bucket for in-stream sub-sampling
- Flagging tape
- Stop-watch or timer

- Chain of Custody forms and labels
- Data Sheets on water resistant paper
- Field Notebooks with water resistant paper
- Multi-meter for collecting water parameters
- Tape measure (for measuring width and length of station)
- Handheld GPS Unit, with extra batteries
- Writing utensils (waterproof)
- Digital camera with extra batteries
- Proper eye protection for handling ethanol
- Waders for each crew member
- Applicable scientific collection permits
- Sunscreen
- Insect repellent
- Drinking water/snacks/lunches for the crew

3.0 Sampling Procedures

Benthic macroinvertebrates will be collected across a 100-meter stretch of stream using methods that are in accordance with the Oklahoma Water Resources Board's Beneficial Use Monitoring Program (BUMP) guidelines (OWRB 2001). A total of 8 samples will be collected from each location and benthic processing will follow the USEPA Rapid Bio-assessment Protocols (RBP) multi-habitat approach (Barbour et al., 1999). Appropriate metrics that are relevant to the Illinois River watershed will be used to measure the ecological health of the benthic community.

Prior to sampling, it is necessary to:

- Locate the predetermined site using a hand-held GPS unit.

- Determine if flow conditions are less then 3 cm above the seasonal base flow levels.
- Determine if at least 5 days have passed since the last high-flow event and that it has been at least 2 weeks since the stream has transitioned from no-flow to base flow conditions (OWRB, 2004).
- Measure out and mark the appropriate length of stream to be sampled (100 m).
- Use the multi-meter to measure the various in-stream parameters for the site.
- Record the percentage of each habitat type in the reach and note the sampling gear used (Barbour et al., 1999).

3.1 Collection from Riffles

Collection of benthic macroinvertebrates from riffles will be done with the 1 m^2 kick nets. The most suitable substrate for this sampling technique is composed of gravel or cobble that measures 3-30 cm in the longest dimension. Riffles with substrates composed of bedrock or clay are not suitable for this type of sampling.

A total of three 1 m^2 areas must be sampled at each site. The areas can be of any shape as long as the area is equivalent to 1 m^2. One sample should be in the fastest part of the riffle where the largest rocks and smallest amount of interstitial sediment are found. The second sample should be in the slowest part of the riffle where the smallest rocks and greatest amount of interstitial substrate are found. The third sample should be located in an intermediate area (OWRB, 2004).

These procedures should be followed at each sample location:

- Support the 1 m^2 kick net so that any dislodged organisms will drift downstream into the net. The bottom of the net must be flush against the bottom of the stream and the current must be strong enough to ensure that organisms will be forced into the net.
- Agitate the 1 m^2 area of substrate immediately upstream from the net. This can be done by vigorously kicking the substrate to a depth of at least 12 cm. Continue until all rocks in the area have been agitated to the point that any macroinvertebrates will have been dislodged and will drift into the net.
- Any rocks that are too large to kick should be scrubbed with a brush or by hand on all surfaces.

- Sampling is complete when it is apparent that no new detritus, organisms, or fine sediments are being dislodged.
- Rinse all leaves, sticks, and large debris caught in the net so that the organisms attached to the debris are carried into the sample jar. The volume of the samples should be reduced so that all three $1m^2$ samples will loosely fill a 1 liter jar ¾ of the way.
- After all three samples have been placed in the jar, add 100% ethanol to the jars until full.
- Label the jars properly and fill out a COC for each jar.

3.2 Collection from Streamside Vegetation

Collection of samples from streamside vegetation requires the use of a D-ring dip net. The net should be 34 cm wide and made of #30 size mesh. Only vegetation that is in current and has fine structure that is sufficient to provide habitat for macroinvertebrates should be sampled. This habitat is typically found along undercut banks where the fine roots of grasses, sedges, and trees hang in the water (OWRB, 2004).

The following procedures should be employed:

- Position the dip net completely around the vegetation (or immediately downstream of the vegetation being sampled if the current is strong enough to force dislodged organisms into the net).
- Dislodge organisms attached to the vegetation by shaking the roots by hand or vigorously shaking the net around the roots.
- Total sampling time at each site should be 3 minutes, not to include time spent between sampling areas.
- Rinse all leaves, sticks, and large debris caught in the net so that the organisms attached to the debris are carried into the sample jar.
- After the sample has been placed in the jar, add 100% ethanol to the jar until full.
- Label the jars properly and fill out a COC for each jar.

3.3 Collection from Woody Debris

Collection of samples from woody debris will also require the use of a D-ring dip net. The net should be 34 cm wide and made of #30 size mesh. Samples should be taken from dead wood (with or without bark) that is in current strong enough to provide habitat for organisms that require flowing water to collect particles or for other reasons. Woody debris of all sizes and states of decay should be sampled at each site so that a representative sample is collected (OWRB, 2004).

The following procedures should be employed:

- Dislodge organisms attached to the debris by vigorously shaking the net around the debris or by shaking the debris by hand while it is within the net. Debris that is too large to shake should be rubbed or brushed by hand with the net positioned immediately downstream.
- Total sampling time at each site should be 5 minutes, do not include time spent between sampling areas.
- Include samples from woody debris of as many sizes and states of decay as possible.
- Rinse all leaves, sticks, and large debris caught in the net so that the organisms attached to the debris are carried into the sample jar.
- After the sample has been placed in the jar, add 100% ethanol to the jar until full.
- Label the jars properly and fill out a COC for each jar.

3.4 Hester-Dendy Samplers

The collection of macroinvertebrates from the Hester-Dendy samplers will require two visits to the site. During the first visit, the samplers will have to be placed on site. The samplers will be collected 6 weeks later during the return visit and the macroinvertebrates that have accumulated on the surfaces can be sampled.

The Hester-Dendy samplers should be placed in the stream so that they will remain submerged during the entire incubation period. The water surrounding the sampler should have some current (>0.2 fps) but the sampler should be placed away from the middle of the channel so that it will be less likely to become entangled with debris (CDPR, 2005).

3.4.1 Sampler Deployment
To deploy the Hester-Dendy sampler:

- Tie a brick or large stone to the Hester-Dendy sampler so that the sampler will lie on the bottom of the stream.
- Attach a small float or buoy to the Hester-Dendy sampler with enough rope so that the buoy will remain on the surface in case of a high-flow event.
- Use flagging tape or other markers along stream bank to aid in location of samplers during retrieval.

3.4.2 Sampler Retrieval
To retrieve the Hester-Dendy sampler:

- Carefully approach the samplers without disturbing the water surrounding them.
- Place a D-ring dip net immediately downstream of the sampler to collect any organisms that may detach during removal of sampler.
- Gently detach the sampler from the weight and buoy and remove it from the stream.
- Scrape or brush all surfaces of the plates within the Hester-Dendy sampler and rinse into a sample jar.
- Check the D-ring dip net for any organisms that may have escaped the sampler during retrieval and place them in the sample jar.
- After the sample has been placed in the jar, add 100% ethanol to the jar until full.
- Label the jars properly and fill out a COC for each jar.

4.0 Preservation Techniques and Quality Control

To ensure that all samples are in sufficient condition for picking, sorting, and identification of macroinvertebrates when received by the laboratory, certain procedures must be followed:

- The sample jars must be packed so that the collected materials will loosely fill the jars ¾ full. This will allow for adequate amounts of ethanol to be added for sample preservation.
- After collecting each sample, be sure to fill the remaining ¼ of the jars with 100% ethanol immediately to prevent decay.
- Label all samples on the outside of the jar with sample ID, date, location, time, type of sample (riffle, vegetation, woody debris), and sampler's name. All information should also be written (in pencil) on a small sheet of paper that will be placed in the jar.
- Complete a chain of custody form for each sample location and ship to laboratory for analysis.

5.0 Documentation

Bound field logbooks will be used for the maintenance of field records. All aspects of sample collection and handling as well as visual observations should be documented in the field logbooks. Supplemental information may be documented on benthic macroinvertebrate sampling field sheets provided. All entries in field logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.

6.0 Additional Information

Other contact information:

Sample Verification Lab:
Great Lakes Environmental Center (GLEC)
739 Hastings Street
Traverse City, MI 49686
Contact: Mailee W. Garton, (231) 941-2230
E-mail: mgarton@glec-tc.com

Sample Identification Contact:
Renee Mulcrone- CDM
215 South Washington Square
Suite 160
Lansing, MI 48933

7.0 References

Barbour, M.T., J. Gerritsen, B.D. Snyder, and J.B. Stribling. 1999. Rapid Bioassessment Protocols for Use in Wadeable Streams and Rivers: Periphyton, Benthic Macroinvertebrates, and Fish, 2nd Edition. EPA 841-B-99-002. U.S. Environmental Protection Agency; Office of Water; Washington, D.C.

California Department of Pesticide Regulation (CDPR). 2005. Standard Operating Procedure: Procedure for Collecting Benthic Macroinvertebrates Using a Hester- Dendy Sampler. California Department of Pesticide Regulation; Environmental Monitoring Branch; Sacramento, California.

Oklahoma Water Resources Board (OWRB). 2004. Water Quality Monitoring Program: Field Sampling Protocol for Water Quality Assessments of Streams and Rivers, Draft Copy. Oklahoma Water Resources Board; Water Quality Programs Division; Oklahoma City, Oklahoma.

Rabeni, Charles F. 1996. *Invertebrates.* Pages 335-352 *in* B.R. Murphy and D.W. Willis, editors. Fisheries Techniques, 2nd edition. American Fisheries Society, Bethesda, Maryland.

8.0 Revised Dates*

The following revision dates are applicable to this SOP:

Revision 1 – January 19, 2006

Prepared: Brian Bennett

Review: Tony Gendusa

Approved: [signature]

Date Approved: 6-Feb-2007

1.0 Overview and Application

This standard operating procedure (SOP) describes field procedures used for collection of stream habitat data in the Illinois River watershed of eastern Oklahoma and western Arkansas. In this protocol, the term "habitat" refers to the characteristics of the in-stream and riparian habitat that may influence the biotic community of a stream. This will include the physical characteristics such as stream width, flow, depth, and substrate. Procedures for on site measurement of various water quality parameters such as temperature, conductivity, and dissolved oxygen will also be discussed. This SOP will be used during field work and will follow the standards outlined in the USEPA's Rapid Bioassessment Protocols (RBP) for Use in Wadeable Streams and Rivers (Barbour et al., 1999).

The Illinois River watershed in eastern Oklahoma and western Arkansas has been receiving inputs from agricultural runoff, including inputs from fields where poultry waste has been applied. These wastes, along with the chemical constituents that make up the poultry wastes, have apparently impacted the water quality and sediments in the streams and lakes in the Illinois River Watershed. The purposes of the work being performed are to evaluate and document 1) the linkage and relationship, if any, between the disposal of poultry wastes and environmental contamination within the Illinois River Watershed, and; 2) the resulting harm/injury to resident biotic communities that may have resulted from the disposal of poultry wastes within the Illinois River Watershed.

2.0 Sampling Methods Summary

The methods for conducting a stream habitat assessment can vary greatly in scope and application. Both quantitative and qualitative data are typically collected, but the amount and level of detail is often limited by the available equipment, timeframe, or personnel. This protocol combines aspects of the USEPA's *Rapid Bioassessment Protocols (RBP) for Use in Wadeable Streams and Rivers* and the Oklahoma Water Resources Board's (ORWB) *Field Sampling Protocol for Water Quality Assessment of Streams and Rivers.* A combination of data

sheets adopted from both sources will be used. During stream habitat assessment, physical characteristics and water quality parameters will be measured. A separate, visual-based assessment will also be conducted. These assessments will be done in the field by adequately trained crew members. Water quality parameters will be measured using various portable meters. Stream velocity will be measured using a Marsh-McBirney Flo-mate 2000 portable velocity flow meter.

2.1 Sampling Locations

Water quality parameters will be measured at a minimum of 1 location within the 100m sampling reach at each of the 13 streams. Velocity, width, depth and substrate material will be assessed at 5 transect locations within each reach. At each transect, velocity and depth will be measured at three different points; once in the center of the channel, and once on each side of the center point. Substrate material will be assessed at 10 equally-spaced points across each transect. All visual-based characterizations will also be conducted at each site.

2.2 Data to be Collected

At each sampling location, a variety of physical variables should be recorded in order to quantify factors that may have an influence on the resident biotic communities and/or the efficacy of the sampling techniques employed. Data collected should include, but are not limited to:

- Average stream width, depth, and velocity.
- Water temperature, conductivity, pH, and dissolved oxygen (DO) content.
- Current weather conditions.
- Surrounding watershed features.
- Amount and types of riparian and aquatic vegetation.
- In-stream characteristics (channelization, morphology, etc.).
- Sediment and substrate characters.

- Stream subsystem, type, and origin.
- Map of the sampling reach.
- Visual-based habitat assessment scores.

2.3 General Supplies

The following is a list of the minimum supplies needed for a stream habitat assessment event:

- Multi-meters for collecting water parameters
- Marsh-McBirney Flo-mate 2000 portable velocity flow meter
- Tape measure (for measuring width and length of station)
- F low meter wading staff, calibrated in .1-foot intervals.
- Data Sheets
- Field Notebooks with water resistant paper
- Handheld GPS Unit, with extra batteries
- Writing utensils (waterproof)
- Digital camera with extra batteries
- Sunscreen
- Insect repellent
- Drinking water/snacks/lunches for the crew

3.0 Sampling Procedures

The procedures for conducting stream habitat analysis will consist of several parts. There will be quantitative water quality measurements conducted, visual-based stream habitat

assessments, and miscellaneous site characterizations. Procedures outlined below have been adopted from the RBP and will outline factors involved in filling out the supplied habitat characterization forms.

Prior to sampling, it is necessary to:

- Locate the predetermined site using a hand-held GPS unit.
- Measure out and mark the appropriate length of stream to be sampled (100m).

3.1 Water Quality Measurements

Water quality measurements are to be made using available digital water quality meters. Temperature, specific conductance, pH, and dissolved oxygen (DO) should be measured from a representative habitat within the sampling reach at each site. For most meters, this will involve placing the probe in the water column and recording the parameters once the meter has stabilized. Water temperature should be recorded in degrees Celsius and specific conductance should be recorded in micro-Ohms. Only properly maintained and calibrated meters should be employed. Consult the operating manual for all devices to ensure proper maintenance and use.

Dissolved oxygen may be measured using a Chemetrics V-2000 photometer and the appropriate cartridges. The meter should be zeroed with a blank cartridge regularly for calibration. The operating procedures for this device are somewhat different than most other multi-meters and are outlined below:

- Using the supplied plastic measuring cup, collect approximately 50ml of water from a representative area within the sample location. Be careful to try and limit the amount of oxygenation that may occur through agitating the sample.
- Locate the appropriate DO cartridge and insert the tip of it into the sample.
- Snap off the tip of the cartridge by pushing it against the inside of the cup below the surface of the water. This will allow the sample water to be pushed into the cartridge and mixed with the internal solution.
- Invert the cartridge slowly several times to thoroughly mix the solution and then dry off the outside of cartridge.

- Place the cartridge into the photometer and set the program to measure DO.
- Replace the cover and initiate a reading.
- Results will be displayed in 2 minutes and should be recorded in parts per million (ppm).

3.2 Collecting Flow and Discharge Data

In order to estimate the discharge of a stream, average width, depth, and velocity must be calculated. To get estimates on these parameters, 5 cross-current transects will be conducted at each site. These transects will be equally spaced throughout the 100-meter reach of stream. Measurement procedures are outlined below:

- At each transect, measure the wetted width of the stream perpendicular to the current direction.
- At the midpoint of the stream, use the graduated wading staff to measure the depth. Then place the probe attached to the Marsh-McBirney at approximately 6/10 of the depth from the bottom.
- Face the front of the probe upstream and allow the flow meter to stabilize and record the velocity reading.
- Repeat the measurements at points halfway between the midpoint and each bank.

Once three points have been measured at each of the five transects, it will be possible to compute the average width, depth, and velocity. These data will enable the computation of an estimate of discharge volumes for each site.

3.3 Collecting Substrate Data

In order to estimate the types and percentages of various substrates present at each sampling location, a Wolman pebble count will be conducted. At each of the five transects used for flow and discharge measurements, a total of 10 equally distributed point samples will be taken. To conduct a Wolman pebble count:

- Estimate where the 10 equally distributed sample points should be located based on the width of the stream at each transect.
- Wade out to the first point, and without looking down, use a pointed stick or pole to reach down through the water and touch the substrate.
- Then reach down at the point you are touching with the pole and examine the substrate so that you can record which of the seven types of substrate it represents (bedrock, boulder, cobble, gravel, sand, silt, or clay).
- Repeat this process at each of the 10 points in all 5 transects (McMahon, 1996).

This will result in a total of 50 sampling points for each 100m reach. This information can be used to estimate the percentage of each type of substrate found in the stream sampling location.

3.4 The Visual-Based Habitat Assessment

The visual-based habitat assessment consists of a matrix of habitat factors that can be qualitatively evaluated and scored in order to compare habitat quality between multiple sites. This approach tends to be rather suggestive and different examiners are likely to score the same site differently. Therefore, it is recommended that the visual habitat assessment be conducted by the same, properly trained individual or group at each location. This should help to assure a more consistent scoring regime and will allow for more accurate comparisons to be made.

The habitat assessment matrix is adopted from the RBP and both low and high gradient assessments may be completed. Each parameter is assessed and a score of 1-20 is assigned based on the observer's assessment. The following is an outline of the 9 parameters to be assessed on each sheet:

1. **Epifaunal Substrate/Available Cover-** the amount and variety of natural structures in the stream suitable for refuge, feeding or spawning sites. Structures may include cobble in riffles, large rocks, woody debris, and undercut banks.
2. **Embeddedness or Pool Substrate Characterization-** embeddedness is evaluated in high gradient areas and refers to the extent to which rocks and snags are covered in

finer, sediment materials. In low gradient areas, the pool substrate characters are evaluated for variety and firmness.

3. **Velocity/Depth Combinations or Pool Variability-** in high gradients, the patterns of velocity and depth (slow-deep, fast-deep, slow-shallow, fast-shallow) are assessed. For low gradients rates of the four pool types (large-deep, small-deep, large-shallow, and small-shallow) are examined.

4. **Sediment Deposition-** evaluates the amount of sediment that has accumulated in pools and any changes to the stream that have occurred as a result of large-scale sedimentation.

5. **Channel Flow Status-** the degree to which the stream channel is filled with water.

6. **Channel Alteration-** a measure of the large-scale changes in the shape of the stream channel.

7. **Frequency of Riffles or Channel Sinuosity-** in high gradient areas, the frequency of riffles is used to asses the heterogeneity of the available habitats in the stream. For areas with a lower gradient, the curviness or sinuosity of the stream is used because this will reflect the variability of habitat types within the stream.

8. **Bank Stability-** a measure of the condition of the stream banks and the amount or potential for large-scale erosion. Each bank is scored separately.

9. **Width of the riparian Vegetative Zone-** this is an estimate of the distance from each bank a zone of naturally occurring vegetation is found and is scored separately for each bank.

Each parameter is described in more detail on the data scoring sheets and an extensive explanation of each characteristic is given in the RBP (pages 5-10 thru 5-30). Before completing a visual-based evaluation, the observer should become familiar with all aspects of the scoring guidelines as described in the RBP.

3.5 Other Analyses

Several other parameters of a streams habitat quality may be assessed for each site. These may be either quantitative or qualitative, and may require knowledge of other areas or features in the watershed. Such parameters should include, but are not limited to:

- **Stream subsystem-** is the stream flow perennial, intermittent, or tidal.
- **Stream type-** is it a warm-water or coldwater stream.
- **Stream origin-** is the main source of the stream's water glacial, a swamp or bog, spring-fed, run-off, or a mixture of origins.
- **Stream Order**
- **Predominant surrounding land usage-** is the watershed primarily forest, commercial, industrial, residential, or agricultural land.
- **Riparian vegetation-** what are the most dominant types of riparian vegetation: trees, shrubs, grasses, or herbaceous plants. The dominant species present may also be noted, if known.
- **Aquatic Vegetation-** indicate the dominant type (rooted-emergent, rooted- submerged, rooted-floating, free floating, floating algae, and attached algae).
- **Woody debris-** an estimate of the amount or area of the woody debris found in a stream.
- **Periphyton color and texture-** green, brown, yellow, and slimy, discoloration, gelatinous, filamentous, or others.
- **Turbidity-** can be directly measured or qualitatively described (clear, slightly turbid, turbid, stained, or opaque).
- **Weather conditions-** air temperature, precipitation, cloud cover, wind speed and direction.
- **Site Map-** a detailed drawing of the sample site should be made. It should include distinct features, sampler placements, and flow regimes.

All of these factors may have an impact on the quality of habitat available for aquatic organisms or on the ability of some parameters to be measured. In general, as much of the supplied data sheets as practical should be filled out for each site. Appropriate headers should also be completed to include station ID, stream name, times, dates, location, and crew members present.

4.0 Quality Control

To ensure that quality data is collected at each site, a number of precautions must be taken. All instruments used in water quality or flow velocity measurements should be properly maintained and regularly calibrated. Members of the field crew should be fully trained and evaluated on each task they will perform. The visual-based assessment should consistently be done by the same, properly trained individual in order to limit the suggestiveness of this analysis. The procedures laid out in this and other applicable protocols (RBP, ORWB) should be examined and followed to ensure that quality data is acquired.

5.0 Documentation

Bound field logbooks should be used for the maintenance of field records. All aspects of sample collection and handling as well as visual observations shall be documented in the field logbooks. Supplemental information may be documented on the field data sheets provided. All entries in field logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.

6.0 Additional Information

Other contact information:
Ronald French
CDM
100 North Tucker Blvd.
Suite 550
Saint Louis, MO 63101
Telephone: 314-241-8510

Bert Fisher, PhD
Lithochimeia, Inc.
222 South Kenosha Ave.
Tulsa, OK 74120
Telephone: 918-382-9784

STOK0020913

7.0 References

Barbour, M.T., J. Gerritsen, B.D. Snyder, and J.B. Stribling. 1999. Rapid Bioassessment Protocols for Use in Wadeable Streams and Rivers: Periphyton, Benthic Macroinvertebrates, and Fish, 2nd Edition. EPA 841-B-99-002. U.S. Environmental Protection Agency; Office of Water; Washington, D.C.

McMahon, T.E., Zale, A.V., and D.J. Orth. 1996. Aquatic Habitat Measurements. Pages 83-120 *in* Murphy, B.R., and D.W. Willis, editors. 1996. Fisheries techniques, 2nd Edition. American Fisheries Society, Bethesda, Maryland.

Oklahoma Conservation Commission (OCC). 2002. Periphytometers & Processing for Chlorophyll-a Measurement. SOP No: IIA-06.1. Oklahoma Conservation Commission, Water Quality Division, Oklahoma City, Oklahoma.

8.0 Revised Dates*

The following revision dates are applicable to this SOP:

Revision 1 - January 19, 2006

Prepared: Brian Bennett Review: Ron French

Approved: Roger L. Olsen Date Approved: 2/06/07

1.0 Overview and Application

This standard operating procedure (SOP) describes field procedures used for sampling during the 2006 River and Biological sampling program. The purpose of these biological investigations is to determine the level of biological productivity (eutrophication) associated with various water quality parameters, primarily; phosphorus, nitrogen, and dissolved oxygen. A 2-phased approach will involve preliminary site investigations of over 200 stream locations followed by more intensive water quality and biological community assessments of approximately 70 sites within and around the Illinois River watershed. In the first phase, sampling teams will be sent to select locations throughout the watershed to collect field water quality measurements including pH, dissolved oxygen (DO), water temperature, conductivity, and turbidity. Water samples will also be collected at each site for analysis of phosphorus concentrations. During the second phase, up to 70 of the original locations may be selected for additional surface water sampling along with benthic and sestonic chlorophyll analysis, macroinvertebrate community sampling, algal community sampling, and habitat evaluations. This SOP will be used during sampling and is based in part upon the USEPA's Rapid Bioassessment Protocols (RBP) for Use in Wadeable Streams and Rivers (Barbour et al., 1999). Some modifications to these protocols were made based upon direct consultation with Dr. R. Jan Stevenson of Michigan State University.

The Illinois River watershed in eastern Oklahoma and western Arkansas has been receiving inputs from agricultural runoff, primarily from fields where poultry waste has been applied. These wastes, along with the chemical constituents that make up the poultry waste, have impacted the water quality and sediments in the streams and lakes in the Illinois River Watershed. The purposes of the work being performed are to evaluate and document 1) the linkage and relationship between the disposal of poultry wastes and environmental contamination within the Illinois River Watershed, and; 2) the resulting harm/injury to natural systems that may have resulted from the disposal of poultry wastes within the Illinois River Watershed.

2.0 Sampling Methods Summary

The 2006 River and Biological sampling program will consist of 2 phases to be conducted in late summer of 2006. The first phase will consist of an initial screening for water quality parameters at a number of locations throughout the Illinois River watershed as well as at several comparison locations outside the watershed but within USEPA Eco-region III. Data from this initial investigation may be used to select sites for the second phase of sampling. Phase 2 of the River and Biological sampling will consist of habitat, biological, and water quality assessments at approximately 70 locations.

2.0.1 Initial Water Quality Investigations (Phase 1)

Each of the locations identified for sampling during Phase 1 of the investigation will be visited and a variety of water quality measurements will be collected. All sites will be visited between 5am and 9am in an attempt to document the lowest daily concentrations of dissolved oxygen. An Oakton model 300 multi-meter will be used to collect pH, conductivity (μS), and water temperature (^{o}C) measurements in the field. Dissolved oxygen will be measured using a Chemetrics V-2000 photometer and Vacu-vials. Initially, Oxygen 2 Vacu-vials with a range of 2 - 15ppm will be used to evaluate DO concentrations. If the concentration of DO at any site is less then 2ppm, Oxygen 1 Vacu-vials (0.2 - 2ppm) will be employed. Turbidity will be measured in the field using a Hach 2100p portable turbidimeter or equivalent. Additionally, water grab samples will be collected at each location, put on ice, and transported to a fixed field laboratory for analyses of phosphorus.

At each sample location, field teams will also complete a basic habitat evaluation form which will include a site map, general description of flow regime, and a location description. A handheld GPS unit may be employed to mark the sampling point for each site. Digital photographs may also be taken at each site for future reference.

During Phase 1 of the investigation, a field laboratory will be established at a fixed location to assist in sampling logistics and to perform various preliminary laboratory procedures. At the fixed based field laboratory, the water samples collected will be analyzed for reactive phosphate using a HACH DR2800 or DR5000 spectrophotometer and AccuVac ampules. Initially, method 8048 will be used (0.020 to 2.00 mg/L, molybdate in ascorbic acid, blue color measured at 880 nm). If concentrations are greater than 2 mg/L, method 8114 will be used (0.3 to 45 mg/L, molybdovanadate in acid, yellow color measured at 420 nm). As soon as the phosphorus results are available, the values of phosphorus and DO will be evaluated to assist in determination of the locations of potentially impacted rivers and streams.

2.0.2 Secondary Biological Screening (Phase 2)

Based upon the results of the preliminary investigation as well as on other factors (i.e. chicken house density), approximately 70 locations from Phase 1 will be selected as sites for secondary biological screening. This second round of sampling will consist of biological sampling of periphyton and macroinvertebrates, benthic and sestonic chlorophyll *a* sampling, and additional water quality sample collection. The periphyton community will be assessed by determining the percent of periphyton coverage on in-stream substrate at each site using a procedure developed by Dr. R. Jan Stevenson and outlined in the USEPA Rapid Bioassessment Protocols. This procedure involves using a viewing bucket marked with a grid and a biomass scoring system to conduct a semi-quantitative measurement of the periphyton community at each location. At least one sample of periphyton will be sent to a lab for species identification and density.

Macroinvertebrates samples will be collected at 3 randomly chosen locations within each sample site. A 1 meter squared kick net will be used at each spot with a timed sediment agitation interval. Macroinvertebrates will be collected and preserved with isopropyl alcohol. The samples will later be sorted and identified to the lowest possible taxa at an offsite location.

Benthic chlorophyll samples may be collected using an SG-92 or similar device to remove and collect the periphyton from a known area of a cobble substrate. Each sample location will be divided into 5 cross-channel transects within areas of consistent flow (i.e. riffles). At each of the 5 transects, 5 rocks will be randomly selected and periphyton will be removed from a known surface area of each of these rocks. The scrapings will then be combined into a brown bottle and frozen until shipment to a laboratory for analysis. A total of 5 benthic chlorophyll samples will be collected at each location (i.e. one sample from each transect). A 500ml water sample will also be collected from each site for sestonic chlorophyll analysis. This sample will be homogenized, preserved with $MgCO_3$, and filtered onto a wetted 0.7 micron, glass fiber filter pad. This filter will then be wrapped in foil and frozen until shipped to a laboratory for analysis.

Additional water samples may be collected from phase 2 sites and be sent to a qualified laboratory for a additional analyses. The analyses for these samples may include, but are not limited to:

- Total phosphorus, dissolved total phosphorus, and soluble reactive phosphorus
- Total Kjeldahl nitrogen and inorganic nitrogen (nitrate + nitrite)
- Total organic carbon (TOC)

At approximately 40 of the biological sampling locations, additional water samples will be collected and analyzed for a full suite of chemical parameters. These parameters may include, but are not limited to:

- Dissolved metals and total metals
- General water quality parameters (sulfate, chloride, bicarbonate/carbonate)
- Bacteria, Estrogens, and potentially PCR

At each secondary biological screening site, crew members will also conduct a more detailed habitat evaluation and standard forms will be completed. The data to be recorded will include percent canopy cover, width and depth of stream, substrate characteristics, flow direction, estimated flow velocity, etc. A site map will be drawn and GPS coordinates will be recorded at the site of sample collection. Photographs of the sites will also be taken.

2.1 Sampling Locations

Approximately 300 locations will be identified as potential sampling sites for Phase 1 investigations. A majority of sites will be located within the Illinois River watershed; however several sites may be selected as comparison locations which are outside of the watershed but within USEPA Eco-region III. Prior to sampling, sites will be selected based on the following criteria:

- Historical water quality data including past results from this study
- Proximity to various waste application sites
- Density of chicken houses within the sub-basin
- Accessibility by field staff
- Beneficial use of stream and applicable state water quality standards/criteria
- Streams that cross the Oklahoma/Arkansas state line
- Land use and degree of human activity

The primary criteria for selecting sites will be chicken house density in the sub-basin and accessibility from public property, typically from bridges and road crossings. Sites initially selected for sampling may be removed from the list if discovered to be inaccessible or without adequate flow during field visits to the sites. If possible, a relatively consistent number of sampling locations will be selected from each range of chicken house densities (0-1, 2-4, 4-6, 6-8, 8-10, and >10 chicken houses per square mile).

Phase 2 sampling locations will be a subset of the locations from Phase 1 and will be identified based on similar criteria. The results of Phase 1 field measurements of dissolved oxygen and phosphorus may be used as additional criteria for site selection during the secondary biological screening. Some of the selected sites may not be suitable for biological screening based on habitat and flow characteristics and may be replaced with alternative locations during field sampling.

Benthic chlorophyll sampling, periphyton viewing bucket transects, and macroinvertebrate sampling will be conducted in areas of sufficient flow (primarily riffles) as near to bridge or road crossing as possible. Backwater areas and stream segments without any discernable flow will not be characterized or sampled for these parameters. Typically one or two riffles will be sampled at each site, with transects being established at equal intervals along the reach.

2.2 General Supplies

The following is a list of the minimum supplies needed for initial water quality investigations (Phase 1):

- Oakton model 300 temperature/pH/conductivity meter
- Chemetrics V-2000 photometer and Vacu-vials (Oxygen 1 and Oxygen 2 vials)
- Hach 2100p portable turbidimeter or equivalent
- Hach DR2800/5000 spectrophotometer and AccuVac ampules (field laboratory only)
- Sample bottles with labels
- Sterile nitrile or latex gloves
- Data sheets on water resistant paper

- Field notebooks with water resistant paper
- Chain of custody forms and labels
- Handheld GPS unit, with extra batteries
- Digital camera with extra batteries

The following is a list of the minimum supplies needed for secondary biological screenings (Phase 2):

- Scraping tools (e.g., toothbrushes, razor blades)
- SG-92 periphyton sampler or equivalent
- 1/4 inch sections of PVC pipe (1.5" diameter)
- Suction bulbs or syringes
- Preservative (Lugol's solution or 10% formalin solution)
- Vacuum pump, filter apparatus, filters, foil, $MgCO_3$ (field laboratory only)
- Sterile nitrile or latex gloves
- Chain of custody forms and labels
- Data sheets on water resistant paper
- Field notebooks with water resistant paper
- Tape measure (for measuring width and length of station)
- Handheld GPS unit, with extra batteries
- Writing utensils (waterproof)
- Coolers with ice

- Digital camera with extra batteries
- Applicable scientific collection permits with team member who appears on permit

3.0 Sampling Procedures

Specific procedures for sampling during each phase of the river and biological sampling program are described in the following sections.

3.1 Sample Collection During Initial Water Quality Investigations (Phase 1)

Field teams will consist of at least 2 members and will be assigned sites to visit each morning. All sites will be visited between 5am and 9am in order to assess the lowest daily concentrations of dissolved oxygen. Field crews will be given the locations and GPS coordinates for each site by a field manager. Once on site, the crews will assess the current flow conditions to determine if there is adequate surface water at the site to enable sample collection. The crews will fill out the supplied sample location data form. Photos and GPS coordinates will be taken of all sites. If water is present, the crews will collect water samples and take field parameter measurements at each site.

3.1.1 Surface Water Sampling

Water samples will be collected by taking a direct grab sample. Crew members will use sterile nitrile/latex gloves and fill the sample containers by submerging them in an area of representative stream flow until container is filled. If the water levels are insufficient to fully submerge the container, a properly decontaminated intermediate container may be used to dip and pour into the sample containers. Care should be taken to minimize any disturbance of the sediments in the vicinity of sample collection. All grab samples should be taken while facing upstream so as to minimize any impact of the crew member on the sample. Once the sample containers have been filled, the field team will properly seal and label the bottles and place them in a cooler on ice until returned to the lab for processing.

3.1.2 Water Quality Measurements

Field water quality parameters will be measured using the Oakton temperature/pH/conductivity meters, the Chemetrics spectrophotometer, and Hach turbidimeters. All meters should be calibrated and maintained as described in the manufacturers' manuals and in Standard Operating Procedure 8.0: Water Quality Meters. Measurements should be taken in areas of

representative flow at each site, as near to the water sample location as possible. Care should be taken to minimize any disturbance of the sediments in the area of parameter measurements. A brief outline of the procedures required for each instrument is provided below:

- *Oakton model 300 temperature/pH/conductivity meters:*
  Field measurements using the Oakton multi-meters are collected by placing the probes attached to the handheld read out device directly into the stream. The probe should rest on the stream bottom above any soft sediment or debris. Once in position the handheld should be turned on and after the values stabilize, readings may be recorded on the field data sheets. Temperature is recorded in degrees Celsius (°C), pH in standard units, and conductivity in micro-siemens per centimeter (μS/cm).

- *Chemetrics V-2000 photometer and Vacu-vials for dissolved oxygen (DO) measurements:*
  To accurately measure concentrations of dissolved oxygen using a Chemetrics photometer, a 25ml sample of the stream water should be collected in the small graduated cylinder supplied with the Vacu-vials. Care should be taken to avoid disturbing the water in the vial and possibly oxygenating the sample. Immediately after collecting the water, an Oxygen 2 Vacu-vial (2.0 - 16.0ppm) should be inserted into the sample and the glass tip must be broken off to allow the water to flow into the vial, mixing with the Oxygen 2 solution. The vacu-vial should be gently inverted several times to allow mixing and wiped clean with a lint-free cloth. The vacu-vial will then be placed in the V-2000 photometer and covered with the supplied cap. The Chemetrics meter can be powered on and program 751 selected (Oxygen 2). Once initiated, the program will take a sample measurement after a 2 minute holding time and results will be displayed in parts-per-million (ppm) of dissolved oxygen concentrations. If an out of range error is reported, the procedure should be repeated with the Oxygen 1 Vacu-vials (0.2 - 2.0ppm) and corresponding program 750 on the V-2000 photometer. Once a reading has been obtained, the crew member will record the measurement on the datasheet.

- *Hach 2100p portable turbidimeter:*
  Turbidity measurements using the Hach 2100p turbidimeter will also require collecting a 25ml sample of the stream water. This sample should be collected and poured into a sample cell. Sample cells should be clean and free of any scratches in order to obtain the most accurate readings. Once filled, the sample cell should be gently tapped to remove any air bubble that may have formed inside the cell. The cell should then be wiped with a soft, lint-free cloth to remove any water, smudges, or fingerprints on the outside of the sample cell. A thin film of the supplied silicone oil should be applied to the sample cell before placing it in the turbidimeter. Once the sample cell has been loaded, the cap

should be closed and the turbidimeter powered on. The measurement is started after the crew member presses the read button and results will be displayed in nephelometric turbidity units (NTU). This reading may then be recorded on the field data sheet.

3.2 Sample collection and site assessments during Secondary Biological Screening (Phase 2)

During Phase 2 sampling, field teams will consist of 1 field biologist and 1-2 additional field crew members. The field biologist will be responsible for making habitat and biological observations at each site using both qualitative and quantitative methods. Other crew members will assist and will record data in the field book and on the data sheets. At each site, water samples will be collected, a rapid periphyton survey will be conducted, and benthic macroinvertebrate and benthic chlorophyll samples will be collected.

3.2.1 Surface Water Sampling

The collection of water samples at each site selected for secondary biological screening will be used to verify the results of the Phase 1 samples and to provide media for an extended list of analyses. The samples will be collected in a similar location and manner as those collected during Phase 1 sampling. A direct grab method will be employed, and all samples will be properly labeled, preserved (if necessary), and stored on ice until returned to field laboratory. Samples collected for sestonic chlorophyll *a* analysis will be preserved with $MgCO_3$ and filtered onto 0.7 micron glass fiber filters at the field lab. The filters may then be sealed in aluminum foil, labeled, and frozen until shipment to an analytical laboratory.

3.2.2 Rapid Periphyton Surveys

Periphyton communities at each site will be assessed using semi-quantitative measurement procedures developed by DR. R. Jan Stevenson of Michigan State University and outlined in the USEPA Rapid Bioassessment Protocols (1999). A viewing bucket will be marked with a grid consisting of 50 points and will be used to make observations of the periphyton community in areas of relatively high flow at each site, primarily riffles or runs. Approximately 20 observations will be made at each site. Within each stream reach, a 5-10 transects that cross perpendicular to the direction of stream flow will be established. Along each transect, 3-5 observations will be made at 1-2 meter intervals, depending on stream width.

The viewing bucket is placed in the water at each interval along the transect and will be used by the field biologist to make observations of the periphyton community. The observer should be facing upstream and should avoid looking at the substrate prior to placing the bucket in order

to limit any operator bias. The observer will count the number of dots on the grid over any algal cover, what general type of cover is present (diatoms, cladophora, spirogyra, vaucheria, general blue-green algae, aquatic moss, etc.), and the approximate length/thickness of the algae at the point. Length or thickness of the algae at any point will be estimated by dividing the microalgal thickness into 6 classes as shown in **Table 1.**

Table 1: Microalgal thickness classification system.

Class	0	1	2	3	4	5	6
Approximate Thickness	0mm	0.1 mm	0.1 - 0.5mm	0.5 - 2mm	2 - 5mm	5 - 20mm	>20mm
Surface Characteristics	Rough, no visible biofilm	Slimy; no visible tracks evident if scraped	Biofilm visible; tracks evident if scraped	Measurable length or thickness of algae			

The number of points covering substrate suitable for macro-algal growth will also be noted on the data sheet. Suitable substrate is defined as being greater then 1 cm in the smallest dimension observed from viewing bucket (i.e. length or width, not depth).

At the center point of each transect, the observer will use a hand-held canopy densitometer to estimate the canopy cover at that transect location. The device is held out in front of the observer at shoulder level and the grid on the reflective surface is used to estimate percent canopy cover by counting all squares that contain a majority of canopy in the reflection. The observer should take care to make sure the densitometer is held level. One reading should be made facing upstream, one facing the right bank, and one facing the left bank from the center of each transect. If transects are very close together, a single canopy reading should be sufficient.

All data will be recorded on the field data sheet along with the depth category of the stream (0-10cm, 10-20cm, 20-50 cm, 50-100cm, or greater then 1m). An estimate of current velocity will also be made and the velocity category will be record as: 0 if there is no current, 1 if current is 0-5cm/s, 2 if current is 5-15cm/s, 3 if current is 16-50cm/s, and 4 if faster then 50cm/s.

3.2.3 Benthic Chlorophyll *a* Sampling

At each of the sample sites, a total of 5 benthic chlorophyll *a* samples will be collected. If possible, one benthic chlorophyll *a* sample should be collected for each transect created during the rapid periphyton survey. The samples will be composite samples of periphyton scraped from 5 separate rocks in each transect. The rocks will be selected by randomly picking suitable

substrate from across each transect. Suitable substrate will consist of cobbles with a surface area sufficiently large to allow for the scraping and removal of a periphyton from a fixed area.

Once the rocks have been collected for a given transect, an SG-92 periphyton sampler or a 1.5-inch diameter ¼ inch ring of PVC pipe will be positioned on the upper surface of the rock. All of the attached algae within this circle will be removed and collected in the sample container. This can be accomplished by using razor blades and/or hard-bristled toothbrushes to dislodge the algae and then rinsing the dislodged algae into a container using a syringe and a small volume of distilled water. Once all of the periphyton has been removed, the rinse water can be collected and placed into a sample container to be composited with the rinse water from the other 4 rocks in the transect.

After all 25 rocks have been scraped and all 5 sample bottles have been filled, a syringe can be used to collect an aliquot of each sample to be used for periphyton identification and enumeration. The sample bottles should be mixed thoroughly and approximately 5-10ml of rinse water should be removed from each sample container and placed into the periphyton identification sample. This sample can then be labeled, preserved with 2% formalin solution, and shipped to a laboratory for taxonomic identification. The volume in all samples containers and the measured surface areas scraped should be recorded in the field book.

3.2.3 Benthic Macroinvertebrate Sampling

At each sample site, benthic macroinvertebrate samples will be collected from the richest habitat (i.e. riffle) at 3 randomly chosen locations within the sample reach. The macroinvertebrates will be collected by agitating a 1-meter square area of substrate directly upstream of the kick net and allowing the current to force the dislodged macroinvertebrates into the net. The amount of effort for agitating the substrate will be limited to 1 minute at each of the 3 locations.

Once captured in the kick net, the macroinvertebrates will be rinsed into a 5-gallon bucket before being carefully transferred into a 1 quart sample container. Large debris (leaves, sticks, etc.) will be rinsed and removed from the sample. After all three sub-samples have been collected; the sample container will be filled with isopropyl alcohol in order to preserve the organisms. Containers will then be labeled and sent to a CDM laboratory to be sorted and identified. A more detailed description of the procedures for sampling benthic macroinvertebrates can be found in *Standard Operating Procedure 7-3: Macroinvertebrate Sampling*.

4.0 Sample Containers, Preservation Techniques, Quality Control

Periodically throughout the sampling, samples will be packed and shipped in coolers to one of several different analytical laboratories (contact information below) depending on analyses required. Preservation techniques will also vary depending on the analyses required. **Table 2** presents a summary of sample types, preservation techniques, and shipping destination for all samples collected in the field. Surface water samples sent to the CDM laboratory in Denver will be preserved and shipped to the appropriate analytical laboratory based on analyses required for each site.

Table 2: Summary of preservation techniques and shipping destinations for each sample type.

Sample Type	Preservation Techniques	Shipping Destination
Benthic Chlorophyll	Freeze samples	Aquatec Biological Laboratory
Sestonic Chlorophyll	$MgCO_3$, filter sample, wrap filter pad in foil, freeze filter pad	Aquatec Biological Laboratory
Periphyton Identification	2% Formalin solution	Jan Stevenson (Michigan State University)
Macroinvertebrate Identification	Isopropyl alcohol	CDM- Lansing (Renee Mulcrone)
Bacteria Analysis	On ice (<4°C)	Environmental Microbiology Laboratory (EML)
Water Chemistry Parameters	On ice (<4°C)	CDM- Denver Laboratory (Todd Burgesser)

Field control samples may be collected by the sampling team and/or created by the CDM laboratory (Denver) to determine whether data are of suitable quality. Control samples may include trip blanks, duplicates, decontamination (rinsate) blanks, or split samples. Duplicates (either co-located or split samples) should be collected at least once for every twenty samples.

5.0 Documentation

Bound field logbooks will be used for the maintenance of field records. All aspects of sample collection and handling as well as visual observations should be documented in the field logbooks. Supplemental information may be documented on sampling field sheets provided. All entries in field logbooks should be legibly recorded and contain accurate and inclusive

documentation of an individual's project activities. Digital photographs and GPS coordinates may also be used to document each sample location.

6.0 Additional Information

Laboratory contact information:

Aquatec Biological Sciences
273 Commerce St.
Williston, VT 05495
Contact: Jennifer Gallant or Phil Downey, (802) 860-1638
E-mail: jgallant@aquatecb.com

Environmental Microbiology Laboratory (EML)
1150 Bayhill Drive, Suite 100
San Bruno, CA 94066
Contact: Cole Mackelprang, (858) 268-2762
E-mail: cmackelprang@emlab.com
Contact: Megan S. Tatreau, 858-268-2770
E-mail: mtatreau@emlab.com

Other contact information:

CDM- Denver Laboratory
2714 Walnut Street
Denver, CO 80205
Contact: Todd Burgesser, (303) 298-1311
E-mail: BurgesserTE@cdm.com

CDM- Lansing Office
215 South Washington Square, Suite 160
Lansing, MI 48933
Contact: Renee Mulcrone, (517) 702-1213
E-mail: MulcroneRS@cdm.com

Lithochimeia, Inc.
222 South Kenosha Ave.
Tulsa, OK 74120
Contact: J. Berton Fisher, (918) 382-9784
E-mail: bfisher@lithochim.com

7.0 References

Barbour, M.T., J. Gerritsen, B.D. Snyder, and J.B. Stribling. 1999. Rapid Bioassessment Protocols for Use in Wadeable Streams and Rivers: Periphyton, Benthic Macroinvertebrates, and Fish, 2nd Edition. EPA 841-B-99-002. U.S. Environmental Protection Agency; Office of Water; Washington, D.C.

Kentucky Department of Environmental Protection (KDEP). 2002. Methods for Assessing Biological Integrity of Surface Waters. Kentucky Department of Environmental Protection, Division of Water, Frankfort, Kentucky.

Stevenson, R.J. and L.L. Bahls. 1999. Periphyton protocols. In M. T. Barbour, J. Gerritsen, & B.D. Snyder, eds. *Rapid Bioassessment Protocols for Use in Wadeable Streams and Rivers: Periphyton, Benthic Macroinvertebrates, and Fish.* Second Edition. EPA 841-B-99-002 United States Environmental Protection Agency, Washington. pp 6-1 through 6-22.

Prepared: Jason Mills Review: Andrew Santini

Approved: Roger L. Olsen Approved Date: 02/06/07

1.0 Overview and Application

This standard operating procedure (SOP) describes field procedures for using an electronic water quality meter to conduct on-site measurements of surface waters. This will include calibration and maintenance procedures. Specific procedures for on-site measurement of various water quality parameters at various sample locations are discussed in other SOPs. This SOP will be used during field work and will follow the guidelines described in the *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual.* (USEPA, 1996).

The procedures in this document are designed for use with multi-parameter water quality instruments, such as the YSI-650 and In-situ Troll 9500. These procedures are generally applicable for most types and models of electronic water quality measurement devices. However, if a different device is used, as in the case of the optical dissolved oxygen sensor on the Troll 9500, the operation manual for that instrument should be consulted and maintenance or calibration procedures in that document should be followed.

2.0 Calibration Procedures

All electronic water quality instruments will be calibrated on a regular basis in order to assure that accurate readings are being made and that the instruments are functioning properly. It is recommended that parameters be calibrated before every sampling event and verified after every event.

Calibration procedures will differ for each parameter being measured, but will generally consist of three parts:

- Initial Calibration (IC): The instrument or meter electronics are adjusted (manually or automatically) to a theoretical value or a known value of a calibration standard.

- Initial Calibration Verification (ICV): Immediately after the initial calibration, the meter is checked or verified by measuring a calibration standard of known value and comparing the measured result to the calibration acceptance criteria listed in this SOP.

- Continuing Calibration Verification (CCV): After field use, the instrument calibration is verified by measuring a calibration standard of known value and comparing the measured result to the calibration acceptance criteria listed in the SOP.

The calibration acceptance criteria are a range of values that the instrument reading must fall within when measuring a standard. **Table 1** contains the calibration acceptance criteria for all parameters measured during the course of this project. If the meter fails to meet these acceptance criteria, it should be recalibrated before use. Calibration standards should not be reused for initial calibrations.

A bound field book or data sheets will be used to record each parameter's IC, ICV, and CCV readings each time the instrument is calibrated. Additional information such as the serial number of the instrument, date and time of calibrations, and user's name should also be recorded in this log. An example log is shown in **Figure 1**.

Table 1. Calibration acceptance criteria for field water quality parameters.

Parameter	Acceptance Criteria
pH	± 0.2 Standard pH units of buffer
Specific Conductance	± 5% of standard value
Temperature	± 0.2° C of NIST-traceable value (with correction factors)
Dissolved Oxygen (DO)	± 0.3 mg/L of theoretical value (see Table 2)
Turbidity	± 5% of standard value
Oxidation Reduction Potential	± 5% of standard value

2.1 Dissolved Oxygen Calibration

Calibrating an electronic water quality instrument to accurately record dissolved oxygen (DO) measurements is somewhat different then calibrating for most other parameters. For membrane technology using YSI-650, DO is calibrated using water saturated air as a baseline measurement. The procedures for initial calibration are outlined below:

- Wet the inside of the calibration chamber with water, pour out the excess water (leave a few drops), wipe any droplets off the membrane/sensor and insert the sensor into the chamber (this ensures 100% humidity).

- Allow adequate time for the DO sensor and the air inside the calibration chamber to equilibrate.

- Once the probe/calibration chamber is stable at ambient temperature, check the air temperature and determine, from the DO versus temperature table (**Table 2**) what the DO should measure. A stable and accurate temperature is required for a valid calibration. The acceptance criterion for DO calibration verification is +/- 0.3 mg DO/L.

- If the acceptance criterion is not met, manually calibrate the DO setting to read the appropriate 100% saturation level for the ambient temperature. Consult the instrument's operation manual for specific instructions on how manually adjust this reading.

Once the initial calibration has been completed, conduct initial calibration verification by re-taking the DO measurement and checking that saturation (mg DO/L) against the 100% saturation number from Table 2. After field use, continuing calibration verification should be conducted (usually within 24 hours). Record all readings on the calibration record sheet.

For optical DO technology using In-Situ Troll 9500, a two-point calibration will be performed at 100% and 0%. 100% DO calibration is performed in water saturated with air, while 0% calibration is performed in an oxygen depleted solution, i.e. sodium sulfite. The procedures for calibration are outlined below:

- Submerge optical DO sensor into a container of distilled water that is aerated using In-situ's DO bubbler assembly (or aquarium pump). It may necessary to aerate the water for five minutes or longer to stabilize the water at 100% oxygen saturation.

- After readings are stable, calibrate the sensor to 100%. Then, remove the optical DO sensor from the distilled water and immerse into a cup of sodium sulfite solution. Run the second point calibration, wait for stabilization, and calibrate sensor to 0%.

Once the initial calibration has been completed, conduct initial calibration verification by re-taking the DO measurement and checking that saturation (mg DO/L) against the 100% saturation number from Table 2. After field use, continuing calibration verification should be conducted (usually within 24 hours). Record all readings on the calibration record sheet.

2.2 Specific Conductance Calibration

Specific conductance is measured in micro-Seimens per centimeter (µS/cm) and is variable with temperature. For example, the conductivity of salt water increases 3% for every 1° C increase in temperature at 0° C, and only 2 % for every 1° C increase in temperature at 25°C. For this reason, most electronic water quality instruments will display a specific conductance reading which is automatically corrected for the ambient temperature.

Calibration of specific conductance measurements requires the use of purchased or laboratory-prepared standard potassium chloride (KCl) solutions with conductivity values that bracket the expected range. When the sample measurements are expected to be 100 µS/cm or greater, use two standard potassium chloride solutions that bracket the range of expected sample conductivities. When the sample measurements are expected to be less than 100 µS/cm, a lower bracket is not required, but one standard potassium chloride solution that is within the range of expected measurements will be used for the calibration and the initial calibration verification (ICV). Steps for calibration are as follows:

- Rinse the probe with de-ionized water (DI) before and between each solution.
- Fill calibration chamber with the first calibration standard.
- Calibrate the instrument with the first standard.
- Verify the calibration of the instrument with the second standard, bracketing the range of expected sample values.
- Accept the calibration if the meter reads within +/- 5% of the value of any calibration standard used to verify the calibration. For example, the acceptance range for a 100 µS/cm standard is 95 to 105 µS/cm. If the meter does not read within +/- 5% of each calibration verification standard, recalibration or maintenance is necessary.
- Check the meter with at least one KCl standard with a specific conductance in the range of conductivity measured in environmental samples. The reading for the calibration verification must also be within +/- 5% of the standard value.
- If new environmental samples are encountered outside the range of the initial calibration, verify the instrument calibration with 2 standards bracketing the range of sample values. If these calibration verifications fail, recalibrate the instrument.

After field use, continuing calibration verification should be conducted. Record all readings on the calibration record sheet.

2.3 pH Calibration

Hydrogen ion activity is often referred to by the units it is measured in: pH standard units. Most electronic water quality instruments have the capability to measure pH. Usually, three standards are used when calibrating pH readings. Laboratory-prepared standard buffer solutions with nominal values of 4.0, 7.0 and 10.0 units are used for most situations because this will bracket the majority of all naturally occurring environmental pH values. Procedures for calibrating a meter for pH are outlined below:

- Rinse the probe with de-ionized water (DI) before and between each standard buffer solution.
- Perform an initial calibration. Most meters are able to conduct a three point calibration, but always use a pH 7 buffer first.
- Perform an initial calibration verification (ICV). Read a buffer as a sample. To be acceptable, calibration verification must be within +/- 0.2 pH units of the stated buffer value. For example, if reading the pH 4.0 buffer, the result must be in the 3.8 to 4.2 range.

After sample measurement(s), perform a continuing calibration verification (CCV) to see if the instrument is still calibrated. Record all readings on the calibration record sheet.

2.4 Temperature Calibration

Several parameters measured with an electronic water quality meter are temperature dependent, and maintaining an accurate temperature reading within the instrument is critical. Temperature can be calibrated by comparing the meter's reading to that of another accurate thermometer. The standard instrument is an NIST-traceable Celsius certified thermometer with scale marks for every 0.1°C increment, a range of 0°C to 100°C (or a range bracketing expected sample temperatures) and correction chart supplied with the certification.

To calibrate, verify that the electronic meter's temperature reading falls within an acceptable range of the NIST-traceable standard thermometer at several different temperatures. Be sure to use any correction factor indicated by the certificate supplied with the NIST-traceable thermometer. Initial calibration, initial calibration verification, and continuing calibration

verification should be conducted in a manner consistent with the procedures for calibrating other parameters. Record all readings on the calibration record sheet.

2.5 Oxidation-Reduction Potential Calibration

Oxidation Reduction Potential (ORP) is a measurement of the potential for a reaction to occur. These reactions mediate the behavior of many chemical constituents in most aquatic environments. A positive value indicates that the water is in an oxidized state. A negative value is indicative of a reducing condition.

Calibration for ORP is similar to other parameters. Rinse the probe with de-ionized water (DI) before and between each standard buffer solution. Use one laboratory-prepared standard solution for ORP that will be similar to expected sample values. An initial calibration is required prior to field use as is initial calibration verification. If the values fall outside of the acceptable range, recalibration or maintenance will be performed. See Table 1 for acceptable verification ranges. A CCV should be conducted after field use, and all readings will be recorded on the calibration log.

2.6 Turbidity Calibration

Turbidity is measured in nephelometric turbidity units (NTU) and can be measured by many electronic water quality instruments. The calibration procedures for turbidity are similar to those of other parameters. Rinse the probe with de-ionized water (DI) before and between each standard buffer solution. Laboratory-prepared standards, such as StablCal® Stabilized Formazin turbidity standard of 20 NTU, will be used for all calibrations greater than zero NTU. The baseline for zero NTU can be measured using laboratory-grade de-ionized water. Use a two point method for calibrating the electronic water quality meter.

Again, an initial calibration is required prior to field use as is initial calibration verification. If the values fall outside of the acceptable range, recalibration or maintenance will be performed. See Table 1 for acceptable verification ranges. A CCV should be conducted after field use, and all readings will be recorded on the calibration log.

2.7 General Supplies

The following is a list of the minimum supplies needed for calibrating water quality instruments:

- Laboratory grade deionized water (or distilled water)
- Spray bottle
- Calibration Solutions (specific brands included for reference)
  - Oakton pH buffer (4, 7, 10)
  - Oakton specific conductivity solution (84 and 447 uS/cm)
  - StablCal® Stabilized Formazin turbidity standard of 20 NTU (deionized water for 0 NTU)
  - NIST traceable thermometer
  - In-Situ sodium sulfite solution (0% oxygen)
  - ORP (240mV)
- Field notebook or field sheets
- Tools for minor equipment repairs

3.0 Maintenance of Water Quality Meters

Routine maintenance should be performed as directed by the operation manual for any electronic water quality meter used in the field. In general, this will involve cleaning and replacing of some parts. If a membrane is used for DO measurements, it should be changed monthly, or whenever calibration procedures fail. Detailed instructions for replacing the DO membrane are available in the specific instruments operation manual. All cables and connections should be checked regularly for any wear, and replaced as needed. Batteries will also be replaced as needed. Any maintenance activities should be recorded in a maintenance log or field book.

4.0 Documentation

Detailed documentation of all calibration and maintenance activities will be kept on the appropriate log (**Figure 1**). Additional documentation of the calibration standards used will be kept and should include:

- The date of receipt, the expiration date and the date of first use for all standards and reagents.
- The concentration or other value for the standard in the appropriate measurement units.
- The vendor catalog number and description for pre-formulated solutions.

When filling out the calibration and maintenance logs, all fields will be completed, and the crew members responsible for any such activities should be noted. Any additional information pertaining to the use or maintenance of water quality instruments should be recorded in a bound field notebook.

5.0 Revised Dates*

The following are other revision dates applicable to this SOP.

Revision 1 – January 18, 2006

6.0 References

U.S. Environmental Protection Agency (USEPA). 1996. *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual*, U.S. Environmental Protection agency; Region 4, Athens, Georgia.

Table 2. Theoretical values for 100% oxygen solubility at various temperatures.

TEMP deg C	D.O. SAT.	mg/L 20%	TEMP deg C	D.O. SAT.	mg/L 20%	TEMP deg C	D.O. SAT.	mg/L 20%	TEMP deg C	D.O. SAT.	mg/L 20%
15.0	10.084	2.017	19.0	9.276	1.855	23.0	8.578	1.716	27.0	7.968	1.594
15.1	10.062	2.012	19.1	9.258	1.852	23.1	8.562	1.712	27.1	7.954	1.591
15.2	10.040	2.008	19.2	9.239	1.848	23.2	8.546	1.709	27.2	7.940	1.588
15.3	10.019	2.004	19.3	9.220	1.844	23.3	8.530	1.706	27.3	7.926	1.585
15.4	9.997	1.999	19.4	9.202	1.840	23.4	8.514	1.703	27.4	7.912	1.582
15.5	9.976	1.995	19.5	9.184	1.837	23.5	8.498	1.700	27.5	7.898	1.580
15.6	9.955	1.991	19.6	9.165	1.833	23.6	8.482	1.696	27.6	7.884	1.577
15.7	9.934	1.987	19.7	9.147	1.829	23.7	8.466	1.693	27.7	7.870	1.574
15.8	9.912	1.982	19.8	9.129	1.826	23.8	8.450	1.690	27.8	7.856	1.571
15.9	9.891	1.978	19.9	9.111	1.822	23.9	8.434	1.687	27.9	7.842	1.568
16.0	9.870	1.974	20.0	9.092	1.818	24.0	8.418	1.684	28.0	7.828	1.566
16.1	9.849	1.970	20.1	9.074	1.815	24.1	8.403	1.681	28.1	7.814	1.563
16.2	9.829	1.966	20.2	9.056	1.811	24.2	8.387	1.677	28.2	7.800	1.560
16.3	9.808	1.962	20.3	9.039	1.808	24.3	8.371	1.674	28.3	7.786	1.557
16.4	9.787	1.957	20.4	9.021	1.804	24.4	8.356	1.671	28.4	7.773	1.555
16.5	9.767	1.953	20.5	9.003	1.801	24.5	8.340	1.668	28.5	7.759	1.552
16.6	9.746	1.949	20.6	8.985	1.797	24.6	8.325	1.665	28.6	7.745	1.549
16.7	9.726	1.945	20.7	8.968	1.794	24.7	8.309	1.662	28.7	7.732	1.546
16.8	9.705	1.941	20.8	8.950	1.790	24.8	8.294	1.659	28.8	7.718	1.544
16.9	9.685	1.937	20.9	8.932	1.786	24.9	8.279	1.656	28.9	7.705	1.541
17.0	9.665	1.933	21.0	8.915	1.783	25.0	8.263	1.653	29.0	7.691	1.538
17.1	9.645	1.929	21.1	8.898	1.780	25.1	8.248	1.650	29.1	7.678	1.536
17.2	9.625	1.925	21.2	8.880	1.776	25.2	8.233	1.647	29.2	7.664	1.533
17.3	9.605	1.921	21.3	8.863	1.773	25.3	8.218	1.644	29.3	7.651	1.530
17.4	9.585	1.917	21.4	8.846	1.769	25.4	8.203	1.641	29.4	7.638	1.528
17.5	9.565	1.913	21.5	8.829	1.766	25.5	8.188	1.638	29.5	7.625	1.525
17.6	9.545	1.909	21.6	8.812	1.762	25.6	8.173	1.635	29.6	7.611	1.522
17.7	9.526	1.905	21.7	8.794	1.759	25.7	8.158	1.632	29.7	7.598	1.520
17.8	9.506	1.901	21.8	8.777	1.755	25.8	8.143	1.629	29.8	7.585	1.517
17.9	9.486	1.897	21.9	8.761	1.752	25.9	8.128	1.626	29.9	7.572	1.514
18.0	9.467	1.893	22.0	8.744	1.749	26.0	8.114	1.623	30.0	7.559	1.512
18.1	9.448	1.890	22.1	8.727	1.745	26.1	8.099	1.620	30.1	7.546	1.509
18.2	9.428	1.886	22.2	8.710	1.742	26.2	8.084	1.617	30.2	7.533	1.507
18.3	9.409	1.882	22.3	8.693	1.739	26.3	8.070	1.614	30.3	7.520	1.504
18.4	9.390	1.878	22.4	8.677	1.735	26.4	8.055	1.611	30.4	7.507	1.501
18.5	9.371	1.874	22.5	8.660	1.732	26.5	8.040	1.608	30.5	7.494	1.499
18.6	9.352	1.870	22.6	8.644	1.729	26.6	8.026	1.605	30.6	7.481	1.496
18.7	9.333	1.867	22.7	8.627	1.725	26.7	8.012	1.602	30.7	7.468	1.494
18.8	9.314	1.863	22.8	8.611	1.722	26.8	7.997	1.599	30.8	7.456	1.491
18.9	9.295	1.859	22.9	8.595	1.719	26.9	7.983	1.597	30.9	7.443	1.489

Derived using the formula in Standard Methods for the Examination of Water and Wastewater, Page 4-101, 18th Edition, 1992

Figure 1 – Field Instrument Calibration Log

CDM Form 1: FIELD INSTRUMENT CALIBRATION RECORDS

INSTRUMENT (MAKE/MODEL#) _____

INSTRUMENT # _____

PARAMETER: *[check only one]*

☐ TEMPERATURE ☐ CONDUCTIVITY ☐ pH ☐ DO
☐ OTHER _____

STANDARDS: *[Specify the type(s) of standards used for calibration, the origin of the standards, the standard values, and the date the standards were prepared or purchased]*

Standard A _____

Standard B _____

Standard C _____

DATE (yy/mm/dd)	TIME (hr:min)	STD (A, B, C)	STD VALUE	INSTRUMENT RESPONSE	% DEV	CALIBRATED (YES, NO)	TYPE (INIT, CONT)	SAMPLER INITIALS

Prepared: Brian Bennett Review: Andrew Santini

Approved: Roger L. Olsen Approved Date: 2/06/07

1.0 Overview and Application

This standard operating procedure (SOP) describes procedures for shipping and generation of chain of custody (COC) documents for samples collected during the course of this project. Procedures for packaging and storage of samples must be maintained to in order to ensure that all samples are viable and received intact by the laboratory contracted to do the analyses. This SOP will be used during field work for the Illinois River Watershed Project.

The Illinois River watershed in eastern Oklahoma and western Arkansas has been receiving inputs from agricultural runoff, including inputs from fields where poultry waste has been applied. These wastes, along with the chemical constituents that make up the poultry wastes, have apparently impacted the water quality and sediments in the streams and lakes in the Illinois River watershed. The purposes of the work being performed are to evaluate and document 1) the linkage and relationship, if any, between the disposal of poultry wastes and environmental contamination within the Illinois River watershed, and; 2) the resulting harm/injury to natural resources that may have resulted from the disposal of poultry wastes within the Illinois River watershed.

2.0 Summary of Shipping and COC Procedures

Sampling for biological and chemical properties of air, surface water, groundwater, sediment, soil, and poultry waste (litter) will be conducted periodically at various locations throughout the Illinois River watershed and in several streams in surrounding watersheds. Due to the wide range of parameters being analyzed, samples will be shipped to multiple laboratories. **Table 1** describes the typical parameters, containers, and laboratories used.

For certain parameters, samples must be preserved immediately after collection. Most laboratories provide bottles containing the necessary types and amounts of preservatives (sulfuric acid, nitric acid, hydrochloric acid, etc.). In some cases, it may be necessary to add specific preservatives after sample collection (e.g. formalin in zooplankton samples). Procedures for collection of all samples are outlined in other SOPs for this project.

2.1 Labeling Samples

All samples collected in the field will be properly labeled at the time of collection so that each sample can be uniquely identified. Labels will either be pre-printed on a non-inkjet printer/copier or clearly written in waterproof ink (i.e. permanent marker). Labels will be adhered to the sample container and multiple layers of transparent packaging tape be wrapped around the container. It is best to wrap the tape completely around the container so that the label is compressed to the sample container. This will help keep moisture away from the label, causing it to become illegible. At a minimum, the following information will be included on all labels:

- Sample identification (procedures for developing sample identification are outlined in other SOPs).
- Sample matrix (surface water, sediment, soil, etc.).
- Parameters to be analyzed for each sample.
- Date sample was collected.
- Time of day sample was collected.
- Name or initials of crew member(s) responsible for sampling.
- Sample Location (often included in the sample identification).
- Name and address of analytical laboratory.
- Name of company/organization responsible for sampling (e.g., CDM).

All of the above information should also be noted on a separate sheet or in a bound field book.

2.2 Storing Samples

Often samples cannot be shipped to a laboratory immediately after collection and will be stored until shipping. In most cases, samples will be kept at a near-freezing temperature of approximately 4° Celsius to maintain the integrity of the sample. This can be accomplished by putting the sample in a cooler with plenty of ice immediately after collection. All glass containers

should be wrapped in bubble wrap and securely packed to reduce the chance of breakage during storage.

Some sample types, if properly preserved, do not require refrigeration. Therefore, storing them on ice is not necessary. Examples of such sample types include: periphyton, zooplankton, and macroinvertebrate samples. These samples should be placed in an empty cooler or strong-sided container to avoid damage or spills. Unless specifically noted, these samples can be stored on ice, even if it is not required.

The maximum amount of holding time before a sample must be shipped to a laboratory for analysis varies depending on the parameters being tested. For example, samples for bacteria analyses will be shipped overnight on the day they are collected. Most other types of samples can be stored for longer times if kept cold, and will be shipped as soon as possible.

2.3 Chain of Custody Documents (COCs)

Whenever a sample is collected, a chain of custody document must be completed. These documents are usually forms distributed by the analytical laboratory, but generic versions are available. Typically, several samples will be included on one COC, provided that they are being sent to the same laboratory for analysis. The information contained in this document is vital for ensuring that the correct sample is analyzed and recorded. A COC also provides important documentation concerning the custody of the sample from collection to analysis.

COC forms contain much of the same information that a sample label will normally contain: the sample ID, collection date and time, location, individuals/groups responsible for sampling, parameters to be analyzed for each sample, etc. In addition, the number of samples included in the shipment, contact information, project or site identification, sample method, and other specific information required by the lab will be included. Most COCs limit the number of sample entries to approximately ten per page, as a result, multiple pages will be used so that every sample is accounted for in the document. COCs will be numbered starting with page 1 and ending with the total number of pages needed. It will not be necessary to include a COC in each cooler when multiple coolers are being shipped to the same laboratory. In this case, one cooler will contain the original COC document. Including a copy of the COCs in each cooler is helpful to the laboratory, but not necessary.

After completing the COC, a member of the sampling team (usually the team leader) will confirm the contents of the COC and will sign and date the form on the appropriate line to denote who is relinquishing the sample(s) and the date/time it was relinquished. A copy of the COC should be

made and filed for internal record keeping. The original COC should be kept in a water-tight plastic bag and placed on the top of a fully packed cooler. If possible, the COC should be taped to the inside of the cooler lid to keep it readily accessible and above any ice in the cooler.

2.4 Packing and Shipping Samples

During most sampling events, samples will be sent to several different labs for analysis. Therefore, it is important to sort samples based on the laboratory before packing them in the appropriate containers. After sorting, the COCs for each laboratory should be completed and should contain information on each sample being submitted.

2.4.1 Containers

All samples should be packed and shipped in a sturdy, water-tight container. Since most samples will need to be kept cold, a plastic cooler is usually the best container to use.

2.4.2 Packing Samples

Samples must be carefully packed into the coolers before shipping so that the bottles will not be damaged. This is especially true when glass containers are used. When shipping any glass containers, all sample bottles will be wrapped in bubble wrap and tightly packed in the coolers. Several layers of bubble wrap should be placed on the bottom of the cooler prior to packing, and any free space between sample bottles should be filled in with bubble wrap. Be sure to leave plenty of space for an adequate amount of ice and attach the COC to the inside lid of the cooler.

2.4.3 Keeping Samples Cold

To keep samples cold during overnight shipping, it is important to pack as much ice as possible into every cooler. In a typical 50 quart capacity cooler, at least one 7-pound bag of ice should be packed on top of the samples. Ice will be double-bagged in sturdy plastic garbage bags or one-gallon Ziploc bags so that water will not leak out once the ice begins to melt.

During especially hot months, it is best to pack the coolers and deliver them to the shipping company as close as possible to that carrier's latest overnight drop off time. This will help to ensure that the coolers are not exposed to unnecessary heat, which can reduce the amount of time the samples will remain at sufficiently cold temperatures. A temperature blank sample should be

placed in the coolers so that the laboratory can measure the ambient temperature in the cooler once the samples have been received.

2.4.4 Sealing Coolers

Once the coolers have been tightly packed with samples and ice, they will be properly sealed for shipping. Most shipping companies have specific requirements for sealing any container that could potentially leak and these policies should be adhered to. In general, coolers should be closed tightly and at least two bands of strong packing tape should be wrapped around the cooler vertically to secure the lid. Additionally, at least two layers of tape should be applied to all seams between the cooler and the lid to prevent leakage. If the cooler has a drain, that drain will be tightly closed and taped to prevent it from opening.

2.4.5 Custody Seals

A custody seal will be applied between the lid and the main body of the cooler. This is a strip of paper that is signed and dated before being adhered to the cooler. Some custody seals have adhesive backs, but should be taped on to ensure that they will not fall off during shipping. Applying a custody seal can be used to prove that the cooler was not opened and that samples were not tampered with during the shipping process. Upon receipt of the cooler, the laboratory can check to see if the seal is still in place and note it on the COC.

2.4.6 Shipping Coolers

After the coolers have been properly sealed, the appropriate shipping label can be applied. The shipping address and contact information for the current analytical laboratories used for this project are listed in the following section. A return address with the company name (CDM) should be included in the shipping label. The coolers can then be transported to the shipping company and sent via overnight delivery to the labs. A receipt should be retained and the labs should be contacted to inform them of the number of coolers and samples they will be receiving the following day.

2.6 General Supplies

The following is a list of the minimum supplies needed for packaging and shipping of samples:

- Plastic coolers (typical large cooler - 70 quart or 32-inches by 16-inches by 18-inches)
- Heavy-duty plastic 30 gallon size garbage bags
- Re-sealable 1 gallon size plastic bags
- Clear 2-inch wide packing tape
- Bubble wrap, half-inch bubble preferred
- Ice (minimum one 7-lb bag per cooler)
- Chain of custody forms (laboratory specific or general)
- Custody seals
- Shipping labels with return addresses

3.0 Laboratory Shipping and Contact Information

The following is a current list of contact information and shipping addresses for analytical laboratories utilized for this project. Refer to **Table 1** for a list of samples that will typically be sent to each laboratory.

General Engineering Laboratories, LLC
701 Pine Ridge Rd., Unit 5
Golden, CO 80403
Contact: Paul Winkler, (720) 253-3093
E-mail: Paul.winkler@gel.com

Aquatic Research, Inc.
3927 Aurora Avenue North
Seattle, WA 98103
Contact: Steve Lazoff, (206) 632-2715

A&L Analytical Laboratories, Inc.
2790 Whitten Rd.
Memphis, TN 38133
Contact: Jimmy Ferguson or Scott McKee, (800) 264-4522

E-mail: smckee@allabs.com

Environmental Microbiology Laboratory
1150 Bayhill Drive, Suite 100
San Bruno, CA 94066
Contact: Megan S. Tatreau, (858) 268-2770
E-mail: mtatreau@emlab.com

Idaho State University - Department of Biological Sciences
650 Memorial Drive
Pocatello, ID 83209-8007
Contact: Erin O'Leary-Jepsen, (208) 282-4890

Aquatec Biological Sciences
273 Commerce St.
Williston, VT 05495
Contact: Jennifer Gallant or Phil Downey, (802) 860-1638
E-mail: jgallant@aquatecb.com

Great Lakes Environmental Center
739 Hastings Street
Traverse City, MI 49686
Contact: Mailee W. Garton, (231) 941-2230
E-mail: mgarton@glec-tc.com

Alpha Woods Hole Analytical Labs
Eight Walkup Drive
Westborough, MA 01581-1019
(800) 624-9220
www.alphalab.com
Contact: Denise McLaughlin
508-439-5124

Water's Edge Scientific, LLC
S2756A County Road T
Baraboo, WI 53913
Contact: Debbie Nieuwenhuis, (608) 355-7930
E-mail: debnie@watersci.com

Jeff Janik, Ph.D.
2928 Boathouse Avenue
Davis, CA 95616
530-304-2286
E-mail: jjanik@omsoft.com

Reservoirs Environmental, Inc.
2059 Bryant St.
Denver, CO 80211
Contact: Jeanne Spencer Orr, (303) 964-1986

CDM- Denver Laboratory
2714 Walnut Street
Denver, CO 80205
Contact: Todd Burgesser, (303) 298-1311
E-mail: BurgesserTE@cdm.com

CDM- Lansing Office
215 South Washington Square, Suite 160
Lansing, MI 48933
Contact: Renee Mulcrone, (517) 702-1213
E-mail: MulcroneRS@cdm.com

4.0 Documentation

Bound field books should be used for the documenting field activities, as well as, aspects of sample handling. COC forms will be completed and the originals sent with the samples to the laboratories. A copy of the COC will be retained by the field crew for internal record keeping.

5.0 Additional Information

Other contact information:

Bert Fisher, PhD
Lithochimeia, Inc.
222 South Kenosha Ave.
Tulsa, OK 74120

Telephone: 918-382-9784

Ronald French
CDM- Saint Louis
100 North Tucker Blvd.
Suite 550
Saint Louis, MO 63101
314-241-8510

6.0 Revised Dates*

The following are other revision dates applicable to this SOP.

Revision 1 – April 12, 2006

Table 1. Summary of typical sample parameters, bottles, and analytical laboratories used during biological and surface water sampling of lakes and streams.

Parameters	Bottles		Analytical Labs
Total TAL Metals + Phosphorus + Molybdenum (6010/6020)	500 mL	Plastic (HNO_3)	A&L Analytical Laboratories, Inc.
Dissovled TAL Metals+ Phosphorus + Molybdenum (6010/6020)	500 mL	plastic (HNO_3)	A&L Analytical Laboratories, Inc.
TKN, ammonium	500 mL	Plastic, (H_2SO_4)	A&L Analytical Laboratories, Inc.
Total Suspended Solids (TSS), Total Dissolved Solids (TDS), Chemical Oxygen Demand (COD), Nitrate+Nitrite, pH, Spec. Conductivity	1 liter	plastic	A&L Analytical Laboratories, Inc.
Anions (Sulfate, Chloride, Alkalinity)	500 mL	Plastic	A&L Analytical Laboratories, Inc.
Total Organic Carbon (TOC)	2 x 40 mL	VOA vials (HCl)	A&L Analytical Laboratories, Inc.
Total P/Total Dissolved P/Soluble Reactive P (4500PF)	125 mL	plastic	Aquatic Research, Inc.
Chlorophyll a	250 mL	brown plastic ($MgCO_3$)	Aquatec Biological Sciences Great Lakes Environmental Center
Zooplankton	125 mL	Plastic (formalin)	Aquatec Biological Sciences
Phytoplankton	125 mL	Plastic (lugols)	Aquatec Biological Sciences Jeff Janik, PhD
Trihalomethane Formation Potential (TFP)	1 liter	Amber glass	A&L Analytical Laboratories, Inc Alpha Analytical Labs
Trihalomethanes (THM)	2 x 40ml	VOA vials (Ascorbic Acid)	Alpha Analytical Labs
Haloacetic Acids (HAA)	4 x 40ml	VOA vials (NH_4Cl)	Alpha Analytical Labs
Estrogen Metabolites	1 liter	Amber glass, (H_2SO_4)	General Engineering Laboratories
Bacteria (7 types)	500 mL	sterile plastic	Environmental Microbiology Laboratory
Polymerase Chain Reaction (PCR)	1 liter	sterile plastic	Idaho State University
Periphyton	250 mL	brown plastic	Water's Edge Scientific, LLC
Benthic Macroinvertebrates Identification	125 ml	Plastic (isopropyl alcohol)	CDM- Lansing
Hester-Dendy Samples	1 quart	plastic (isopropyl alcohol)	CDM- Lansing
High Flow Samples (Isco bottles)	1 liter	plastic	CDM- Denver

Prepared: J.B. Fisher, D. Brown Review: Jeremy Berblinger

Approved: Roger L Olsen Date Approved: 2/06/07

1.0 Overview and Application

This standard operating procedure (SOP) is applicable to the collection of representative aqueous samples of runoff from fields ("Edge of Field Samples" or "EOF Samples") and includes samples collected directly from naturally ponded or flowing water and from passive samplers installed to collect water running off fields.

2.0 Selection of Edge of Field Sampling Locations

Sample locations will be selected at public right-of-way locations adjacent to contract growers' farms or company-owned facilities where aqueous runoff from the facilities is occurring during or immediately after precipitation events. Fields where documentation of litter application from a specific farm and/or Integrator is available from the Oklahoma Department of Agriculture, Food and Forestry will be considered for sample collection. Additionally, fields observed to have had application during the year will be targeted for sample collection.

Representative samples are required. In order to collect a representative sample, the sampler should, from a consideration of local topography or direct observation of water flow, determine that the water to be collected is field runoff. Samples should be collected within 1 to 12 hours of the beginning of runoff. It is permissible to sample during periods of active rainfall.

3.0 Sampling Documentation

3.1 Sampling Log Book and Sampling Forms

1. A Sampling Log Book and Sampling Forms will be maintained.
2. Pages in the Sampling Log Book will reference specific Sampling Forms by use of the Sample ID, if appropriate.
3. The Sampling Log Book shall be bound and shall be constructed of waterproof paper.

4. Entries in the Sampling Log Book or on the Sampling Form will be made in black permanent ink.
5. Each page of the Sampling Log Book will be dated.
6. The preparer will initial each page of the Sampling Log book.
7. For each location sampled, the following information (if available) will be recorded in the Sampling Log Book or on the Sampling Forms:
    a. GPS coordinates and/or physical description of the location,
    b. Identification of the Sample Station,
    c. Identification of the property owner (if known),
    d. Identification of the grower and/or Integrator (if known) responsible for the litter applied to the field,
    e. The amounts, rates and dates of prior litter/manure applications to specific field associated with the runoff sample (confirm from State Reports if possible),
    f. Notes on weather (temperature, wind, last precipitation event, etc),
    g. Type of vegetation currently on the field, if any, and any known vegetation grown in past 5 years,
    h. Use of adjacent fields, and;
    i. Other information as appropriate or relevant.

3.2 Photographic Record

A photographic record will be made and maintained for all sampling activities at the EOF location, lighting and weather permitting.

All photographs made will be time and date stamped.

3.3 Chain-of-Custody

A Chain-of-Custody will be prepared for each set of samples for the analytical laboratory.

The Chain-of-Custody to the lab will, at a minimum, contain the following information:

1. The project name, ***Illinois River Watershed or Oklahoma Poultry Project,***
2. Name of person or entity collecting samples,

3. Signature blocks with dates and times for all persons having custody (sampler, shipper, processing laboratory, etc),
4. For each sample related to a Chain-of-Custody:
    a. The unique numeric identifier on the submitted sample container/bag,
    b. The date and time the sample was collected,
    c. The sample "matrix" (i.e. Water).

4.0 Sampling

Sampling situations vary widely and therefore no universal sampling procedure can be recommended. However, sampling of liquids from the above mentioned location is generally accomplished through the use of one of the following samplers or techniques:

- Dip Sampling
- Passive Runoff Collector

These sampling techniques will allow for the collection of representative samples from the majority of circumstances in which runoff water from fields can be accessed.

There are two primary interferences or potential problems associated with the collection of Edge of Field Samples. These include cross-contamination of samples and improper sample collection.

Cross-contamination problems can be eliminated or minimized through the use of dedicated sampling equipment. If this is not possible or practical, then decontamination of sampling equipment is necessary. Improper sample collection can involve using contaminated equipment, excessive disturbance of the sample site, and sampling in an obviously unrepresentative location. Following proper decontamination procedures and minimizing disturbance of the sample site will eliminate these problems.

4.1 Equipment and Supplies

Equipment needed for collection of surface water samples includes:

- Passive Runoff Collector (if installed)
- Dip Sampler (if no Passive Runoff Collector is installed)
- Funnel
- Sample bottles prepared as needed for the analyses to be performed
- Ice

- Cooler(s)
- Chain of custody forms
- Field notebook or field data sheets
- Appropriate writing implements for recording data and labeling sample bottles
- Camera and film (or Digital Camera)
- Decontamination equipment and reagents
- Safety equipment
- Global Positioning System (GPS)
- Maps (paper or electronic)

4.2 Sample Collection - Preparation

Determine the extent of the sampling effort, the sampling methods to be employed, and which equipment and supplies are needed.

Obtain the necessary sampling and monitoring equipment.

Decontaminate or pre-clean the equipment, and ensure that it is in working order.

Prepare scheduling and coordinate with staff, clients, and regulatory agency, if appropriate.

Load the GPS unit with relevant electronic maps and sampling locations (if known).

4.3 Sample Collection

Passive Runoff Collector

A passive runoff collector may be used at an edge of field site if such a sampler can be installed at the site and if sufficient runoff can be recovered from the collector following a runoff event. To collect a sample from a passive runoff sampler, remove the sampler from its installation hole and pour collected runoff water directly into a large container for later processing and transportation.

Dip Sampler

A dip sampler is useful for situations where a passive runoff sampler was not installed or in situations where a passive runoff sampler would not be able to collect a sufficient volume of water. A dip sampler can be any device that can be dipped into an accumulation of runoff water to recover runoff water without unduly disturbing that accumulation of water. Water collected by dip sampling is transferred directly into a large container for later processing and transportation.

4.4 Handling of Samples

Samples for bacteria will be placed into a sterile bottle and shipped directly to the laboratory. The remaining sample will be processed (filtered and preserved) as appropriate at the CDM laboratory in Denver or the staging facility in Tulsa.

4.5 Field QA/QC Samples (Edge of Field)

1. Enough sample for Duplicate Samples will be collected in the field. The duplicate will be either a co-located or split sample.
2. Decontamination Blank: a sample of the final decontamination rinsate may be collected and sent to the laboratory for analysis.

4.6 Decontamination Procedures

After each collection of an EOF sample, all sampling equipment that will be reused will be decontaminated. That is, decontamination will occur between every sampling event or location. To the extent possible, disposable sampling equipment should be used. All reusable sampling equipment shall be decontaminated using a non-phosphate detergent and three de-ionized water rinses between Sampling Areas.

5.0 Person(s) Collecting Samples and Observing Sampling

Personnel from Lithochimeia or CDM will conduct the EOF sampling at each opportunistic location. CDM or Lithochimeia personnel will process samples, chain-of-custody, coordinate shipping, etc.

6.0 Identification of Samples

Identifying information to be recorded on the sample label for edge of field samples:

1. Alphanumeric identification of the Sample Type: EOF.
2. Alphanumeric identification of the Station: (Way Point or Spread Site ID)
3. Numeric date designation or sequence designation (MMDDYYYY or 01, 02 etc.)
4. The following sample number is an example of the first runoff sample collected from Spread Site 30:

   EOF-Spread 30-01

5. If appropriate, samples sent to the analytical laboratory will have alphanumeric identification of the type of sample added to the end of the number:
    a. B = laboratory duplicate
    b. C = reference liquid (standard)
    c. D = decontamination blank (added in the field)
    d. E = laboratory QA/QC (extra volume)
6. Date of sample collection (only on chain-of-custody),
7. Time of sample collection (only on chain-of-custody),
8. Initials of the person collecting the sample (only on chain-of-custody).

When sending samples to the analytical lab, identifying information marked on the sampling container will be protected by covering the identifying information with clear plastic tape.

7.0 Shipment of Samples to the processing laboratory and to the analytical laboratory

Samples will be processed (filtered and preserved as appropriate) at the staging facility in Tulsa or at the CDM Denver laboratory.

1. Once placed in sampling containers (laboratory provided sample containers), samples will be placed on ice (sealed in plastic bags) within insulated protective containers.
2. If possible, samples will be shipped immediately via overnight shipment to the analytical laboratory.
3. Samples should not be held more than 24 hours before shipment.
4. Samples will be sent to the laboratory under a Chain-of-Custody.
5. A custody seal will be placed on the outside of the container across the area between the lid and the container. The custody seal will be signed.

6. The Chain-of-Custody will be sealed in a plastic bag and placed within the insulated protective container holding those samples to which it refers.

8.0 Analytical

8.1 Laboratory

The laboratory conducting the analyses shall be experienced in conducting the specified analyses and shall have certifications to conduct the specified analyses.

All analyses and sample preparation shall be conducted using accepted and published protocols and/or methods.

8.2 Analytical Protocols

Aqueous samples are typically analyzed for the long list of parameters. Table 1 provides the parameters and analytical methods for the long list.

Parameter	Method
Total dissolved solids, TDS	160.1
Total suspended Solids, TSS	160.2
Total organic carbon	415.2
pH	150.0
Specific Conductivity	120.1
Total Phosphorus (P), nonfiltered	365.2, persulfate digestion
Total P, ortho/reactive, nonfiltered	365.2, direct
dissolved P total, filtered	365.2, persulfate digestion
Dissolved P, ortho/reactive, filtered	365.2, direct
Nitrogen, nitrate + nitrite	353.3
Total Nitrogen	TKN, 351.3
Nitrogen, ammonium	350.2
TAL Metals, dissolved (filtered)	EPA SW-3050/6010/6020
Dissolved Molybdenum (Mo)	EPA SW-3050/6010/6020
Dissolved Phosphorus (P)	EPA SW-3050/6010/6020
TAL Metals, total (nonfiltered)	EPA SW-3050/6010/6020
Total Molybdenum (Mo)	EPA SW-3050/6010/6020
Total Phosphorus (P)	EPA SW-3050/6010/6020
Sulfate, filtered	375.1
Alkalinity, filtered	310.2
Chloride, filtered	325.1
Bacteria	
Total coliform	SM-9221B

enterococcus	SM-9230B
Fecal coliform	SM-9221E
e-coli	SM-9221F
staphylococcus	BAM 12
campylobacter	BAM, CHAP. 7
salmonella	BAM 5
17α-estradiol,17β-estradiol, estrone, estriol	LC-MS-MS

8.3 Data Reporting

1. Data from the laboratory shall be reported in both electronic and paper reports.
2. Data reports shall include all quality control data generated, including results for duplicates, blanks and spikes, as applicable.
3. Data reports shall include a copy of the Chain of Custody accompanying each set of samples submitted

9.0 Health and Safety Plan:

A health and safety plan that is specific to this sampling protocol will be prepared and reviewed by all samplers.

When working with potentially hazardous materials, follow EPA, OSHA, and specific health and safety procedures. When sampling runoff from fields containing known or suspected hazardous substances or infectious materials, take adequate precautions.

Prepared: Jeremy Berblinger Review: Darren Brown

Approved: Roger L. Olsen Date Approved: 2/06/07

1.0 Overview and Application

This standard operating procedure (SOP) describes field procedures used for collection of groundwater samples from the Illinois River watershed (IRW) of eastern Oklahoma. Groundwater samples will be collected from up to fifty existing wells locations within the Oklahoma portion of the IRW. Locations within various potentially impacted areas and areas suspected to have minimal impact will be selected for sampling. Procedures for on site measurement of various water quality parameters such as temperature, conductivity, turbidity, pH, oxidation-reduction potential, and dissolved oxygen will are provided.

Application of poultry house waste to fields has been identified as a probable source of contamination in springs and river base flow. These observations indicate that the source of these waters (i.e., groundwater) is also contaminated. The extent and degree of potential groundwater impact is currently not known. This study will help identify the levels of potential environmental contaminants in the groundwater.

2.0 Sampling Methods Summary

At each sample location, field crews will obtain permission of property owners to sample wells at designated locations. Wells should be allowed to run for fifteen minutes before parameters are recorded and samples collected. Property owners must first sign an access agreement form and grant permission before sampling can take place. Samples will be collected in appropriate containers and shipped directly to the laboratories listed in Section 4.2.

2.1 Sampling Locations

Existing well information in northeast Oklahoma will be reviewed and compiled into a database. The purpose of this subtask is to provide locations where groundwater samples can be collected from existing wells. The data gathered from existing wells will be posted on maps to facilitate well selection.

Existing wells will be targeted with completion depths less than 150 feet in order to focus on groundwater most likely to be impacted by land application. Existing wells within various sub-

watersheds having variable chicken densities will be selected. Existing wells within sub-watersheds having approximately the same chicken densities but at different distances from chicken houses or known application sites will also be selected.

2.2 Sample Types

Water samples will be grab samples. A grab sample is defined as a single aliquot from a specific location or depth at a given point in time. This type of sample represents a single value and can, in certain instances, be used as an alternative to analyzing a number of individual discrete samples and calculating an average value.

2.3 Data To Be Collected

At each sampling location, a variety of physical and water quality parameters will be assessed and recorded. The exact location of the sampling should be measured with a handheld GPS unit and recorded. Water quality parameters will be measured using an Oakton temperature, pH, conductivity multi-meters which will be calibrated and verified before and after each sampling event as described in Standard Operating Procedure 8-1: *Water Quality Meters*. At each sampling location the meter probe will be placed into the sample of the well water and a reading will be recorded for each of the following parameters:

- Temperature in degrees Celsius
- pH
- Specific conductance in micro-Siemens per cm

2.4 General Supplies

The following is a list of the minimum supplies needed for ground water sampling within the IRW:

- Temperature, pH, and conductivity multi-meters
- Sample bottles with labels
- Clear tape
- Coolers with ice
- De-ionized water
- Phosphate-free detergent
- Nitrile gloves
- Field Notebooks with water resistant paper
- Handheld GPS Unit, with extra batteries
- Digital camera with extra batteries
- Tools for minor equipment repairs

- Drinking water

3.0 Sampling Procedures

Procedures for collecting the water samples may vary depending on the type of sample desired. In general, the following procedures should be employed.

- Obtain permission from land owner in the form of a signed access agreement form.
- Locate well, allow water to run for 15 minutes, if permitted by landowner/resident.
- After purging well, fill clean 5- gallon bucket with 1-2 gallons of sample water and submerge temperature/pH/conductivity probe. Wait for meter to stabilize and record readings in field notebook.
- Carefully fill the appropriate sample bottles with the sample water. Samples will typically be collected using (1) 1 gallon clean (unused) jug, (1) 500 ml sterile bottle for bacteria, and (1) 150 ml bottle for phosphorous parameters.
- Store the samples in a cooler with plenty of ice until shipment to appropriate laboratories.
- Bacteria samples will be shipped the same day to Environmental Microbiology Laboratory. Contact information is listed below.
- The phosphorous samples will be shipped the same day to Aquatic Research Inc.
- The 1 gallon jugs will be sent to the CDM Denver lab where parameters to be analyzed will be determined.

Contact information is provided in Section 4.2.

4.0 Sample Containers, Preservation Techniques, Quality Control

Periodically throughout the sampling, samples will be packed and shipped in coolers to one of several different analytical laboratories (contact information below) depending on analyses required (contact information below). Field control samples will be collected by the sampling team to determine whether data are of suitable quality. Control samples may include trip blanks or duplicates. Duplicates should be collected at least once for every twenty samples by filling additional sample containers while the well is still pumping. Decon blanks were not collected as the samples were collected directly from the tap or faucet and sampling equipment was not necessary.

4.1 Decontamination Procedures

To ensure that samples are not contaminated by equipment or containers, it is necessary to follow certain procedures for cleaning or decontaminating equipment. All sampling equipment which is in direct contact with the sample water must be cleaned between each sample collection. Equipment which must be decontaminated will include, but is not limited to: plastic

5-gallon bucket, multi-meter probes, filtering apparatus, beakers or flasks, and volumetric measurement devices.

Procedures for decontamination are as follows:

- Rinse all surfaces with de-ionized or distilled water.
- Using a spray bottle, apply a layer of phosphate-free detergent to all surfaces.
- Rinse all surfaces again with de-ionized or distilled water until all detergent has been removed.
- If appropriate, rinse the sampling container with the collected water.

4.2 Laboratory Contact Information

The following is a list of contact information and shipping addresses for all analytical laboratories used for water samples from IRW Groundwater Sampling.

Environmental Microbiology Laboratory
1150 Bayhill Drive, Suite 100
San Bruno, CA 94066
Contact: Megan S. Tatreau, 858-268-2770
E-mail: mtatreau@emlab.com

CDM Denver Lab
2714 Walnut Street
Denver, CO 80205
303-308-2310
Contact: Todd Burgesser
E-mail: burgesserte@cdm.com

Aquatic Research Inc
3927 Aurora Avenue North
Seattle, WA 98103
Contact: Steve Lazoff, 206-632-2715

5.0 Identifying information to be recorded on the sample label for groundwater well samples:

1. Alphanumeric identification of the well: This can be the State Well ID or the name of the current well owner. The prefix of GW will be placed in front of

the Well ID to distinguish that the sample is a groundwater sample. Initially, no prefix was placed in front of the Well ID.

2. The following sample number is an example of the well sample collected from residential well 66783:

   GW-66783

3. Samples sent to the analytical laboratory will have alphanumeric identification of the type of sample added to the end of the number:
   a. Q = field duplicate.
   b. E = laboratory QA/QC (extra volume)
4. Date of sample collection (only on chain-of-custody),
5. Time of sample collection (only on chain-of-custody),
6. Initials of the person collecting the sample (only on chain-of-custody).

6.0 Documentation

Bound field logbooks should be used for the maintenance of field records. All aspects of sample collection and handling as well as visual observations shall be documented in the field logbooks. Supplemental information may be documented on the field data sheets provided. All entries in field logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.

7.0 Additional Information

Other contact information:

Bert Fisher, PhD
Lithochimeia, Inc.
222 South Kenosha Ave.
Tulsa, OK 74120
Telephone: 918-382-9784

8.0 Revised Dates*

The following are other revision dates applicable to this SOP.

Revision 2 – February 5, 2007
Revision 1 – January 4, 2007

Prepared: Jeremy Berblinger Review: Darren Brown

Approved: Roger L Olsen Date Approved: 2/06/07

1.0 Overview and Application

This standard operating procedure (SOP) describes field procedures used for collection of groundwater samples by direct push sampling methods from the Illinois River watershed (IRW) of eastern Oklahoma. Groundwater samples will be collected from approximately fifty locations within the Oklahoma portion of the IRW. Locations anticipated to have saturated unconsolidated sediments within various impacted areas will be selected for sampling. Procedures for on site measurement of various water quality parameters such as temperature, conductivity, pH, oxidation-reduction potential, and dissolved oxygen are provided.

Application of poultry house waste to fields has been identified as a probable source of contamination in springs and river base flow. These observations indicate that the source of these waters (i.e., groundwater) is also contaminated. The extent and degree of potential groundwater impact is currently not known. This study will help identify the levels of potential environmental contaminants in the groundwater.

2.0 Sampling Methods Summary

At each sample location, field crews will set up on staked locations that have been cleared of all utilities. Direct push technology will be used to collect groundwater samples from unconsolidated sediments. Probe rods will be advanced to bedrock or refusal. The screen will be exposed to the unconsolidated material and allow water, if present, to enter rods. Up to thirty minutes will be allotted for water to enter the probe rods. The depth to water will be checked and noted in the field book prior to sampling. The sample will be collected using ¼-inch poly-ethylene tubing fitted with a check valve. The tubing will be vertically manipulated to evacuate groundwater from the probe rods. The water will be transferred from the tubing directly into the appropriate sample containers.

2.1 Sampling Locations

Groundwater samples will be collected by direct push technology from saturated unconsolidated sediments within litter applied fields (if access is obtained) and along select public right-of-way (ROW) points throughout the basin.

- Locations will be selected primarily within areas evaluated by a geologist as having the potential to have saturated unconsolidated sediments. Most of the saturated unconsolidated sediments in this watershed are likely to be predominantly comprised of alluvial material. Within Oklahoma, these locations are typically along developed creeks and rivers. The major creeks and rivers are typically within fracture zones, and are likely to receive groundwater contribution from both bedrock and saturated alluvium.
- Primary targeted areas are the Flint Creek drainage, Illinois River drainage, Ballard Creek drainage, Baron Fork drainage, and Caney Creek drainage. Where appropriate, locations with drainages entering Oklahoma from Arkansas will be selected.
- Preliminary sites will be visually inspected to determine whether saturated unconsolidated sediments may be present. Visual inspection will include observing bedrock outcroppings and the relative position of bedrock material to surface water elevation. If surface water is present below the elevation of the bedrock outcrops, there is low likelihood of having groundwater present within the unconsolidated material.
- Sites within the subpoenaed property will be evaluated for the potential to have saturated unconsolidated material.
- Sites along state and county right-of-ways (ROWs) will be evaluated as potential sampling locations. ROW sites must have sufficient access on shoulder to permit sampling.

2.2 Sample Types

Water samples will be grab samples. A grab sample is defined as a single aliquot from a specific location or depth at a given point in time. This type of sample represents a single value and can, in certain instances, be used as an alternative to analyzing a number of individual discrete samples and calculating an average value.

2.3 General Supplies

The following is a list of the minimum supplies needed for ground water sampling within the IRW:

- Sample bottles with labels
- Direct Push rig (typically a Geoprobe™ rig)
- 1000′ roll of ¼-inch poly-ethylene tubing
- Check valve
- Clear tape
- Flagging tape, stakes, etc.
- Coolers with ice
- De-ionized water
- Phosphate-free detergent
- Nitrile gloves

- Field Notebooks with water resistant paper
- Handheld GPS Unit, with extra batteries
- Digital camera with extra batteries
- Tools for minor equipment repairs
- Drinking water

3.0 Sampling Procedures

Procedures for collecting the water samples may vary depending on the type of sample desired. In general, the following procedures will be employed.

- Feed ¼ -inch poly-ethylene tubing, with check valve connected to the end, inside the probe rods until the bottom of the rods is encountered. Rapidly move the tubing up and down (approximately 1 foot) inside the rod until water can be seen near top of tubing. With the check valve properly functioning, water will enter the tubing each time it is lowered and the check valve will keep the water from leaving the tubing as the tubing is raised.
- Carefully fill the appropriate sample bottles with the sample water. Samples will typically be collected using (1) 1 gallon clean (unused) bottle, (1) 500 ml sterile bottle for bacteria, and (1) 125 ml plastic bottle for phosphorus analyses.
- Store the samples in a cooler with plenty of ice until shipment to appropriate laboratories.
- Bacteria samples will be shipped the same day to Environmental Microbiology Laboratory. Contact information is listed below.
- Phosphorus samples will be shipped the same day to Aquatic Research Inc. Contact information is listed below.
- The 1 gallon bottle will be sent to the CDM Denver lab where parameters to be analyzed will be determined. Contact information is listed below.

4.0 Sample Containers, Preservation Techniques, Quality Control

Periodically throughout the sampling, samples will be packed and shipped in coolers to one of several different analytical laboratories depending on analyses required(contact information below). Field control samples will be collected by the sampling team to determine whether data are of suitable quality. Control samples may include rinsate blanks and duplicates. Duplicates are samples that are collected by filling additional sample bottles while using the tubing and check valve to generate flow through the tubing. If enough water is present, duplicate samples should be collected at least once for every twenty samples.

4.1 Decontamination Procedures

To ensure that samples are not contaminated by equipment or containers, it is necessary to follow certain procedures for cleaning or decontaminating equipment. All sampling equipment which is in direct contact with the sample water will be cleaned between each sample collection. Equipment to be decontaminated will include, but is not limited to: plastic 5-gallon bucket, multi-meter probes, filtering apparatus, beakers or flasks, and volumetric measurement devices. Probe rods and screens will also be decontaminated between sample locations. Downhole sample tubing will be disposed between sample locations.

Procedures for decontamination for small tools and sample equipment are as follows:

- Using a spray bottle, apply a layer of phosphate-free detergent to all surfaces.
- Rinse all surfaces again with de-ionized or distilled water until all detergent has been removed.

For larger equipment and probe rods, the following procedure will be used:

- Spray all surfaces with hot water pressure washer. (i.e., probe rods, screens and check valve)

Decontamination fluids will be discharged to the ground in the immediate vicinity of the decontamination activity. All solids, such as gloves and paper towels will be bagged and disposed in a dumpster serviced by a municipal waste disposal firm.

4.2 Laboratory Contact Information

The following is a list of contact information and shipping addresses for all analytical laboratories used for water samples from IRW Groundwater Sampling.

Aquatic Research Inc
3927 Aurora Avenue North
Seattle, WA 98103
Contact: Steve Lazoff, 206-632-2715

CDM Denver Lab
2714 Walnut Street
Denver, CO 80205
303-308-2310
Contact: Todd Burgesser
E-mail:burgesserte@cdm.com

Environmental Microbiology Laboratory
1150 Bayhill Drive, Suite 100
San Bruno, CA 94066
Contact: Megan S. Tatreau, 858-268-2770
e-mail: mtatreau@emlab.com

5.0 Documentation

Bound field logbooks should be used for the maintenance of field records. All aspects of sample collection and handling as well as visual observations will be documented in the field logbooks. Supplemental information may be documented on the field data sheets provided. All entries in field logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.

6.0 Additional Information

Other contact information:

Bert Fisher, PhD
Lithochimeia, Inc.
222 South Kenosha Ave.
Tulsa, OK 74120
Telephone: 918-382-9784

7.0 Revised Dates*

The following are other revision dates applicable to this SOP.

Revision 2 – January 22, 2007
Revision 1 – January 16, 2007

SOP: 12-1
Standard Operating Procedure
General Water Preparation and Filtering
EOF, Springs and Groundwater

Revision: 0
Initial Date: 02/05/07
Last Revised:

Prepared: Todd Burgesser Review: Tim Cox

Approved: Roger L. Olsen Date Approved: 2/06/07

1.0 Background and Scope

Instructions presented in this standard Operating Procedure (SOP) are for preparing water samples collected as edge-of-field, springs or groundwater. This SOP describes in detail the protocol for the samples as outlined for the Illinois River Watershed Sampling Program.

The Illinois River watershed lies in eastern Oklahoma and western Arkansas. Water quality in this watershed is impaired, likely due primarily to agricultural runoff. The dominant industry in the watershed is poultry, an industry where farmers routinely spread their waste on open fields. The SOP outlined here supports a sampling program aiming to: 1) directly measure and summarize flow-weighted concentrations of key pollutants of concern associated with runoff events in small watershed tributaries, and; 2.) investigate potential correlations between these concentrations and land use characteristics, poultry operations, and storm event hydrographs characteristics.

2.0 Equipment Specifications

A nucleopore or equivalent filtering apparatus (47mm) with 0.45 u nitrocellulose filter and a stand alone vacuum pump is recommended for filtering. This device will filter the water at a rate of approximately 50-mL/minute.

3.0 Sample Receipt and Login

Upon receipt of coolers from field personnel, all coolers are opened and the internal temperature of the cooler is recorded on the chain-of-custody (COC) form. The thermometer used for measuring the temperature will be previously calibrated against a NIST calibrated thermometer. The samples are then removed from the cooler and transferred to a refrigerator until compositing begins. Any discrepancies with the samples and the COC are noted and resolved at this point through conversations with the original sampler and shipper. The COC will be signed and dated with the current date and time upon receipt.

STOK0020967

4.0 Sample Filtering

The filtering apparatus is assembled by placing the fritted glass filter holder into a 250-mL or 500-mL Erlenmeyer filtering flask. A 47 mm nitrocellulose 0.45u filter is placed on the fritted section of the filter holder and a 250-mL funnel is clamped to the filter holder. A vacuum pump is attached to the Erlenmeyer flask and vacuum is applied while pouring sample into the funnel. When the Erlenmeyer flask is full, vacuum is released and the filtrate is transferred to the appropriate sample bottle. A new 0.45u filter is placed onto the filter holder and the process is repeated until all appropriate sample bottles are filled. Section 9 presents sample bottle requirements, preservatives and minimum volumes for the analysis requested. The filtered sample is then distributed to the appropriate sample bottles. Sample bottles that do not require filtering are filled with the from the original sample container received.

5.0 Equipment Decontamination

All equipment including the filtering apparatus, Erlenmeyer flask, graduated cylinders and beakers should be thoroughly cleaned before use by washing vigorously with a brush, hot water and Alconox detergent. The equipment will then be rinsed with de-ionized water to remove any remaining soap followed by a rinse with 10% bleach and a last rinse with de-ionized water. The equipment is allowed to air dry before use.

6.0 Sample Shipping to Contracted Laboratories

After sample filtration and distribution of the sample to sample containers the samples are packed into coolers with appropriate padding (bubble wrap, packing paper). Ice contained in re-sealable baggies is placed within the cooler followed by the completed chain-of-custody form. The cooler is closed and secured with packing tape followed by a signed custody seal. All coolers will be shipped to the respective laboratories vie Federal Express for overnight delivery.

7.0 Analyses, Bottle Requirements, Preservatives and Minimum Sample Volumes, Analytical Laboratory

The following table presents potential analysis, bottle requirements, preservatives, minimum sample volumes and analytical laboratory.

Analysis	Bottle	Preservative	Minimum Volume	Analytical Laboratory
Total Organic Carbon	2-40-mL glass	HCl	80 mL	A&L
Total Metals+Mo+P	500-mL poly	HNO_3	125-mL	A&L
Dissolved Metals+Mo+P (filtered)	500-mL poly	HNO_3	125-mL	A&L
TKN, Ammonia	500-mL poly	H_2SO_4	225-mL	A&L
Sulfate, Chloride, Alkalinity (filtered)	500-mL poly	None	350-mL	A&L
Nitrate+Nitrite, TSS, TDS, pH	500-mL poly	None	400-mL	A&L
Total and Dissolved P; soluble reactive P	150-mL poly	None	100-mL	Aquatic Research
Estrogens	1000-mL amber glass	H_2SO_4	1000-mL	GEL
PCR	1000-mL poly sterile	None	1000-mL	Northwind/Idaho State University
Bacteria	500-mL poly sterile	None	500-mL	EML

8.0 Documentation

Bound laboratory logbooks should be used for the maintenance of field records. All aspects of sample compositing and handling as well as visual observations will be documented in the field logbooks. Supplemental information may be documented on the field data sheets provided. All entries in laboratory logbooks should be legibly recorded and contain accurate and inclusive documentation of an individual's project activities.