

# Appendix 13

## Quality Assurance Project Plan



**QUALITY ASSURANCE PROJECT PLAN  
FOR  
Little Calumet River Watershed Monitoring  
ARN: 6-01**

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June 2007

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Planning Branch Chief	_____ Marylou Renshaw	_____ Date

Copies of the QAPP have been distributed to Greg Bright, Jill Hoffmann, Phil Gralik, and Betty Ratcliff, all of whom have responsibility for implementation of various tasks in the project.

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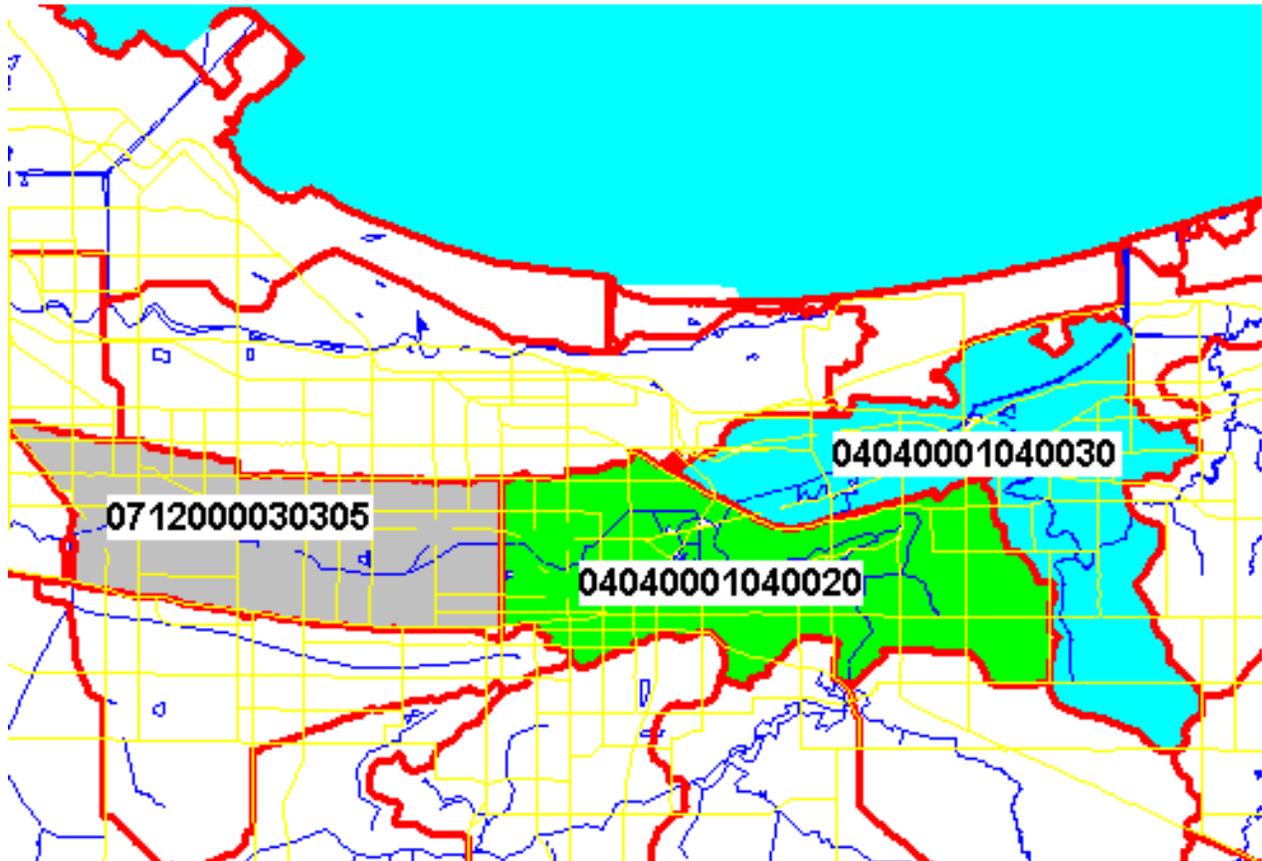
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## 1.0 INTRODUCTION

The Gary Storm Water Management District has received a 319 water quality grant from the Indiana Department of Environmental Management (IDEM) and the United States Environmental Protection Agency (USEPA). The purpose of the grant is to prepare a watershed management plan for three 14-digit subwatersheds in the Little Calumet River basin (Fig. 1). One of the tasks in the project is to monitor water quality using biological and chemical methods and use the information to make decisions that may be used to help prepare the watershed management plan. This document presents a quality assurance plan for monitoring.

Figure 1. The 3 sub-watersheds to be studied



## 2.0 PROJECT DESCRIPTION

### 2.1 General Overview:

The water quality assessment will use water chemistry at seven sites within the watershed. In addition, because the Little Calumet River is on the Indiana impaired waterbodies list because of E.coli contamination, E.coli will be monitored at 40 additional sites to attempt to track down important sources. The information will be used to diagnose water quality problems and propose solutions.

### 2.2 Project Objectives:

The objectives of this project are to characterize the biological and chemical integrity of three 14-digit subwatersheds (07120000303050, 04040001040020, and 0410400001040030) and to make recommendations to solve any identified problems.

*E. coli* are a bacteriological indicator of potential human health effects associated with whole body contact in water. Analysis of *E. coli* concentrations at various sites within the watersheds during warm weather will help determine human health risk and potentially help locate problem sources of bacteria.

### 2.3 Sampling Design:

The overall experimental design is to sample basic water chemistry and bacteria to answer the following questions:

- What is the overall ecological health of the watersheds?
- Where are the E.coli originating?
- What can be done to make the identified problems better?

<u>Parameter</u>	<u>When</u>	<u>Where</u>	<u>Why</u>
Basic water chemistry	low/hi flow 2 times	Little Cal 7 sites	Provide instream data near various urban inputs
<i>E. coli</i> in water	low/hi flow 4 times	Outfalls/ Streams 40 sites	Provide instream data near various urban inputs

Table 1 shows a summary of the types of measurements to be collected as part of this study.

Table 1. Chemical and biological parameters to be measured at each site

Chemical Measurements

Nitrogen (nitrates+nitrites), total phosphorus, total suspended solids, pH, temperature, conductivity, dissolved oxygen, stream flow. These parameters will be measured at 7 sites. Measurements will be made twice during 2007. One event will be immediately following a storm.

*E. coli* Measurements

*E. coli* will be measured at 40 sites, including storm sewer outfalls. Samples at these sites will be collected four times during 2007. One event will be immediately following a storm.

Parameter	Method	Detection Limit	Holding Time	Site
pH	SM 4500 H+	0.1 SU	N/A	Field
Temperature	Thermocouple	0.1 degree	N/A	Field
Conductivity	SM 2510 A	1 uS	N/A	Field
Dissolved oxygen	SM 4500 O G	0.1 mg/l	N/A	Field
NO2+NO3	SM 4500 NO3	0.5 mg/l	28 days	Lab
Total P	SM 4500 P F	0.03 mg/l	2 days	Lab
TSS	SM 2540 B	1 mg/l	7 days	Lab
E.coli	SM 9223 B	1 / 100ml	6 hrs	Lab
Flow	USGS guage [Deep River] [Burns Ditch]	N/A	N/A	N/A

2.4 Project Timetable:

The project will be conducted during 2007 with a final report to be available for inclusion in the watershed management plan by February 1, 2008.

QAPP approved	June 2007
Chemical Sampling	June and September 2007
E.coli Sampling	June, July, August, and September 2007
Data Analysis	September 2007
Final Report	January 2008

### 3.0 PROJECT ORGANIZATION AND RESPONSIBILITY

The Project Manager (Greg R. Bright, Commonwealth Biomonitoring) is responsible for biological quality assurance, management of the project field logistics, the collection, analysis, and interpretation of biological data, and writing the biological report. A copy of the lab's Standard Operating Procedures is attached in the Appendix. Greg Bright will also be responsible for chemistry quality assurance and laboratory chemical analysis. A copy of the lab's Standard Operating Procedures for the required chemical analysis is attached in the Appendix.

Dr. Melody Myers-Kinzie (Commonwealth Biomonitoring) is responsible for chemistry analysis.

The Watershed Communicator (Jill Hoffmann, Empower Results) is responsible for using the data to help local stakeholders make decisions about prioritizing identified problems and solutions.

The Watershed Coordinator (Phil Gralik, R.W. Armstrong) is responsible for coordinating the project with Commonwealth Biomonitoring, IDEM, and the Gary Storm Water District.

The IDEM quality assurance coordinator (Betty Ratcliff) is responsible for oversight of the quality assurance portion of the grant.

The IDEM grant project manager (Skye Schelle) is responsible for oversight of the grant schedule, including water quality monitoring and reporting.

### 4.0 DATA QUALITY OBJECTIVES

#### 4.1 Accuracy/Bias

Accuracy and bias in bacteriological and chemical analyses are dependent on maintenance of standard procedures for sample processing, labeling, and chemistry laboratory procedures.

For the laboratory chemical measurements, we expect accuracies within 10% of the true value, based on previous results obtained by laboratories participating in performance evaluations.

Bias is evaluated by the use of field and laboratory blanks. One field blank will be used for each sampling event.

#### 4.2 Precision

Precision of the laboratory chemical analyses is expected to result in chemical recoveries of 90 to 110%. Precision will be measured by analyzing the results of duplicate samples collected in the field and measuring the relative percent difference. There will be one duplicate collected per sampling event for chemical analysis. For each 40 *E. coli* sample events, there will be 3 duplicate samples.

#### 4.3 Completeness

The “completeness” objective for biological and chemical measurements in this project is 90%. Since there are 14 samples planned for chemical analysis, the objective is to obtain 13 valid chemical samples. For *E. coli*, the number of samples to be collected is 160. Therefore the “completeness” objective for *E. coli* is 144 valid samples.

#### 4.4 Representativeness

The samples collected for chemical and biological analysis should be representative of the ecological health of the site where the sample is collected. To assure representativeness, all samples will be collected on the same day, using the same collection technique. The sites that have been selected for analysis represent the entire watershed.

#### 4.5 Comparability

Comparability is ensured through the use of identical sampling and analysis techniques at each sample site. This also assures that the results may be compared to historical samples of water quality collected in the watersheds by IDEM that use similar techniques.

### 5.0 FIELD PROCEDURES

Chemical sampling will consist of grab samples collected from pooled areas. High density plastic containers will be used to collect all chemical samples. Samples for nitrogen and phosphorus analysis will be preserved with sulfuric acid. All samples will be placed on ice for transport to the lab.

#### Chemistry

Field chemistry measurements will be made using appropriate field meters.

### E.Coli

Sampling and analysis will be carried out by Commonwealth Biomonitoring. Grab samples will be collected in pre-sterilized jars. The standard operating procedure for *E. coli* analysis in water is found in Appendix 3.

### Sample conditions

For chemical sampling at the seven “base” sites, one set of samples will be collected during dry weather (no significant rain within the prior 7 days) during late summer. One sample will be collected during wet weather (at least 0.3 inches of rain within the previous 24 hours) during early spring.

## 6.0 LABORATORY PROCEDURES

### Laboratory Chemistry

Water quality parameters will be measured in the laboratory, using standard operating procedures outlined in Appendix 3.

## 7.0 CUSTODY PROCEDURES

Sample custody will begin with the crew chief and samples are to remain in the custody of the field team until the samples are returned to the appropriate laboratory shipping and receiving room for entering into the sample tracking system. A chain-of-custody form will be completed for all samples. This form will include the sample date, sample time, sample site, and the name of the person collecting the sample. An example chain-of-custody form is attached in Appendix 5.

All sample sites will be assigned a designated number. Sites will be consecutively numbered and all standardized data forms generated from a site will be indexed and computerized according to that number.

Containers will be preserved, labeled, and placed in a sealed cooler for transport to the laboratory. Samples will be retained in the laboratory under chain-of-custody procedures. Samples will be inspected for leakage or damage from transport weekly. Loss of fluid preservatives for community samples will be replaced.

All raw data (including data forms, logbooks, etc.) are retained by the Project Manager (Greg Bright) in an organized fashion and archived for future reference.

## 8.0 CALIBRATION PROCEDURES AND FREQUENCY

Instrument calibration is needed for dissolved oxygen and pH. These instruments will be calibrated daily during each field survey. Records of the calibration will be kept in the field logbook.

## 9.0 PREVENTATIVE MAINTENANCE

The field crew leader is responsible for maintaining all files for all field equipment. Individual team members may be given responsibility for different equipment and its deployment in the field.

A list of critical spare parts that should always accompany field sampling surveys to minimize downtime follows:

- All equipment required in Standard Operating Procedures.
- Extra sample containers
- Extra batteries
- QAPP

## 10.0 DATA REDUCTION, REVIEW AND REPORTING

### 10.1 Raw Data

Field data will be recorded as it is taken. Laboratory data will be recorded on laboratory bench sheets

### 10.2 Data Reduction

Data will be transcribed to a Microsoft Access format.

### 10.3 Data Review

All chemical data will be checked for completeness before leaving a site. Data sheets from each site are checked by the field crew leader to verify accuracy and completeness.

### 10.4 Data Reporting

Chemical data will be reported in mg/l. E.coli data will be reported in MPN/100 ml.

The final report will be organized as a scientific document and shall contain the following sections:

- Table of Contents
- Table of Tables
- Tables of Figures
- Executive Summary
- Introduction
- Methods and Materials
- Results
- Quality Assurance

A final report of the data will be submitted electronically to IDEM in an Access Database format.

## 11.0 QUALITY CONTROL PROCEDURES

Field chemistry quality control procedures include the analysis of duplicate samples at ten percent of all sample sites.

Laboratory quality control procedures include the analysis of spikes, duplicates, and method blanks every tenth sample (see Appendix 3).

## 12.0 DATA QUALITY ASSESSMENT

Specific procedures for assessment of precision and accuracy on a routine basis are outlined and described in section 4.0. The data will be evaluated after each sampling event to assure that the data quality objectives are being met. If data fall outside the project goals of the Data Quality Objectives in Section Four, the laboratory will take corrective action, as stated in Section Fourteen. Data falling outside the data quality objectives will be flagged as follows:

R: Rejected

J: Estimated

Q: One or more of the QC checks or criteria was out of control

H: The analysis for this parameter was performed out of the holding time.

Results will be estimated or rejected on the basis of the following:

Estimated at less than 1.5 x the holding time

Rejected at greater than 1.5 x the holding time

D: Relative percent difference was above acceptable control limits.

Results will be estimated or rejected on the basis of the following:

Estimated at less than 2 x RPD

Rejected at greater than 2x RPD

B: Parameter found in field or lab blank.

Results will be estimated or rejected on the basis of the following:

Estimated at less than 10 x the blank contamination.

Rejected at greater than 10 x the blank contamination.  
U: Results are above the Method Detection Limit but below the reporting limit.  
Results will be estimated.

### 13.0 PERFORMANCE AND SYSTEMS AUDITS

Internal performance and system audits required to monitor the capability and performance of the laboratories will be conducted on appropriate log sheets, data sheets, verification sheets, and calibration equipment log sheets at each site in the field and after each of the two sampling seasons after all data have been collected.. All laboratory audits will be conducted by the Project Manager.

### 14.0 CORRECTIVE ACTION

If water chemistry analyses fall outside the objectives listed in Section Four or if field blanks indicate contamination, the lab or field personnel will not analyze any additional samples until a cause for the discrepancy has been identified. Sample results collected during this time will not be discarded but will be identified as potentially suspect.

### 15.0 QUALITY ASSURANCE REPORTS

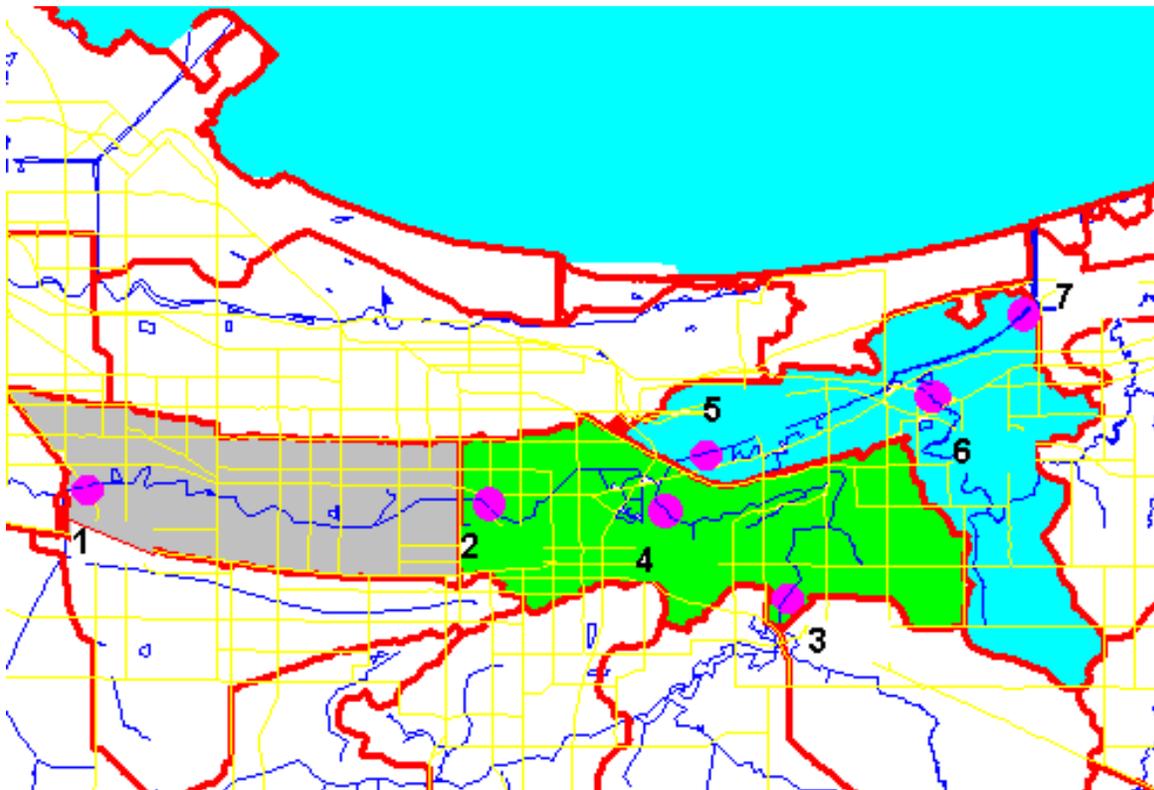
A quality assurance report will be prepared by the project coordinator and will include all pertinent information relating to measurement data accuracy, precision, and completeness, as outlined in the Standard Operating Procedures and this Quality Assurance Program Plan.

### REFERENCES CITED

1. Standard Methods for the Examination of Water and Wastewater, 18th Edition, Edited by Arnold E. Greenberg, Lenore S. Clesceri, and Andrew D. Lewis, 1992.

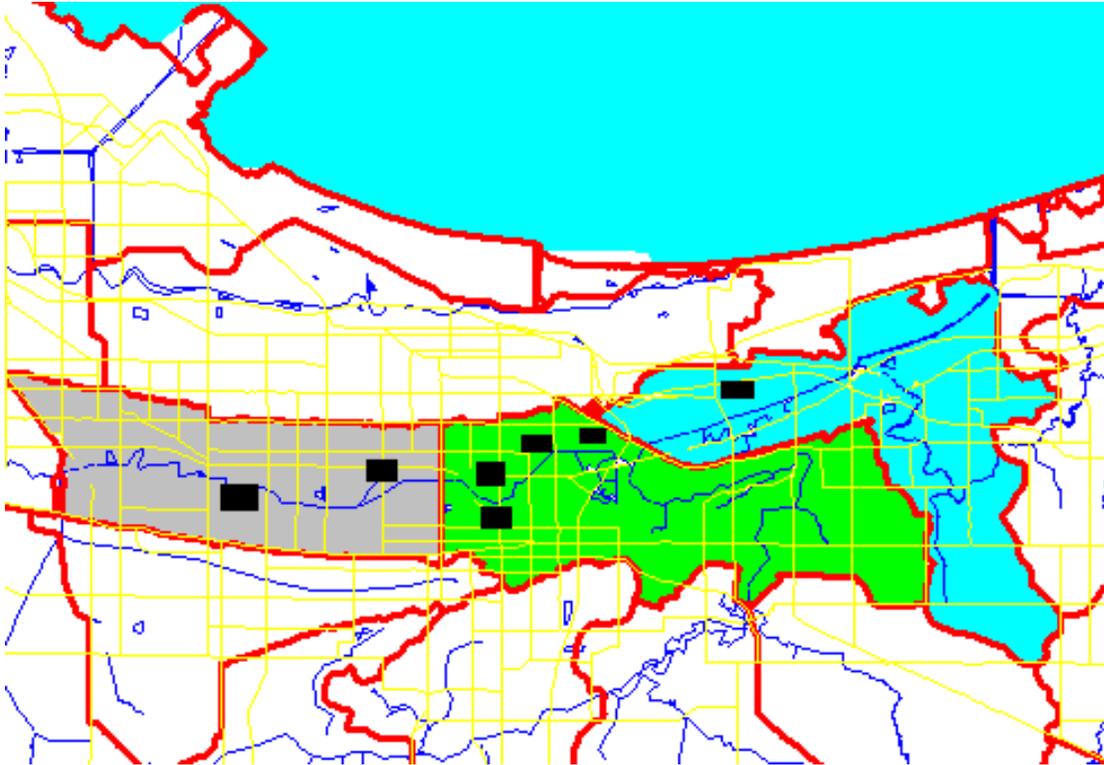
APPENDIX 1. - “Base” Sampling Sites for Chemistry

	Latitude	Longitude
1 Little Calumet River upstream	41.34.06	87.28.28
2 Little Calumet River above Deep River	41.33.56	87.21.20
3 Deep River upstream	41.32.14	87.15.18
4 Deep River downstream	41.33.47	87.17.27
5 Little Calumet River below Deep River	41.34.37	87.16.45
6 Willow Creek near mouth	41.35.34	87.12.45
7 Little Calumet River above Burns Harbor	41.36.10	87.11.35



### Additional Storm Sewer Outfall Sites

Multiple outfalls are present within each box. Latitude and Longitude values of individual outfalls will be assigned at sampling and reported in the final report



APPENDIX 2 - Standard Operating Procedures for Laboratory Water Chemistry

Total Suspended Solids  
Nitrogen (Nitrate + Nitrite)  
Total Phosphorus  
E. coli

## **Total Suspended Solids (TSS)**

### Reference

*Standard Method 18<sup>th</sup> Edition for the Examination of Water and Wastewater, 2540; A.*

### Sample Handling and Preservation

Samples are to be collected without any preservatives being added to them.

### Apparatus and Materials

- Analytical Balance
- Drying Oven
- Desiccator
- Vacuum pump
- Connection Tubing
- Baking pans used in drying oven
- Pre-weighed paper filters, with trays
- Suction Flask
- Membrane Filter
- Membrane Filter Funnel
- Clamp
- Metal or Plastic tweezers

### Reagents

Deionzied Water

### Procedures

Assemble the suctioning apparatus to filtering apparatus.

Place the membrane filter inside the suction flask

On the TSS record sheet write down the pre-weighed filter number and weight in the correct spaces provided. Place that filter on top of the membrane filter, then place the membrane funnel and clamp the funnel down to the suction flask.

Shake the sample to have a representative sample.

Pour off 100 ml of sample into the filtering apparatus

Pump air out of the filtering appratus.

Rinse the sides of the beaker with deionized water getting all particles off the walls of the beaker. Pour that into the membrane funnel with the rest of the sample. Once the sample has gone through the pre-weighed filter, rinse the funnel for any remaining particles.

After all water has been suctioned through the pre-weighed filter, turn off air manifold valve. Release the clamp. Remove the membrane funnel. Use the tweezers to remove the pre-weighed filter and place that filter in its original tray.

Before placing the next clean pre-weighed filter on the membrane filter, remember to clean the membrane funnel before the next sample is analyzed.

Place the tray in a baking pan that can be placed in the drying oven once the baking pan is full or all of the samples have been analyzed.

Weigh the filter after drying. Calculate TSS as the dry weight of the filter after drying minus then original weight of the filter.

#### Detection Limit

1 mg/l

#### Quality Assurance/Quality Control

There should be a duplicate analyzed every tenth sample.

**Nitrogen (Nitrate + Nitrite)**

1) Scope

This procedure uses cadmium reduction and a colorimetric technique to determine nitrite plus nitrate nitrogen.

2) Reference

Standard Methods 4500 NO<sub>3</sub>

3) Sample Handling and Preservation

Samples are to be collected with sulfuric acid in a pre-preserved bottle.

9.4 Apparatus and Materials

1) Colorimeter

9.5 Reagents

1) Hach NitraVer 3 and NitroVer 6 reagents

9.6 Procedures

1) Shake the sample container to get a well mixed sample

2) Pour off 5 ml. Add one packet each of Hach NitraVer 3 and NitraVer 6 reagents.

3) Allow color to develop for 30 minutes.

4) Place sample in a colorimeter. Measure absorbance at 540 nm.

5) Determine sample concentration by graphical interpolation.

7) Detection Limit - 0.5 mg/l

8) Quality Assurance/Quality Control

Duplicate every tenth sample. A method blank is analyzed every tenth sample and method blank spike preceding method blank, should be analyzed every tenth sample. Also a sample spike is to be analyzed with each batch. If a batch does not contain 10 samples, a method blank and method spike blank is to be analyzed along with that batch.

## **Total Phosphorus**

1) Scope

This procedure uses sample digestion, ascorbic acid, and a colorimetric technique to determine total phosphorus.

2) Reference

Standard Methods 4500 P F

3) Sample Handling and Preservation

Samples are to be collected with sulfuric acid in a pre-preserved bottle.

4) Apparatus and Materials

1) Colorimeter

2) Hot Block

5) Reagents

1) Deionzed Water

2) Nitric Acid

3) Hanna Phosphate Reagent (HI 93713-0)

6) Procedures

1) Shake the sample container to get a well mixed sample

2) Take the well-mixed sample and pour 50 mL into the digestion cups.

3) Add 1.5 mL of concentrated nitric acid into the sample.

4) Heat in the hot block at sample temperature of 95°C until sample is approximately 5 ml.

5) Remove samples from the hot block and allow sample to cool. Bring the sample volume back up to 50mL with DI water.

6) Once sample has been digested, pour off 10 ml. Add one packet of Hanna phosphate reagent.

7) Allow color to develop for 30 minutes.

8) Place sample in a colorimeter. Measure absorbance at 660 nm.

9) Determine sample concentration by graphical interpolation.

7) Detection Limit - 0.03 mg/l

8) Quality Assurance/Quality Control

Duplicate every tenth sample. A method blank is analyzed every tenth sample and method blank spike proceeding method blank, should be analyzed every tenth sample. Also a sample spike is to be analyzed with each batch. If a batch does not contain 10 samples, a method blank and method spike blank is to be analyzed along with that batch.

### **Appendix 3. Bacteriological Analysis - E. coli**

#### Location

This procedure is performed in the bacteriological laboratory of Commonwealth Biomonitoring

#### Purpose

This method is used to determine the number of colonies of Escherichia coli (E. coli) in environmental samples.

#### Scope

This procedure uses the m-colibblue technique with filtration

#### Reference

*Standard Methods 20<sup>th</sup> Edition – Method 9223 B*

#### Sample Handling and Preservation

Samples are to be collected in a sterile bottle provide by the lab.

#### Apparatus and Materials

Petri Dishes  
Filter Assembly  
Incubator

#### Reagents

m-colibblue

#### Procedures

Filter 100 ml sample through sterilized filter apparatus. Remove filter and place in Petri Dish with m-colibblue media. Incubate at thirty-seven degrees C for 24 hours. Count the number of colonies present and record on the attached data sheet.

#### Quality Assurance/Quality Control

A blank sample is analyzed with every batch, to provide assurance of a contamination free work area for that day. Duplicates are analyzed every tenth sample.

BACTERIOLOGICAL DATA  
m-Colibblue Procedure

SAMPLE DATE

SAMPLE TIME

ANALYSIS DATE

ANALYSIS TIME

TYPE OF SAMPLE

DILUTIONS USED

SITE NUMBER

RED COLONIES

BLUE COLONIES

TOTAL  
COLONIES

non-E. coli

E. coli

Total  
coliforms

1

2

3

4

5

6

7

8

9

10

11

12

13

APPENDIX 4. CHAIN OF CUSTODY FORM

**Commonwealth Biomonitoring, Inc**  
**8061 Windham Lake Drive**  
**Indianapolis, IN 46214**  
**317-297-7713**

**CLIENT NAME:** Gary Storm Water Utility  
**PURPOSE OF SAMPLE:** Water quality monitoring  
**SAMPLE IDENTIFICATION NUMBERS:**  
**DESCRIPTION:** \_\_\_\_\_  
**DATE SAMPLE COLLECTED:** \_\_\_\_\_  
**NAME OF PERSON COLLECTING SAMPLE:** \_\_\_\_\_  
**VOLUME OF SAMPLE:** \_\_\_\_\_  
**SAMPLE CONTAINER:** \_\_\_\_\_  
**NUMBER OF CONTAINERS:** \_\_\_\_\_  
**SAMPLE STORAGE:** \_\_\_\_\_  
**PRESERVATIVES:** \_\_\_\_\_

**Relinquished by:** \_\_\_\_\_

**Date:** \_\_\_\_\_ **Time:** \_\_\_\_\_

**Received by:** \_\_\_\_\_

**Date:** \_\_\_\_\_ **Time:** \_\_\_\_\_

**Relinquished by:** \_\_\_\_\_

**Date:** \_\_\_\_\_ **Time:** \_\_\_\_\_

**Received by:** \_\_\_\_\_

**Date:** \_\_\_\_\_ **Time:** \_\_\_\_\_

**COMMENTS:**