Ohio EPA Credible Data Program –
QAP: Quality Assurance Pan

Butler County Streams –
Water Quality Stream Sampling Program

March through November 2016

Project Leaders:

Tera Ratliff – QDC# 00989
Miami University
212 Pearson Hall
700 East High Street
Oxford, OH 45056
(513) 529-0180
ratliftj@miamioh.edu

Lynn White – QDC# 00422
Butler Soil and Water Conservation District
1802 Princeton Rd
Hamilton, OH 45011
(513) 785-6666
WhiteLR@butlercountyohio.org

Robert Lentz – QDC# 00423
Butler County Storm Water District
1921 Fairgrove Avenue
Hamilton, Ohio 45011-1965
(513) 785-4101
LentzB@stormwaterdistrict.org

Teresa Barnes – QDC# 00982
Butler County Storm Water District
1921 Fairgrove Avenue
Hamilton, Ohio 45011-1965
(513) 785-4142
Barnest@bceo.org

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3. Quality Assurance Policy

Butler County Stream Team is a volunteer stream-monitoring program designed to help fulfill the education and public outreach component of the Butler County Storm Water District's (BCSWD) NPDES Phase II storm water permit. The goal of the program is to provide the most accurate and reliable data possible while engaging the community and educating them about streams, human impacts, and storm water flow.

All samples will be collected by a team of volunteers who have attended a training session and performed the necessary steps to reliably collect a water quality sample for a project leader. Signatures of volunteers document when they received this training (See Appendix A). Any samples not collected by a trained volunteer will not be accepted.

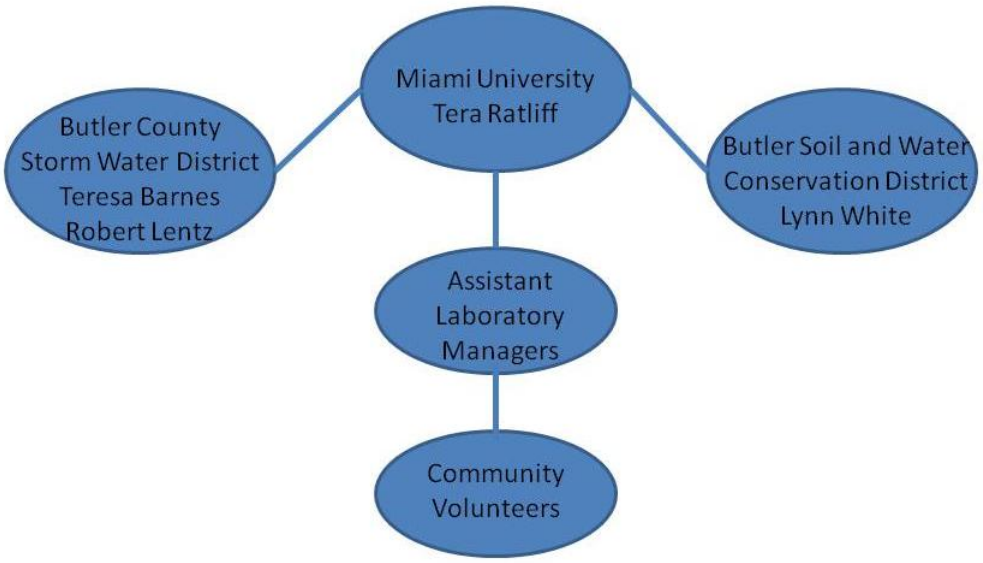
The laboratory will use only equipment and protocols certified as appropriate for collecting OEPA Level II credible data. Community volunteers participate in the analysis of the water samples. All volunteers will receive training on proper protocol for handling samples and equipment. At least two project leaders will be present in the laboratory supervising the volunteers.

Each QDC Level 2 project leader involved will support the Quality Assurance Plan (QAP) in principle and detail. Each project leader will be alert to problems and sources of error, will advise volunteers and other project leaders when errors in protocol are observed, and will consider how to make procedures more accurate and reliable.

This Quality Assurance Manual for the Butler County Stream Team has been specifically designed to ensure the production of technically sound, legally defensible data of proven quality in a timely manner.

4. Laboratory Organization and Responsibility:

Butler County Stream Team laboratory is housed in Pearson Hall on the main campus of Miami University in Oxford, Ohio. Tera Ratliff is employed with Miami University and will serve as the Laboratory Manager for the project. Tera will be assisted in the laboratory each month by Teresa Barnes, Robert Lentz, Lynn White and/or graduate students who are assigned to the project. Tera and the graduate assistants will directly supervise any volunteer participating in the laboratory.



Position Descriptions:

Laboratory Manager: The Laboratory Manager is responsible for overseeing the day to day operations of the Laboratory. The Laboratory Manager is the final authority on all issues dealing with data quality and has the authority to require that procedures are amended or discontinued, or analyses suspended or repeated. The Laboratory Manager is QDC Level 3 certified.

Assistant Laboratory Managers: There will be one full time and one part time Institute of Environmental Studies Graduate student assisting the Laboratory Manager. Prior to supervising volunteers within the laboratory, each will be trained in all aspects of laboratory management. Each will assist the laboratory manager as needed on lab maintenance and preparation, stocking and ordering of supplies, resolving lab problems, etc.

Additional Team Members: As listed on the cover page, all team members have a Bachelors of Science Degree and QDC Level 2 certification.

Training Procedures:

In order to be selected as a project participant, volunteers will attend the mandatory annual training held February 13, 2016. During this training session, the volunteers will be instructed (both with verbal descriptions and physical demonstrations) how to properly collect a Chemical Water Quality grab sample. This includes information such as :

- The sample bottles will be triple rinsed with water from the sampling site before a representative sample is taken.
- The samples will be collected between 7:30 and 10:00 am on the second Saturday of each month.
- Volunteers are required to sign the samples into one the five provided iced cooler by 10:30 am.
- Volunteers will be required to demonstrate their understanding of the procedures.
- Volunteers are provided with clean sample bottles, labels for the specific sample site, a safety vest and a device to collect the sample from the bridge. (Due to site restrictions, there are a few sites that are not collected from a bridge.)

Team members and volunteers attend additional training as available and necessary.

Volunteers will also assist with the laboratory analysis of the samples. There will be a maximum of 9 volunteers within the lab during each sampling date. These volunteers will receive individual and specific training for procedures being performed. Both the volunteer and trainer will sign and date a station specific document once the station specific training is received. The volunteers will be observed and monitored by no less than two (2) of the above listed QDC members and laboratory assistants(s). For methods that require increased technical laboratory skill, the volunteer will be directly supervised during the analysis procedure.

Project Leaders meet quarterly to review and discuss any issues and/or processes and attend additional training as available and necessary.

5. Data Quality Objectives:

Precision

The laboratory objective is to meet the precision demonstrated for the analytical methods on similar samples and to meet data for the analysis published by the US EPA. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability).
Precision is documented on the basis of replicated analysis, through lab duplicate samples. The required precision for duplicate samples greater than 2 times the reporting limit is 25% Relative Percent Difference (RPD).

Accuracy

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for these analytical methods on similar samples and to meet the recovery data published by the US EPA. Accuracy is defined as the degree of bias in a measurement system. Accuracy is documented on the basis of using laboratory control samples. The required accuracy for laboratory control samples is +/- 10%.

Reporting Limits

The reporting limits must be equal to or greater than the calculated Method Detection Limit (MDL) for a procedure. Ideally the reporting limit will be the MDL at a minimum. Values that fall below the MDL shall be reported but limited as below the MDL. The standard reporting limits for the Butler County Stream Team are listed below:

Table 1

Parameter Tested	**Laboratory Analysis Method**	**Detection Limit**
Bacteria	IDEXX Colilert QuantiTray 2000	10-24190/100mL
Conductivity	Hach SensION5 Direct Measure Method EPA 2510-B	0.1µS – 199mS
Nitrate	Hach TNT plus, LR 835	0.23 - 13.5mg/L
Total Phosphorus	Hach TNT plus, LR 843	0.15 – 4.5mg/L
Total Dissolved Solids (TDS)	Hach SensION5 Empirically derived using Conductivity value	~ 0.1 – 100g/L
Turbidity	LaMotte 2020e EPA Method 180.1	0 – 2000 NTU

Quality Control Samples

For this monitoring program, the following laboratory quality control samples will be analyzed; standards, blanks, known samples and lab duplicates.

6. Sample Collection and Analytical Method Variances:

Sample Collection Variances

For this monitoring program, samples are being collected by a dedicated crew of trained volunteers. While each volunteer will receive the same training, there may be slight variances within the method of sample collection between volunteers; in addition, there are times that a volunteer cannot participate on the given Saturday for a month. A trained volunteer will be found to be a substitute and collect the samples; there might be some variation between the usual and substitute sampler in collection methods. Although all samples are analyzed within acceptable holding times, there may exist slight variations in cooler holding times dependent on the cooler location. Every effort will be made to minimize these variances.

Analytical Method Variances:

The laboratory will be using volunteers to assist in the performance of the analytical tests. Every effort will be made to eliminate variances between volunteers. These volunteers will receive individual and specific training for procedure being performed. The procedure for each method will be posted at the station. Both the volunteer and trainer will sign and date a station specific document once the station specific training is received. The volunteers will be observed and monitored by no less than two (2) of the above listed QDC members and laboratory assistants(s). For methods that require increased technical laboratory skill, the volunteer will be directly supervised during the analysis procedure.

7. Laboratory Equipment and Instrument Lists:

The Laboratory uses standard equipment for each parameter analyzed as described in the Standard Operating Procedures (SOP). An alternate method will only be used if absolutely necessary. Please see the Standard Operating Procedures in Appendix B for additional information.

8. Sample Receipt and Custody Procedures:

Receiving Samples:

A Stream Team member will place a cooler at designated location by 8:00 am on the second Saturday of each month of the program. Chain of custody procedures are followed.

Sample Login:

As samples are logged into the lab for analysis, the sample is shaken vigorously 25 times and given a number. The sample number is placed on pre-printed labels for that sample site, the labels are placed on the prepared containers and a small portion of the sample is then poured into the prepared containers. For the laboratory duplicate, the same sample is poured into two separate prepared containers. The containers are then distributed to each station for analysis. The portion of the sample used to test for bacteria remains in the original sample bottle.

Sample Security:

Each cooler is secured with a lock. Only trained volunteers and laboratory personnel are provided access to the lock.

Sample Storage:

This is not applicable as each sample is anaylzed within 6 hours of receipt at the laboratory.

Sample Tracking:

Samples can be tracked using the computer system. The data is also accessible from the Streambank.info website. It allows for archiving of analytical results and electronic reporting. In addition to the computer data file, the raw data exists as laboratory bench sheets, which are archived both as a paper file and scanned electronically.

Sample Disposal:

Once analysis is completed and reviewed samples are disposed of properly.

9. Laboratory Standard Operating Procedures:
See Appendix B for SOP's for each method.

10. Calibration Procedures:
See Appendix B for SOP's for each method.

11. Preventive Maintenance and Documentation:

Location of Instrument Manuals

The equipment manuals are located in the lab near the equipment. They are also available on the lab computer in pdf form.

Schedules for Performance of Routine Equipment Maintenance

Routine and critical maintenance requirements for each instrument are detailed in the Standard Operating Procedures. Analytical balances are serviced and checked on an annual basis by the manufacturer.

12. Internal Quality Control Checks:

A sample bottle of ASTM Type 1 Water is placed in each sample collection cooler at the time the cooler is placed at the drop-off point. This is run through the lab as a sample, to guard against sample bottle contamination.

Each 10^{th} sample that is processed through the lab will be processed twice, as a lab duplicate.

Two (2) matrix spike/matrix spike duplicates per month will be run through the stations.

All volunteers will provide 2 duplicate samples over the course of the project period. During their assigned month, the volunteer will take the first water sample and label as usual. Once this is complete, the volunteer will then take a second sample, exactly as he/she collected the first sample. This bottle will receive a "duplicate" sample label.

13. Data Reduction Review and Reporting:

Data Reduction

Nitrate and Phosphate are reported to 3 significant figures.
Turbidity and pH are reported to 2 significant figures.
Total Dissolved Solids and Conductivity are reported to 1 significant digit for values less than one (1) and rounded to the nearest whole number for values higher than one (1).

Data Review:

Data review is first done by the analyst then is checked by the lab supervisor who then approves it for reporting.

Data Reporting:

Data reporting is done by the laboratory supervisor or appointed designee.

14. Standard Corrective Action Procedure:

Errors, deficiencies, deviations, or laboratory events or data that fall outside of established acceptance criteria should be investigated. In some instances, corrective action may be needed to resolve the problem and restore proper functioning to the analytical system. The investigation of the problem and any subsequent corrective action taken is documented in the Maintenace Records Books.
Check data for calculation or transcription error. Correct results if errors have occurred.
Check to see if sample(s) were prepared and analyzed according to the approved method and SOP. If it was not, prepare and/or analyze again.
Check calibration standards against an independent standard or reference material. If calibration standards fail, re-prepare calibration standards and/or recalibrate instrument and reanalyze affected sample(s).
If a known sample fails, analyze another known sample.
If a second sample fails, recalibrate instrument and reanalyze affected sample(s).
If a method blank fails, analyze another method blank.
If a second method blank fails, re-prepare and reanalyze affected sample(s).

15. External and Internal Audits and Accreditations:

Internal performance Audit is an independent check, by the lab manager to evaluate the data produced by the laboratory's analytical system. The performance audit verifies the ability of the lab staff to quantify compounds in check samples. The lab maintains current QC data sheets and certified values. These samples are analyzed as part of the routine operations. QC samples are indicators of the analysts' ability, the procedure, and the instrumentation. If QC samples do not fall within the limits established by the provider or within house limits, and investigation is begun to determine the source of the problem. Any problems are reported by the Lab Manager to other partners. All team members meet quarterly to discuss problems and/or issues with documented meeting minutes.

16. Document Retention and Control:

Laboratory bench sheets (LBS) are used to record information from routine laboratory operations, including sample preparation and analysis. Individual bench sheets are designed for each parameter. Pages are dated and scanned into the computer. Hard copies are stored for 3 years.

Data from the LBS are entered into an excel spreadsheet; all parameters analyzed are stored associated with the sample site information. These excel spreadsheets will be stored electronically and uploaded to streambank.info.

17. Procedures for Procurement and Process Control:

Reagents, Standards, and Supplies

Brand names and product numbers for any reagents and/or standards used in sample processing is contained in the method Standard Operating Procedure (SOP). In the event an item needs to be substituted due to time constraints or discontinuation, the substitute must be approved by the lab manager. It is the responsibility of the laboratory personnel to date all chemicals as they are received as well as when they are opened. Hard Copies of the Material Safety Data Sheets are kept at the laboratory work stations and digital copies are available on the laboratory computer. Water used in the preparation of standards or reagents must be Type 1 ultrapure water. See SOPs for additional information.

Purchasing Equipment

The Institute for the Environment and Sustainability (IES) at Miami University is financially responsible for the purchase and maintenance of all laboratory equipment. When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the laboratory manager will be responsible for obtaining quotes and securing the universities approval for the purchase. The IES director has the final purchasing decision.

Service to analytical instruments (except analytical balances and pipettes, which are serviced annually) is performed on an as need basis. See SOPs for additional information.

Appendix A

Butler County Stream Team 2016 Training

Name (printed)	Name (signature)	Date
Albert, Suellen	Suellen Albert	2-13-16
~~Alexander, Austin~~		
Ballenger, Laurie	Laurie Ballenger	2/13/2016
Brooks, Mary	Mary Brooks	2/13/2016
Burcham, Dave	Dave Burcham	2/17/2016
~~Cameron, Amy~~		
Chrisman, Dave	Dave Chrisman	2/13/16
Coffin, Susan	Susan Coffin	2/13/16
Coffin, Nataniel	Nathaniel COFFIN	2/13/16
Cullum, Mary	Mary Cullum	2/13/16
ERNSTING ~~Ernstein~~, Kent	Kent Ernsting	

Butler County Stream Team 2016 Training

Name (printed)	Name (signature)	Date
Gage, Holly	Holly Gage	2/17/2016
Gaker, Karen	Karen Gaker	2-13-16
Holliday, Chuck	Chuck Holliday	2-13-16
Hooke, Anne	OUT OF THE COUNTRY	
Jones, Elise Elisa	Elise Jones	2-13-16
Jones, Emma	Emma B Jones	2-13-16
Leap, Walter	Walter Leap	2/13/16
McCollum, Donna	Donna S McCollum	2-13-16
Nurabas, Theresa	Theresa Nurabas	2/13/16
Obrebski, Jean Ann	Jean Ann Obrebski	2/17/16
Obrebski, Chelsea	Chelsea Obrebski	2/17/16

Butler County Stream Team 2016 Training

Name (printed)	Name (signature)	Date
Paddock, Nathan		
Saunders, Charlie		2/13/16
Schneider, Al		2/13/16
Stuck, Rich		2/17/16
Thomas, Beverly		2/13/2016
Towers, Pat		2/22/16
~~Towers, Kathleen~~		
Walker, Bill		2/13/16
Zazycki, Suzi		2/17/16
Terry Haynes-Toney		2/13/16
Gabby Garrett		2/13/16

Butler County Stream Team 2016 Training

Name (printed)	Name (signature)	Date
Katrina Hyde	Katrina Hyde	2/13/16
Paige Hyde	Paige Hyde	2/13/16
Amy Paddock	Amy Paddock	2/13/16
Ellen Guest	Ellen Guest	2/13/16
Brigitte Cornett	Brigitte Cornett	2/13/16
Elizabeth Downs		2/13/16
LYNN WHITE		2/13/16

e_guest45387@yahoo.com

Butler County Stream Team 2016 Training

Name (printed)	Name (signature)	Date
Barnes, Teresa		2/13/16
Crout, Kelly		
Downs, Elizabeth		2/13/16
Goins, Chuck		
Hoit, Daniel		
Lentz, Bob		2/13/16
Ratliff, Tera		
White, Lynn		2/13/16

Appendix B

Conductivity

Written by: Tera Ratliff

Date: 1-March-2016

SUMMARY

This procedure is used for measuring conductivity and total dissolved solids in water samples using a Thermo Scientific Conductivity/TDS/Salinity meter.

EQUIPMENT AND SUPPLIES

Conductivity meter	180µS/cm standard
Stir bars	kimwipes
Gloves	Stir plate

NOTES

- Samples need to be analyzed as soon as possible (within 6 hours of receipt at the lab).
- **Stir bars used in this method are to be cleaned by triple rinsing with DI water. Do not acid wash!**

STANDARDS

1. 180µS/cm conductivity standard is purchased from the Hach company (product # 2307542). This solution is poured into a cup for calibration in sampling days. The used standard is disposed of at the end of the day and the stock is replaced on a bi-annual basis. This solution is used to calibrate the conductivity and total dissolved solids at the beginning of each sampling date. **The calibration must be completed before any samples can be run and standard must read within 10% error before proceeding with samples for the day!**

SAMPLE PREPARATION AND STORAGE

Sample are collected in coolers containing ice packs to keep them as cool as possible in the field and should be stored at 4˚ C immediately upon arrival in the lab. Samples are to be analyzed within 6 hours of arrival at the lab.

SAMPLE ANALYSIS

1. Make sure meter is on and ready to take a measurement. If this is not the case, find a lab manager and inform them of the issue.
2. Rinse electrode with DI water over waste cup. Blot (don't rub) off excess with a Kimwipe.
3. Place the plastic cup containing the stir bar and sample on the stir plate and turn on.
4. Place the electrode in the sample, being sure that the metal prong housed within the electrode opening is submerged. If you are not sure what this means, please see a lab manager for clarification.
5. Press TDS to measure Total Dissolved Solids, and record the value on the data sheet.
6. Press Cond to measure Conductivity, and record the value on the data sheet. The order of these measurements is not important, but make sure that you are placing them in the correct column on the data sheet.
7. Repeat steps 2-6 for the remaining samples.

CLEAN UP

Throw used kimwipes, and alcohol swabs in the trash. Straighten bench space. Dump waste container and turn off all equipment. Place stir bars by the sink to be washed.

Turbidity

Written by: Tera Ratliff

Date: 1-March-2016

SUMMARY

This procedure is used for measuring turbidity in water samples using a LaMotte 2020 turbidity kit.

EQUIPMENT AND SUPPLIES

LaMotte 2020 turbidimeter
Stir bars
Gloves
10 NTU turbidity standard
kimwipes
Stir plate

NOTES

- Samples need to be analyzed as soon as possible (within 6 hours of receipt at the lab).
- Make sure glass vials used in this procedure are free from scratches.
- **Stir bars used in this method are to be cleaned by triple rinsing with DI water. Do not acid wash!**

STANDARDS

1. 10 NTU turbidity standard (EPA compliant) is purchased from the Fisher company (product # 03920554). This solution is replaced on an annual basis. This solution is used to calibrate the turbidimeter at the beginning of each sampling date. **The calibration must be completed and standard must read within 10% error (blank must read <0.1) before any samples can be run!**

SAMPLE PREPARATION AND STORAGE

Sample are collected in coolers containing ice packs to keep them as cool as possible in the field and should be stored at 4˚ C immediately upon arrival in the lab. Samples are to be analyzed within 6 hours of arrival at the lab.

SAMPLE ANALYSIS

1. Make sure turbidimeter is on and ready to take a measurement. If this is not the case, find a lab manager and inform them of the issue.
2. Place the plastic cup containing your sample on the stir plate and insert a stir bar into the sample. Let the sample mix before pouring it into the sample vial.
3. Fill the vial to the line with the sample. Pour sample down the side of the vial to avoid bubbles. Cap the vial and wipe with a Kimwipe to remove any residual.
4. Insert the vial into the turbidimeter, making sure to line up the notch on the tube with the arrow on the instrument. If you are not sure what this means, please see a lab manager before proceeding.
5. Close the lid, and press OK to scan the sample. Record the results on the datasheet.
6. Give the plastic cup containing the remaining sample and the stir bar to the person at the conductivity station.
7. Discard the water from the vial into the waste container. Rinse 3 times with DI water. This vial can be re-used for the remaining samples today as long as you rinse 3 times between each one.
8. Repeat steps 2 through 7 for the remaining samples.

CLEAN UP

Throw used kimwipes, and alcohol swabs in the trash. Straighten bench space. Dump waste container and turn off all equipment.

Total Coliform and *E Coli*

Written by: Tera Ratliff

Date: 1-March-2016

SUMMARY

This procedure is used for measuring total coliform and *E Coli* in water samples using IDEXX Colilert and Quanti- Tray 2000 kits.

EQUIPMENT AND SUPPLIES

Isotemp forced air incubator at 37°C
pipette tips
Autoclaved ultrapure (Type 1) water
Colilert Kit and Quanti-Trays
100mL graduated cylinder (sterilized)
Autoclave Tape
Quanti-Tray sealer
UV Lamp
10mL pipette
Sample Log
New sample bottles
Bunsen burner
Gloves
Autoclave
1L Wheaton Bottles

NOTES

- All glassware used for samples, standards, or blanks needs to be autoclaved for 30 minutes at 121°C prior to use.
- Samples need to be analyzed as soon as possible (within 6 hours of receipt at the lab).
- Pipettes used in this procedure need to be calibrated annually and checked using a balance on a monthly basis or as problems arise.
- Incubator temperature should be confirmed using a glass thermometer in a monthly basis. If the temperature is off, use departmental incubators on the 2nd floor, and call for service.
- **Always use gloves during all steps of this process.**

REAGENTS

1. All chemicals used in this procedure are included in the Colilert/Quanti-Tray 2000 combo kit. Use care when handling so as not to contaminate them.

CONTROLS

1. Positive and negative controls are run whenever the lot number of the IDEXX kit changes. An IDEXX quality control kit (#UN3373-WQC-TCEC) containing *Escherichia coli*, *Klebsiella pneumoniae* and *Pseudomonas aeruginosa* is used. Organisms are contained on a small disk and kept frozen. When ready to run controls, the disk is placed in 100mL of autoclaved water, shaken up, and colilert is added. This is then run through as a sample to test the effectiveness of the kit.
2. In addition to prepared controls, a negative control containing just autoclaved water and the colilert pack is run through at the end of aliquoting to ensure that contamination did not occur during the process.

SAMPLE PREPARATION AND STORAGE

Sample are collected in coolers containing ice packs to keep them as cool as possible in the field and should be stored at 4˚ C immediately upon arrival in the lab. Samples are to be analyzed within 6 hours of arrival at the lab.

SAMPLE ANALYSIS

Day Before Sampling:

1. Turn on incubator and set at 37°C.
2. Cover 100mL glass graduated cyinder with foil.
3. Fill autoclavable bottles with UV treated Ultrapure water found in the CAWS lab (267B).
4. Cap bottles and autoclave bottles and graduated cylinder using the liquid 6 setting (30 minutes at 121°C). Make sure that you mark some of the bottles with autoclave tape, so we know the cycle was successful.

5. Once bottles have cooled, set up Bunsen burner on clean bench space. Flame the tips of the graduated cylinder and the bottle of water that you are working with anytime the lid is replaced. **Never** sit the lid inside facing down on the counter. **Always** use gloves during all steps of this process.
6. Using the graduated cylinder, measure 90mL of autoclaved water into each bottle. Bottles used in this process should be either new or autoclaved. Cap immediately. Never sit caps on the countertop.
7. The last bottle you fill for the day should get 100mL of water instead of 90mL. This bottle serves as a negative control and needs to be run through steps 3 through 7 listed below in Day of Sampling.

Day Of Sampling:

1. Turn on Quanti-Tray Sealer to warm up.
2. Shake the sample bottle vigorously a minimum of 25 times.
3. Using a clean pipette tip, pipette 10mL of sample into a 90mL ultrapure aliquot bottle.
4. With the packet facing away from you, gently open Colilert pack and pour into bottle.
5. Shake gently to mix well.
6. Use one hand to hold open the Quanti-tray and squeeze gently. Pull the foil tab to open the tray. Do not touch the inside of the foil or the tray. Again, you should be wearing gloves!
7. Pour the mixture into the tray. Place the tray into the rubber insert with the well facing down, and tap gently a few times to release air bubbles.
8. Put tray through the sealer, and record the sample number and date on the back. Once you have 5-10 trays, place them in the incubator and record the samples numbers and time in the notebook.

Day After Sampling:

1. After 24 hours have passed (but before 27 hours), samples are ready to count. Two people must do the counts at all time to ensure accuracy. If at

any time both people do not agree on the counts, they must recount until they reach an agreed upon number. There is a color comparator in the fridge in 105 to settle disputes.

2. Yellow well are total coliform. Count the number of large and small wells for each sample and record the number.
3. Turn off lights and place trays under a UV light. Count the number of large and small wells that fluoresce, and record the number. These are *E Coli*.
4. Enter the values into the IDEXX MPN generator and record values.
5. Check values of blanks and controls. If any of the blanks are positive, record cooler that blank came out of and report it to the lab manager. If the positive control (if applicable) is not positive or the negative (if applicable) not negative, rerun the controls using the same lot number, and report this to the lab manager.

CLEAN UP

Turn off sealer. Throw used pipette tips and empty Colilert containers in the trash. Straighten bench space.

Nitrate

Written by: Tera Ratliff

Date: 1-March-2016

SUMMARY

This procedure is used for measuring nitrate in water samples using Hach TNT 835 low range nitrate kits.

EQUIPMENT AND SUPPLIES

Hach DR2800 Spectrophotometer
1000µl pipette
kimwipes
sample racks
pipette tips
Eye Droppers
Acid washed squeeze bottle
Analytical balance with at least 2 decimal places
200µl pipette
Hach TNTplus 835 kits
alcohol swabs
timer
100mL Volumetric Flasks
Ultrapure water (Type 1)
Gloves

NOTES

- All glassware (with the exception of the Hach kit vials) used for samples, standards, or blanks needs to be acid-washed prior to use.
- Samples need to be analyzed as soon as possible (within 6 hours of receipt at the lab).
- Never pour leftover chemicals down the drain or in the trash. There are appropriate receptacles located in the hood in Room 105.
- Check the date balances were last calibrated before using. They should be calibrated by a professional on an annual basis and checked using calibrated weights monthly.

- Pipettes used in this procedure need to be calibrated annually and checked using a balance on a monthly basis or as problems arise.
- Wear gloves at all times during procedure!
-

REAGENTS

1. All chemicals used in this procedure are included in the Hach TNT 835 kit. Use care when pipetting these reagents and dispose of appropriately.

STANDARDS

1. 100mg/L NO3-N (NIST) stock solution for making standards is purchased from the Hach company (product # 194749). This solution is stable in the refrigerator for about six months after opening or until expiration date on bottle (whichever comes first).
2. For all standards, mix stock in ultrapure (type 1) water. These should be prepared no more than 24 hours before each sampling event. Place a 100mL volumetric flask on the analytical balance, and tare. Add amount of 100mg/L stock standard given below using a new eye dropper. Use the squeeze bottle to dilute to mark with type 1 ultrapure water. Store in acid washed, glass, amber bottle. 0ppm is simply ultrapure water.

0.5ppm: 0.5g stock solution	2.500ppm: 2.5g stock solution
7.500ppm: 7.5g stock solution	10.00ppm: 10g stock solution .

   These standards are run by lab personnel at the beginning of each sampling day to test the efficiency of laboratory equipment. If the standards are off by more than 10%, recalibrate spec and rerun standards. If they are still off, record the values and make sure that linearity is retained in the event that sample values need adjusted at a later date (ex. purchased standards are also off by same amount).
3. In addition to prepared standards, a wastewater standard (Hach # 2833249), drinking water standard (Hach # 2833049), and 1mg/L No3-N standard (Hach #

204649) are run through as samples to test the accuracy of the volunteer running the samples. These standards need to be within 10% of their expected values in order for accuracy to be considered achieved.

SAMPLE PREPARATION AND STORAGE

Sample are collected in coolers containing ice packs to keep them as cool as possible in the field and should be stored at 4˚ C immediately upon arrival in the lab. Samples are to be analyzed within 6 hours of arrival at the lab. If it is not possible to analyze samples within this time range, samples must be frozen. Freezing samples should only happen in cases of extreme emergency or complete equipment failure.

SAMPLE ANALYSIS

1. Make sure vial is marked with correct sample number.
2. Check to make sure spectrophotometer is on and reads “please insert barcode cuvette”. If this is not the case, find a lab manager and inform them of the issue. If you are unfamiliar with using a pipette, please see a lab manager. **Please note:** Check the lot numbers on the sample box prior to starting a new box. Whenever the lot number on the sample vials changes, please rerun a blank and a standard before running any more samples. If the values are off or you are unsure how to do this, please see a lab manager.
3. Pipette 1000µL (1mL) of sample into the reagent vial using a new pipette tip and the 1000 µL (blue top) pipette. Place in test tube rack. You can set up about 5 samples before continuing to step 4.
4. Pipette 200µL of solution A into each of the reagent vials using a new pipette tip and the 200 µL (yellow top) pipette. Caution: Vial will get very warm upon addition of this chemical. Cap quickly and handle vial by the lid.
5. After capping, invert vial 2-3 times until no more streaks are present in the solution. Set timer for 15 minutes.

6. While reaction is occurring, wipe down vials with an alcohol swab to remove and dust, fingerprints, etc.
7. **Sample one is used to blank the machine. Please see a lab manager when this sample is ready.** When timer beeps, remove cover from spec. Place the vial into the cell holder, making sure to align barcode with arrow, and quickly replace cover. This test is time sensitive! Read results as quickly after reaction time as possible.
8. Record value on the data sheet. Note: If the machine reads **Under Range**, make sure to record that on the data sheet. If the machine reads **Over Range**, put sample aside to be diluted and rerun. For dilution: Pipette 500µL of sample and 500µL of ultrapure water into a new vial and repeat steps 3-6. Make sure to mark the sample on the sheet as diluted. Retain both undiluted and diluted values.

CLEAN UP

Throw used pipette tips, kimwipes, and alcohol swabs in the trash. Place capped reaction vials in designated disposal bin. Straighten bench space.

Total Phosphorus

Written by: Tera Ratliff

Date: 1-March-2016

SUMMARY

This procedure is used for measuring total phosphorus in water samples using Hach TNT 843 low range phosphorus kits.

EQUIPMENT AND SUPPLIES

Hach DR2800 Spectrophotometer
2000µl pipette
200µl pipette
kimwipes
sample racks
pipette tips
Eye Droppers
Acid washed squeeze bottle
Analytical balance with at least 2 decimal places
1000µl pipette
Hach TNTplus 843 kits
Hach DRB200 heating blocks
alcohol swabs
timers (at least 2)
100mL Volumetric Flasks
Ultrapure water (Type 1)
Gloves

NOTES

- All glassware (with the exception of the Hach kit vials) used for samples, standards, or blanks needs to be acid-washed prior to use.
- Samples need to be analyzed as soon as possible (within 6 hours of receipt at the lab).
- Never pour leftover chemicals down the drain or in the trash. There are appropriate receptacles located in the hood in Room 105.
- Check the date balances were last calibrated before using. They should be calibrated by a professional on an annual basis and checked using calibrated weights monthly.

- Pipettes used in this procedure need to be calibrated annually and checked using a balance on a monthly basis or as problems arise.
- Wear gloves at all times during procedure!

REAGENTS

1. All chemicals used in this procedure are included in the Hach TNT 843 kit. Use care when pipetting these reagents and dispose of appropriately.

STANDARDS

1. 50mg/L $PO4^{-3}$ (NIST) stock solution for making standards is purchased from the Hach company (product # 17149). This solution is stable in the refrigerator for about six months after opening or until expiration date on bottle (whichever comes first).
2. For all standards, mix stock in ultrapure (type 1) water. These should be prepared no more than 24 hours before each sampling event. Place a 100mL volumetric flask on the analytical balance, and tare. Add amount of 50mg/L stock standard given below using a new eye dropper. Use the squeeze bottle to dilute to mark with type 1 ultrapure water. Store in acid washed, glass, amber bottle. 0ppm is simply ultrapure water.

0.5ppm: 1g stock solution	1.000ppm: 2g stock solution
2.500ppm: 5g stock solution	4.00ppm: 8g stock solution .

These standards are run by lab personnel at the beginning of each sampling day to test the efficiency of laboratory equipment. If the standards are off by more than 10%, recalibrate spec and rerun standards. If they are still off, record the values and make sure that linearity is retained in the event that sample values need adjusted at a later date (ex. purchased standards are also off by same amount).

3. In addition to prepared standards, a wastewater standard (Hach # 2833249) and drinking water standard (Hach # 2833049 are run through as samples to

test the accuracy of the volunteer running the samples. These standards need to be within 10% of their expected values in order for accuracy to be considered achieved.

SAMPLE PREPARATION AND STORAGE

Sample are collected in coolers containing ice packs to keep them as cool as possible in the field and should be stored at 4˚ C immediately upon arrival in the lab. Samples are to be analyzed within 6 hours of arrival at the lab. If it is not possible to analyze samples within this time range, samples must be frozen. Freezing samples should only happen in cases of extreme emergency or complete equipment failure.

SAMPLE ANALYSIS

Digestion:

1. Make sure vial is marked with correct sample number.
2. Check to make sure heating blocks are on and heated to 100° C and spectrophotometer is on and reads “please insert barcode cuvette”. If this is not the case, find a lab manager and inform them of the issue. If you are unfamiliar with using a pipette, please see a lab manager. Please note: Check the lot numbers on the sample box prior to starting a new box. Whenever the lot number on the sample vials changes, please rerun a blank and a standard before running any more samples. If the values are off or you are unsure how to do this, please see a lab manager.
3. Unscrew cap from the vial, and gently place on counter thread side up. Make sure that you keep the reagent caps in order. In case of a lot number change within a batch, it is best that a cap remain with its original vial.
4. Pipette 2000µL (2mL) of sample into the reagent vial using a new pipette tip and the 2000µL (green top) pipette. Place in test tube rack.
5. Repeat steps 3 and 4 for the next 14 samples. Once you have 15 vials, carefully remove the protective foil from the vial lid. Flip the cap over the

vial so that the side containing the reagent is facing the vial opening. Screw cap tightly onto the vial.

6. Invert the vial 2 to 3 times to remove the reagent from the cap. Verify that there is no reagent left in the cap.
7. Insert vials into the heating block, making sure to maintain the sample order. Close the lid. Try to minimize the amount of time the heater blocks are left open to avoid unnecessary fluctuations in temperature.
8. Press start to begin the 60 minute digestion.
9. Repeat steps 2 through 7 for the next 15 samples. We have enough heating blocks to have 4 batches going at a time.
10. After the timer goes off, carefully remove the vials from the heater block. Place the vials in a rack, and place the rack in the refrigerator for 15 minutes or until they are around room temperature. Press start again on the heater block to keep it heated and reset the timer.

Color Reaction:

1. Pipette 200µL of Reagent B into the cooled sample vial using the 200 µL (yellow top) pipette. Try to minimize the amount of time the lid is off of the reagent container. It is light sensitive.
2. Screw the gray DosiCap C onto the vial.
3. Invert the vial 2 to 3 times to remove the reagent from the cap. Verify that there is no reagent left in the cap.
4. Set timer for 10 minutes.
5. While reaction is occurring, wipe down vials with an alcohol swab to remove and dust, fingerprints, etc.
6. When timer beeps, again invert the vial 2 to 3 times to mix.
7. Sample one is used to blank the machine. Please see a lab manager when this sample is ready. Remove the cover from spec. Place the vial into the cell holder, making sure to align barcode with arrow, and quickly replace cover. This test is time sensitive! Read results as quickly after reaction time as possible.

8. Record value on the data sheet. Note: If the machine reads **Under Range**, make sure to record that on the data sheet. If the machine reads **Over Range**, put sample aside to be diluted and rerun. For dilution: using 1000µL pipette (blue top), pipette 1000µL of sample and 1000µL of ultrapure water into a new vial and repeat steps all prior steps. Make sure to mark the sample on the sheet as diluted. Retain both undiluted and diluted values.

CLEAN UP

Turn off heater blocks. Throw used pipette tips, kimwipes, and alcohol swabs in the trash. Place capped reaction vials on designated disposal bin. Straighten bench space.

Acid Washing

Written by: Tera Ratliff

Date: 1-March-2016

SUMMARY

This procedure is used for cleaning glassware and sample bottles used in sample collection and nitrate and total phosphorus analysis.

EQUIPMENT AND SUPPLIES

Hydrochloric Acid	5 Gallon Tub
1L Graduated Cylinder	DI Water
Acid-Proof Gloves	Lab coat

NOTES

- Place glassware on side to dry to encourage air flow.
- **Always use gloves during all steps of this process.**
- **Make a fresh bath each time you wash dishes.**
- This solution will burn holes in your clothes. Please wear a lab coat to protect yourself.

REAGENTS

1. 1M HCl Bath: Under the hood: Fill the 5 gallon tub with 8L of DI water. Using the graduated cylinder, add 827mL of concentrated HCl. Using remaining 1.173L (still under hood), rinse the graduated cylinder to remove remaining acid and add to tub. **Caution: Always add acid to water not the other way around. Heat is released when acid is added to water. Adding the water first, leads to a more dilute initial acid concentration, and a lower amount of heat is released. Rinse graduated cylinder under hood to avoid inhaling acid fumes.**

WASHING PROCEDURE

1. Rinse dishes 3 times in DI water.
2. Rinse dishes 3 times in the freshly prepared acid bath.
3. Rinse dishes 3 more times in DI water.
4. Place caps inside down on clean bench paper and bottles on their sides to dry.

CLEAN UP

Neutralize acid bath with sodium bicarbonate until it stops bubbling. Liquid can be poured down the sink once it has been neutralized. Rinse out the tub, and straighten the bench space.