EXAMPLE SMALL WASTEWATER TREATMENT PLANT LABORATORY QUALITY MANUAL*

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* Formerly titled "Quality Assurance Document for a Small Wastewater Lab"

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EXECUTIVE SUMMARY

Example Wastewater Treatment Plant Laboratory Quality Manual is designed to provide an example of the minimum documentation needed to fulfill the requirements of NR 149.37. This manual is not designed to be a complete guidance document for commercial laboratories, as it is written based on the analytical testing requirements associated with small wastewater treatment plants only.

ACKNOWLEDGEMENTS

In addition to the DNR staff from the various offices who provided assistance in writing, reviewing and providing suggestions for the document, there are also a number of external individuals, including the staff of the plant that served as the model for this document, who provided insight into wastewater laboratory operations.

Many of the Staff members of the Lab Certification and Registration Program (LCRP) also provided input for this latest version of the manual. The end result is the production of a document which will be invaluable to both the DNR staff and wastewater laboratory personnel as both a reference and guidance document.

Editor's Note: *This document constitutes the fourth edition of* EXAMPLE SMALL WASTEWATER TREATMENT PLANT LABORATORY QUALITY MANUAL. *This fourth edition is an extensive revision concomitant to the 2008 update of NR 149. Specific products and brand names listed in this manual are given as examples only and do not represent an endorsement by the Wisconsin Department of Natural Resources. Tree City is a fictitious community.*

This is an example quality manual and its intent is to demonstrate one way in which the requirements of NR 149.37 can be met. It is the responsibility of each facility to develop and follow their own unique quality manual.

PREFACE

Presented here is a copy of the quality assurance manual for the laboratory at the Tree City Wastewater Treatment Facility. Quality manuals similar to the one presented here are required by NR 149.37 (1). Also, they are valuable as a guide to maintaining analytical performance and assuring compliance with other requirements of NR 149.

This material is being distributed as guidance for use by laboratory personnel regarding elements of a quality assurance program. The Tree City facility is a small to medium sized (about 1 mgd of combined domestic and industrial wastewater). Only the essential elements of a quality assurance document are included in this "model" plan. Therefore this represents what might be considered a minimum program. Some laboratories may choose to expand this by providing more detail.

Please note that, where details are provided, they are specific to Tree City.

In addition to an application and payment of fees, NR 149 requires:

1. Following standard operating procedures (SOPs) based on approved methods of analysis.

2. Using approved methods for sample collection, handling, and preservation and performing all testing within regulatory holding times.

3. Analyzing and passing at least one reference sample per year for tests that require them.

4. Preparation and adherence to a written Quality Manual. (This manual can also be called a Quality Assurance Manual or any other applicable title)

5. Performance of quality control samples including analysis of blanks, Laboratory Control Samples (LCS), second source standards (i.e., Initial Calibration Verification (ICV), and continuing calibration verification (CCV))

6. Documentation which substantiates those requirements is being met. Records must be retained for at least three years.

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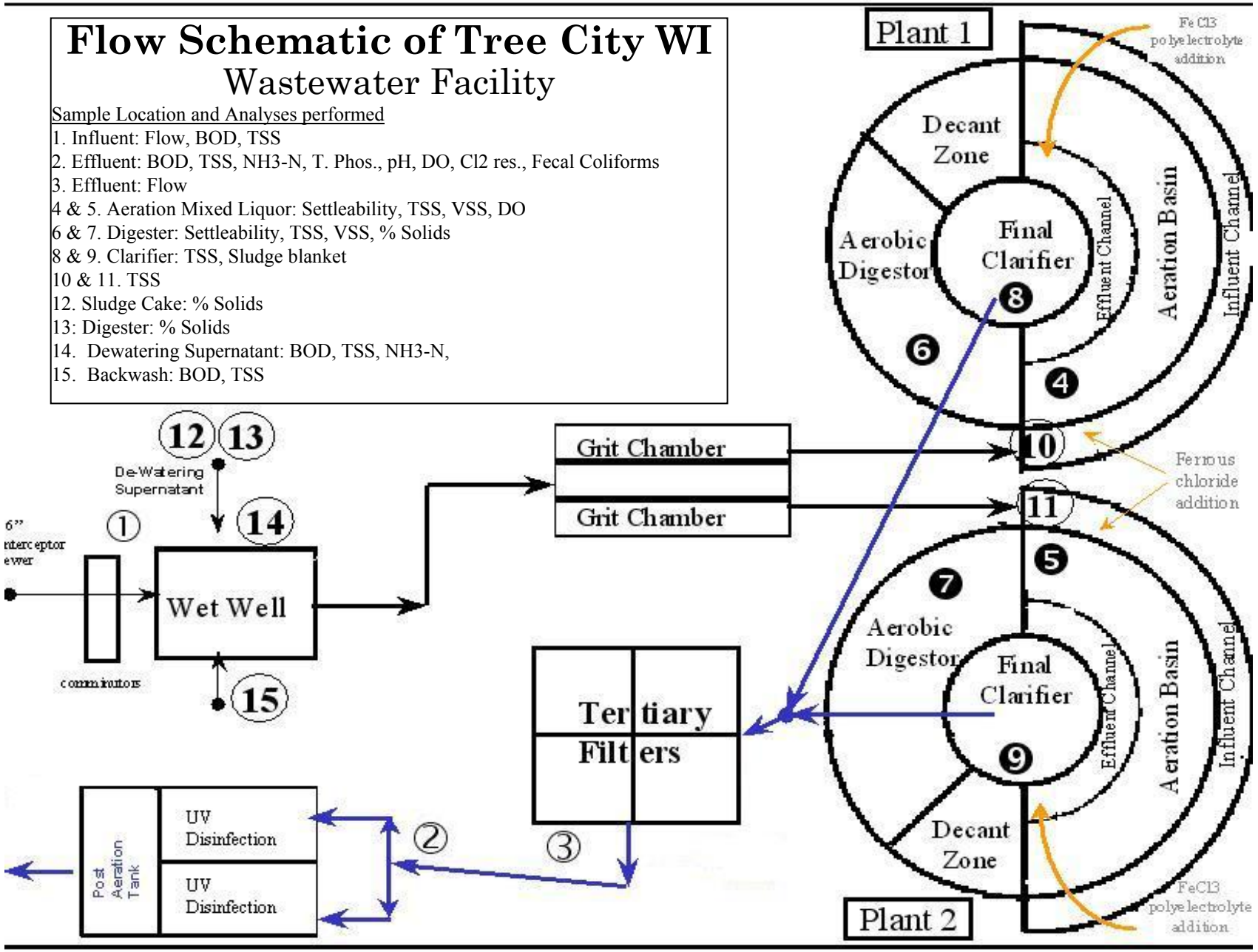
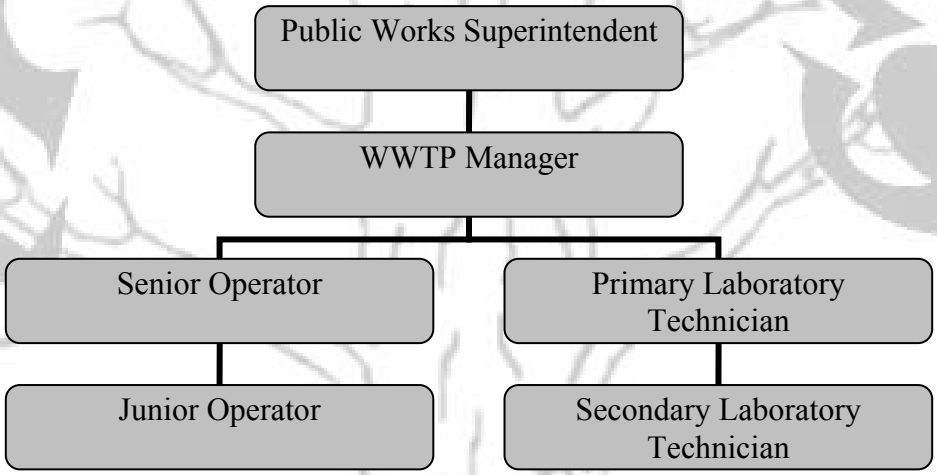


1. INTRODUCTION

The laboratory at the Tree City Wastewater Treatment Facility performs analyses necessary both for compliance with requirements specified by the plant's WPDES permit and process control. The lab may also be used to run tests for charge-back of treatment costs to industrial users and other communities. Quality Assurance (QA) is critical in producing sound, defensible data. These data provide the empirical evidence upon which decisions are based. The purpose of this manual is to outline QA activities performed in the lab and to fulfill the requirements set forth in NR 149.37.

2. LABORATORY ORGANIZATION AND RESPONSIBILITY

Organizational Structure of the Tree City Wastewater Treatment Plant (WWTP)



a. The individuals listed below are responsible for ensuring the production of valid laboratory measurements and the routine assessment of measurement systems for precision and accuracy.

i. Superintendent- Highly experienced plant operator with supervisory experience that is responsible for overall plant performance and compliance with WPDES permit. This includes affective wastewater treatment as well as the generation of valid and legally defensible data by the plant's internal analytical laboratory. The manager is trained and has extensive knowledge related to federal, state, and local laws which regulate wastewater treatment and discharge.

ii. Laboratory Technician- Individual with a sufficient combination of education, experience, and training to competently generate valid and legally defensibly analytical data. This person understands the fundamental conceptual theory behind the procedures performed. The person is familiar with and follows this QA manual, NR 149, and has intimate knowledge of all analytical methods. The primary lab analyst demonstrates these traits for all methods through the successful performance of Initial Demonstration of Capability (IDC), by ongoing success in the analysis of Proficiency Testing (PT) samples, and in regularly meeting all method quality control specifications.

iii. Backup Laboratory Technician - The backup analyst does the laboratory work when the primary laboratory analyst on the weekends, or when primary laboratory personnel is sick or on vacation. The same requirements of the main analyst are required of any backup, weekend, or fill-in analysts.

b. All analysts are required to perform an IDC for each method. Because the source methods (i.e., Standard Methods) upon which the analytical procedures performed at Tree City are based do not contain specific IDC procedures, laboratory management has instituted the following IDC methodology: The IDC consists of documenting that data generated by each new analyst meets all QC parameters for two consecutive analyses. The IDC analytical runs include a Laboratory Control Sample (LCS), where applicable, which is prepared by a second analyst. The LCS concentration is unknown to the analyst in training, when possible. At Tree City, the results of the LCS for the IDC need to be $\pm$ 15% to be acceptable. Copies of IDC documentation are permanently maintained in the employees training file. The IDC is a one-time requirement per method for each analyst.

3. PROCEDURES FOR RETENTION, CONTROL AND MAINTENANCE OF DOCUMENTS USED IN OR ASSOCIATED WITH ANALYSES

a. Records and Documents retention and control procedures.

i. All records of equipment calibration and maintenance, QC tests, sampling, standard and reagent preparation, and sample analysis are retained for at least three years (five years for sludge data) at the treatment facility office in fire resistant file cabinets.

ii. All raw data is kept, no matter how rough in appearance. If data contained on any record is transcribed to facilitate summarizing or neatness, the original record is also be kept.

iii. All observations are recorded in ink.

iv. Errors made in documentation are corrected by drawing a single line through the entry. The correct observation is then written next to original observation.

v. Records are available only to authorized laboratory staff.

b. Administrative records maintained

i. The laboratory's accreditation certificate from the Wisconsin Laboratory Certification program is conspicuously displayed on the wall near the laboratory entrance.

ii. Personnel records are maintained for all lab staff. These records include qualification, experience, training, and IDC documentation.

c. Analytical Records.

i. The Tree City Laboratory maintains all records containing raw data and calculations which are needed to reconstruct all results reported on the DMR for which the laboratory is registered.

ii. The laboratory has developed benchsheets for all routine analyses and documentation. Other data are recorded in applicable logbooks.

iii. The laboratory documents at least the following:

1. Sample ID- samples are identified by the sample site (i.e., influent or effluent) and collection date.

2. Analysis Time- unless the sample is not analyzed on the day the sample is collected by the lab, the analysis time and date is noted on the benchsheet.

3. Preservation status- samples arrive to the laboratory immediately after collection from refrigerated autosamplers. Therefore, samples by Tree City personnel are known to be thermally preserved when they arrive at the laboratory. Samples for which pH preservation is required are acid preserved as soon as possible after arrival at the laboratory. The preservation status of acid preserved samples is only periodically verified because the buffering capacity of the waste stream is known to be constant.

4. Analyst- the bench sheets indicate the analyst performing the testing as well as the intended analysis.

5. Analytical Procedure- all steps for which the samples are subjected are written out or referenced from the applicable method SOP.

6. Chemical Used- all standards and reagents used in the analysis are referenced on the benchsheet.

7. Data- raw data for both standards and samples are collected.

4. PROCEDURES FOR ACHIEVING TRACEABILITY OF STANDARDS, REAGENTS AND REFERENCE MATERIALS USED TO DERIVE ANY RESULTS OR MEASUREMENTS

a. Analytical Reagent and Standards

i. Purchased Materials

1. Only analytical grade reagents are used. Labels on all chemical reagents are marked with the date received, date opened, and expiration date. The reagent name, lot number, manufacturer, date of receipt, the date of expiration of purchased stock reagents are documented in a logbook upon receipt. Reagents are assigned an internal lot number based on the day they were received and the sequence in which they were logged. For example, a reagent which was the fourth to be logged in on March 31, 2008 is given the lot number 31MAR08D.

2. Standards are labeled and logged-in in the same manner as reagents.

ii. Prepared Materials

1. All in-lab prepared reagents and standards are labeled with the date they were prepared, the material's identity, expiration date, preparer's initials and Tree City assigned lot number. All standards and reagents prepared are assigned a unique lot number and an expiration date. All standards and reagent preparation is documented in a logbook. These records serve to link intermediate and working standards and reagents (children) to their respective originating stocks or neat compounds (parent material). The material name, Tree City assigned lot number, and expiration date of all raw substances used to prepare the material are documented. The procedure used to make the reagent or standard is described. Alternatively, the preparation procedure is referenced from the applicable SOP.

b. Reagent Water Quality

i. Reagent grade water is produced in the lab using a Barnstead Model A1015 still. Water used for ammonia measurements is also passed through a mixed-bed ion exchange column (Barnstead Bantam Deionizer).

Only freshly prepared reagent water is used for ammonia testing to prevent the water from picking up ammonia from the air. Reagent water used to make dilution water for BOD analyses is stored in glass carboys stoppered with clean cotton plugs. Reagent water for tests other than ammonia and BOD is stored in tightly stoppered glass carboys.

5. PROCEDURES FOR HANDLING SAMPLES

a. Samples are collected to fulfill permit requirements for testing plant influent, effluent, and hauled sludge as well as for industrial and process control monitoring. Wastewater testing requirements are summarized below. Schematic reference numbers correspond to those on the plant schematic (page IV).

Table 1 - WPDES Permit Requirements

SAMPLE LOCATION	SAMPLE TYPE	SCHEMATIC REFERENCE	PARAMETERS TESTED	MONITORING FREQUENCY
Influent	Continuous	1	Flow	Totalized Daily
Influent	24-hr composite (flow proportional)	1	Biochemical Oxygen Demand Total Suspended Solids	Daily
Effluent	24-hr composite (flow proportional)	2	Biochemical Oxygen Demand Total Suspended Solids Ammonia-Nitrogen Total Phosphorus	Daily
Effluent	Grab	2	Dissolved Oxygen pH	Daily
Effluent	Grab	2	Fecal Coliform #	Twice weekly

- Ultraviolet (UV) disinfection only required during the period from May 1 to September 30 in any given year.

i. The permit also requires that a sludge characteristic report be submitted annually for quarterly analyses. Sludge analyses for non-routine parameters are performed by a certified commercial laboratory.

b. Sample Handling

i. Samples are identified by collection site and date of collection.

ii. All samples from ammonia and phosphorous are acid preserved when brought into the laboratory.

iii. If analysis is not initiated immediately, samples are refrigerated.

Table 2 - Process Control Monitoring

SAMPLE LOCATION	SAMPLE TYPE	SCHEMATIC REFERENCE	PARAMETERS TESTED	MONITORING FREQUENCY
Aeration Tank	Outlet grab	4 & 5	Settleability (30 min.) Total Suspended Solids Volatile Solids	Daily
Aeration Tank	Contents in-place	4 & 5	Dissolved Oxygen	Continuous
Solids Concentrator	Product-grab	12 & 13	Percent solids	As needed
Solids Concentrator	Decant-grab	14	Biochemical Oxygen Demand Total Suspended Solids Ammonia-Nitrogen	As needed
Digester Contents	Grab	6 & 7	Settleability (30 min.) Percent solids Total Suspended Solids Volatile Suspended Solids	Daily
Clarifier	Grab	8 & 9	Blanket Depth Total Suspended Solids	Daily
Return Sludge	Grab	10 & 11	Total Suspended Solids	Daily
Filter Backwash	Grab	15	Biochemical Oxygen Demand Total Suspended Solids	As needed

c. Sampling

i. A flow-proportional automatic sampler is used to obtain sample from the influent channel upstream of both the raw wet-well and ultraviolet (UV) disinfection. These samplers receive signals from the influent flow meter so that sampling is done in a flow proportional mode. Samplers have refrigeration units that maintain sample temperatures at ≤ 6 °C.

ii. The operator collects samples from the automatic samplers at approximately 7:00 a.m. by replacing the filled polyethylene sample containers with clean containers and transporting samples directly to the lab. Flow meter readings and temperature of the automatic sampler are recorded when samples are collected. Samples

are uniquely identified by the sample date (date the majority of the 24 hour composite sample is collected), collection date (date sample is collected), collection site (i.e., influent or effluent), by sample type, and exact sample collection time. The hold time is calculated by the collection date, but the sample date is the date for which the results are reported. All sample bottles are clearly labeled with a durable marking with the sample identification, time and date of collection, chemical preservation, initials of sampler, and the intended analysis.

iii. Samples are allowed to equilibrate to room temperature while calibration checks are performed. Analyses begin at approximately 8:00 a.m.

iv. Grab samples are collected during the mid-afternoon by the operator in plastic bottles for direct transport to the lab. Samples for pH are tested as soon as they are brought back into the laboratory. Samples for fecal coliform are collected in a sterilized glass jar containing a drop of 10% sodium thiosulfate solution. Testing is initiated shortly thereafter.

v. Processed sludge for the required quarterly Sludge Characteristic Report is collected by compositing hourly grab samples. These are collected during a normal 7-hour run of the solids processing unit. Sludge samples collected for the analysis of percent solids, pH, and nutrients (ammonia-nitrogen, Total Kjeldahl Nitrogen [TKN], total phosphorus) are exempted from the requirement that they be performed by a registered/certified laboratory as outlined in NR 219.07. Samples for metals and other non-routine analyses are composited into a polyethylene container provided by the commercial lab, refrigerated, and transported to the commercial lab the following day. The commercial laboratory provides sample bottles containing required preservative. Samples are stored in coolers within the refrigerator until they are delivered to the commercial laboratory. Sample delivery is done in person or by a shipping company. Typical analyses required include: arsenic, cadmium, chromium, copper, lead, mercury, nickel and zinc.

vi. All sludge (Solids Matrix) results must be reported on a dry weight basis. If data are received "as is" or calculated based on "wet weight", they can be converted to dry weight using the following formula:

$$\frac{\text{Analyte (dry weight in mg/kg)}}{(\%\ \text{solids} / 100)} = \text{Analyte (wet weight in mg/kg)}$$

For example: zinc = 2.5 mg/kg (dry), 80% solids

Therefore…

$$\frac{2.5\ \text{mg/kg (dry)}}{(80\% / 100)} = 3.1\ \text{mg/kg (wet)}$$

vii. On occasion, the DNR region may require the analysis of additional parameters from the list of Conventional Priority Pollutants. These analyses are also performed by a properly certified commercial laboratory which will, in many cases, supply sample bottles containing required preservatives and sampling guidance.

viii. Sample bottles for analyses performed at the wastewater facility are permanently labeled for their appropriate use. Any departure from standard sampling protocol is noted on appropriate bench sheets. Care is taken to ensure that samples are well mixed prior to aliquot withdrawal. All sample bottles are washed using a scrub brush with hot tap water and non-phosphate detergent after each use. Our laboratory has put an approximately one foot length of surgical grade tubing on the tapered laboratory faucet nozzle. This tubing is inserted into the laboratory container near the bottom so that the tap water rinse minimizes the soap bubbles. Bottles are rinsed with cold tap water until the suds come up to the top of the neck and spills out into the drain to insure that detergent residue is removed. Then the bottles are triple rinsed with distilled water. Bottles for phosphorus samples are washed with a non-phosphate detergent and rinsed with 1:10 hydrochloric acid. Bottles for fecal coliform are sterilized before use.

ix. Sample handling/preservation requirements for wastewaters are in Table 3. Sample handling and preservation methods required by state and federal laws are followed (see NR 219, Table F).

Table 3. Sampling Handling Guidelines

PARAMETER	SAMPLE TYPE	PRESERVATION	CONTAINER	@MAXIMUM HOLDING TIME	*ANALYTICAL METHOD
Biochemical Oxygen Demand	24-hr composite [flow proportional]	Cool, ≤ 6°C	Polyethylene	48 hours	5210 B
Total Suspended Solids	24-hr composite [flow proportional]	Cool, ≤ 6°C	Polyethylene	7 days	2540 D
Ammonia-Nitrogen	24-hr composite [flow proportional]	Cool, ≤ 6°C; H_2SO_4 to pH <2	Polyethylene	28 days	4500-NH_3 F
Total Phosphorus	24-hr composite [flow proportional]	Cool, ≤ 6°C; H_2SO_4 to pH <2	Polyethylene	28 days	4500-P B(5) & 4500-P E
pH	Grab	None	Polyethylene	Analyze immediately	4500-H^+ B
Dissolved Oxygen (DO)	Grab	None	Glass (reinforced with scotch tape for safety)	Analyze immediately	4500-O G
Fecal Coliform Bacteria Per our current WPDES permit	Grab	Cool, ≤ 10°C;	Polypropylene or other sterilizable material	6 hours	9222 D

NOTES: @ From time of completed sampling
*Standard Methods for the Examination of Water and Wastewater, American Public Health Association, 19th edition

6. MAJOR ANALYTICAL INSTRUMENTS AND SUPPORT EQUIPMENT

a. The Tree City Laboratory is outfitted with all with the equipment required to correctly perform the testing for which it is registered. All equipment is kept in working order by adherence to routine and preventative maintenance schedules. Non-routine maintenance is performed as needed and is usually associated with a corrective action (see section 9).

b. Laboratory Equipment

i. YSI 5100 Dissolved Oxygen Meter

1. Self-stirring Dissolved Oxygen probe YSI 5010

ii. Orion 720Aplus Advanced Ion Selective Meter

1. Orion 9512BNWP Ammonia Electrode
2. Orion 8256BN PerpHecT® ROSS® Combination pH Electrode

iii. Genesys 10 Spectrophotometer

c. Laboratory Support Equipment

i. NAPCO 8000DSE Autoclave (Slow Exhaust Model)– 30 liter Front Loading Compact Autoclave

ii. Barnstead – Fistreem III Glass Still Model A1015 still

iii. ISCO Model 103B Refrigerated Autosampler (x2)

iv. Hach model 205 BOD incubator

v. SO-LOW Model DHN4-24GD-S Laboratory Refrigerator

vi. Sartorius CP Analytical Balance Model EW-11218-04

7. PROCEDURES FOR CALIBRATION, VERIFICATION, AND MAINTENANCE OF MAJOR ANALYTICAL INSTRUMENTS SUPPORT EQUIPMENT

a. Sampler Cleaning

i. The sampler tubing is cleaned at least twice monthly using the following procedure and is documented in maintenance logbook:

1. Pump HOT tap water through the tubing and run the sampler for at least two (2) minutes.

2. Rinse the tubing by pumping a 20% hydrochloric acid or a 20% nitric acid solution for two minutes. This acid rinse may be reused up to four (4) times before it is discarded. Safety precautions are used in the handling and disposal of these acid solutions (wear safety gloves and glasses).

3. Re-rinse the tubing again: pump HOT tap water through it for at least two (2) minutes.

4. Rinse the tubing by pumping distilled water through the system for at least one (1) minute. After one minute, stop the pump and allow the water to stand in the hose for an additional minute. After this minute, continue pumping the water for one (1) final minute. The distilled rinse is NOT re-

circulated. To ensure the bottles are free of materials that may contribute to the BOD, a weak solution of household bleach (50 mL bleach per 2 L of deionized water) can be used as a final step in the cleaning procedure. *The final rinse, however, must be sufficient to eliminate any traces of the bleach, which may kill natural seed organisms, leading to low bias in BOD values.*

ii. At the time the tubing is cleansed the sampler itself is subjected to a general washing by cleaning the pump, sample carboy container(s), and any internal sampler parts which come into contact with the wastewater.

1. Vigorously scrub all parts with hot tap water and a non-Phosphorus detergent and a brush.

2. Triple rinse (i.e. rinse and drain, rinse and drain, rinse and drain) these items with tap water after washing. With each rinse, the surgical grade tubing on the laboratory sink faucet is inserted as far as possible into the 5 gallon plastic carboy so that the tap water is brought to the top of the container to allow the detergent residue to spill over the top of the container to insure removal of all the detergent.

3. Triple rinse using distilled water.

b. Equipment Maintenance

i. A file is kept for each piece of equipment in the lab. Each file contains the owner's manual, a preventative maintenance schedule, and records of all maintenance and repairs performed including the exact nature of the problem, the date of the repair, what was done, who did it, and the cost. To determine if an instrument malfunction affected analysis results, the dates of breakdown and subsequent repair are considered particularly important. The analytical balance is serviced at least annually. The DO and ammonia probe membranes are replaced every two to four weeks or more frequently if readings become erratic.

c. Labware Cleaning

i. After each use, glassware is washed with non-phosphate detergent, rinsed with tap water, triple rinsed with distilled water, allowed to dry, and stored in a cabinet. The appropriate glassware cleaning procedures depend on the analysis to be performed. Glassware for Phosphorus testing is washed with a non-phosphate detergent, rinsed with tap water by allowing the detergent suds to spill over the top of the container to insure detergent removal, acid-washed after each use with a 10 % hydrochloric acid solution, triple rinsed with distilled water, and then filled with distilled water. Glassware is stored full of distilled water until the test is run. When the total phosphorus test is run, the distilled water is dumped out and the glassware is rinsed with a small amount of distilled water; just enough to coat the inside surface. This distilled water is then discarded. The glassware used for phosphorus is stored segregated from other lab glassware. Even though the phosphorus glassware is segregated, the same piece of glassware is not used for the blank, influent, and effluent sample each time. Any markings on the phosphorus glassware are removed after testing with water or acetone. BOD bottles are always stored dry. The Teflon® siphon tube used for BOD analyses is cleaned monthly with a bleach solution (25 ml bleach / L water) and rinsed thoroughly. All reagent water carboys are cleaned monthly with dilute hydrochloric acid.

d. Instrument Calibration

i. The pH meter, DO meter, and ammonia selective electrode are calibrated each day they are used. If these instruments are used over the course of a day, calibration checks are repeated every 2 hours. The temperatures of the BOD incubator and sample storage refrigerator are measured using non-mercury liquid in glass thermometers with their bulbs immersed in water. On each analysis day that the incubator or storage refrigerator is used, their temperatures are recorded on log sheets. Both the temperature, correction factor, and adjusted temperature are documented on a central log for all thermometers. The temperatures of the solids drying oven, muffle furnace, and fecal coliform incubation bath are recorded on the bench sheets when they are used for analyses. If the temperature is outside of the required range, the thermostat is

adjusted and the adjustments are noted on the log sheet. The results of testing associated with out of specification temperatures are noted on the DMR.

ii. Thermometers used in the lab to measure the temperature of the BOD and fecal coliform incubators are calibrated annually against a thermometer traceable to an NIST (National Institute of Standards and Technology, formerly National Bureau of Standards, NBS). Once a year each NIST traceable thermometer is calibrated against a NIST certified thermometer (borrowed from a neighboring wastewater treatment lab or a Rural Water Circuit Rider). The calibrated thermometers are tagged with their correction factors, and the appropriate factors are applied when documenting any temperatures in the laboratory. If the liquid column in the thermometer splits, it will no longer provide an accurate measurement and is retired.

iii. Thermometers used to record the temperature of influent and effluent 24-hour composite samplers are factory certified traceable to NIST and are sealed in a clear glass bottle filled with ethylene glycol. Each thermometer has a unique serial number and a certificate of NIST traceability. These are replaced or calibrated annually. A record is kept with the serial number on the thermometer and its corresponding correction factor.

iv. The analytical balance is serviced and calibrated by an outside vendor at least annually. Additionally, the analytical balance is put through its internal calibration sequence daily. Balance calibration is verified at least monthly using two S-class weights (100 mg and 1g). The weights used and the mass values obtained are noted in a log book by the balance table. If the balance calibration is off by more than the tolerance provided by the weight manufacturer, the balance is serviced by a manufacturer's representative. The class-S weights are stored with foam rubber padding so that the weights do not hit each other or the side of the container. This is done to maximize the life of the class S weight. The class-S weights are sent out for calibration at least every 5 years.

v. Other specific maintenance and calibration requirements are described applicable SOPs.

8. PROCEDURES FOR EVALUATING QUALITY CONTROL SAMPLES

a. Quality Control Analyses

i. Routine analysis of blanks, second-source Initial Calibration Verification (ICV) standards, primary source Continuing Calibration Verification (CCV) standards, and Laboratory Control Samples (LCS) are performed according to the frequency shown in Table 3. When corrective action does not resolve an LCS failure a nonconformance (formerly called a QC exceedance) is noted on the DMR. At the Tree City wastewater laboratory, replicates and matrix spikes are sometimes used for initial demonstration of capability training purposes. Alternatively, matrix spikes may be run in conjunction with corrective action measures. Results from matrix spike analyses are treated in the manner specified in the following section. Results of the known standard (glucose/glutamic acid) for BOD must be 167.5 – 228.5 mg/L. Records of all of quality control analyses are kept on daily bench sheets and in a separate quality control log book.

1. Method Blanks

a. Method blank (MB) means a sample of clean matrix (i.e., reagent water) that does not contain the analytes of interest. It is processed with and under same conditions as the associated samples in a preparation batch. Method blanks must be processed at least one per preparation batch. If the method does not require a preparation step, a blank different from the calibration blank is still required once per batch. Whenever a method blank contains analytes of interest above the detection limit of an analysis, the laboratory evaluates the nature of the interference and its effect on each sample in a

preparation batch. A sample in a batch associated with a method blank that fails criteria is reanalyzed or qualified on the DMR. NR 149 specifies that the method blank be below the highest of the LOD, 5% of the permit limit for that analyte, or 10% of the sample concentration.

2. Initial Calibration Verification (ICV) and Quality Control Standards (QCS) for total phosphorus

    a. A second source ICV standard is evaluated when total phosphorus analysis is performed. Two different manufacturer's lots are requested from the laboratory supply company whenever a new total phosphorus standard is purchased. The first lot of standard is used for the initial calibration, and the second lot of the standard is used to verify the initial calibration (the ICV). After the initial calibration curve for total phosphorus is generated, (once a year, or as needed), an ICV from a different manufacturer's lot is analyzed immediately to verify that the calibration is valid. Since the Tree City laboratory uses a 0.5 mg/L ICV, the ICV must fall within 0.45 -0.55 mg/L (± 10 % of the true value). By running a second source standard for phosphorus, the Tree City laboratory does not need to purchase or analyze Quality Control Standards (QCS) standards. QCS standards are also not required for BOD, TSS or ammonia.

3. Continuing Calibration Verification (CCV) for Total Phosphorus-

    a. A CCV is analyzed on days other than calibration days (Note: this assumes that batches over 20 samples are never analyzed by the Tree City laboratory). The concentration of the CCV must be within ± 10 % of the expected value. For example, if a 0.6 mg/L CCV is used, then the concentration must be between 0.54 – 0.66 mg/L for the CCV to be acceptable. If the CCV does not meet acceptance criteria, another is CCV is measured. If the results of the second CCV do not pass, then the Tree City laboratory takes corrective action, as required by NR 149.44 (7). After corrective action, Tree City is required to pass two CCVs in a row. If these consecutive CCVs do not pass, a new calibration curve is measured and the analysis of all samples is repeated. Alternatively, Tree City laboratory may report sample results from a run including a failed CCV, but the results are qualified appropriately on the DMR. (Note: CCVs are not needed for ammonia because the instrument is calibrated on each analysis day, assuming less than 20 samples are analyzed in a single analytical batch).

4. Laboratory Control Samples (LCS) …*formerly known as "Known Standards"*

    b. These standards are prepared by or acquired by the lab with a known concentration of the contained analyte. They are used to verify the accuracy of the system. Control charts generated from the previous quarter's LCS data are used by the laboratory to evaluate the acceptability of LCS results.

5. Replicates and Matrix Spikes (MS)

    a. Normally, the Tree City Wastewater Laboratory analyzes LCS samples rather than replicates or matrix spikes.

b. When the Tree City WWTP laboratory obtains ammonia and phosphorus compliance sample concentrations that are significantly different what is expected, the laboratory chooses to optionally analyze replicates and MS as soon as possible. Tree City will analyze an MS and replicate on the same analysis day if sample volume is sufficient. When optional replicates are analyzed, they are compared against in-house precision limits. When optional MS are analyzed, these are compared against the LCS limits. If the optional replicates and MS pass their respective control limits, then the laboratory is reassured that the sample results are valid and corrective action is not needed. When the laboratory chooses to analyze replicates and MS it follow up with corrective action if the replicates and matrix spikes exceed acceptance criteria.

c. If the optional replicate fails its precision limit, then a replicate LCS may be analyzed. Replicate LCS are evaluated against a relative percent difference (RPD) criteria.

6. Proficiency Testing (PT) Samples … *formerly known as Reference Samples*

a. A PT sample is a standard, obtained from approved external sources, whose concentration is unknown to the laboratory. For many tests, at least one set of PT samples must be analyzed every year to renew the lab's registration. At least one set of acceptable results must be obtained. Follow-up PT samples are analyzed if the provider acceptance limits are exceeded.

b. This laboratory chooses to use the State Laboratory of Hygiene PT sample subscription service. The State Lab of Hygiene ships PT samples several times a year. For each study, the laboratory analyzes and reports results by a deadline. For the State Lab of Hygiene PT samples, the laboratory receives a final report with the true values and acceptable ranges 30 days after the results deadline. If all the results are acceptable, the laboratory does not need to do anything else since results are electronically loaded into the lab certification computer system. If one or more failures occur, then the lab automatically receives the next round of PT samples from the State Lab of Hygiene. The Tree City facility obtains annual PT samples from the State Laboratory of Hygiene for ammonia, phosphorus, TSS and BOD.

Table 4. Minimum QC Requirements by Method.

Parameter	Method	Instrument Calibration	Method Blank	Initial Calibration Verification (ICV) Second Source	Continuing Calibration Verification (CCV) Primary Source	Laboratory Control Sample (LCS)	Replicate (Optional for all)	Optional Matrix Spike (MS)	Optional Matrix Spike Duplicate (MSD)[2]	Quality Control Sample (QCS) (formerly called blind standards)
BOD_5	SM 5210B 19th edition	Daily	At least one per analytical batch	NR	NR	GGA[1]	NR	NR	NR	NR
NH3 (as N)	SM 4500-NH_3, 19th edition	Daily	At least one per analytical batch	NR	After 20 samples	One per every analytical batch	NR	NR	NR	NR
TSS	2540 D, 19th edition	See footnote 9	NR	NR	NR	NR	NR	NR	NR	NR
TP	4500 P – B, 5 & E, 19th edition	At least yearly See footnote 4	At least one in every analytical batch	Required only in batch when full curve generated[8]	See footnote 5	One digested per every analytical batch	NR	NR[6]	NR	NR[7]

NR = Not Required

1. If less than 20 samples[3] analyzed in a week then 1 GGA per week. If more than or equal to 20 samples analyzed in a week then one GGA per 20 samples.

2. Sample replicates may be analyzed in place of matrix spike duplicates when there is a high probability that a replicate pair will contain the analytes of interest at or above the limit of quantitation of an analysis

3. Process, or other non-regulatory samples, which happen to be analyzed in the same analytical batch as regulatory samples, count towards the at least 20 samples requirement.

4. Full calibration curve using a minimum of three standards shall be run a minimum of every year. A new standard curve shall be generated sooner if a) the lot of the primary standard (i. e., parent pedigree standard) used to produce the standards for the calibration curve can no longer be used to make fresh CCVs, b) non-routine maintenance is performed on spectrophotometer, c) the check standard analyzed with each analytical batch continues to fall outside of 90-110% recovery after it has been shown to be properly digested and prepared.

5. If initial calibration day then CCV required after 20 samples[3]. If not a calibration day then CCV must be analyzed before samples are analyzed and after every 20 samples.

6. Matrix spikes may be processed for phosphorous, at the frequency of at least one sample per preparation batch, in place of laboratory control samples, if the acceptance criteria for corresponding laboratory control samples are used to evaluate the matrix spikes and the laboratory takes the corrective action required when matrix spikes fail established laboratory control sample acceptance criteria.

7. Laboratories that do not use second source standards to verify the accuracy of initial calibrations shall analyze quality control standards (formerly known as blind standards) as defined in s. NR 149.03 (57), three times per year at evenly spaced intervals for all certified or registered analytes determined by tests amenable to fortification, and for which known quality control samples are commercially available.

8. ICV is not necessary if the laboratory chooses to run quality control standards. See footnote 7.

9. Balance should be calibrated at least annually by a balance service. Check accuracy of the balance at least monthly using NIST traceable weights.

Table 5 - Quality Control Limits

	ICV, CCV, and LCS	REPLICATES[2]		
TEST	**Control Limit**	**<R>**	**Warning Limit**	**Control Limit**
BOD	198 ± 30.5	0.45	0.45	1.1
TSS	NA	0.6	0.6	1.5
Ammonia-Nitrogen	± 10%	0.05	0.05	0.13
Total Phosphorus	± 10%	0.07	0.07	0.18
pH	NA	0.1	0.3	0.4[1]

1 For those laboratories whose WPDES permit requires grab effluent pH monitoring, pH registration is only optional. Therefore, all pH precision limits are optional for all laboratories.

2 If optional replicates are analyzed.

The control limits listed in this table represent limits which can be achieved with the proper training and quality control program. Each lab must set their own limits based on the results of their QC tests. If the limits calculated using actual monitoring data are significantly different than those described in this table, an analytical problem should be suspected. The appropriate corrective action measures should be taken to determine the nature of the problem and make any necessary modifications to procedures to correct the problem. If control limits cannot be improved, refer to DNR district office for further guidance.

9. PROCEDURES FOR INITIATING, FOLLOWING UP ON AND DOCUMENTING CORRECTIVE ACTION ADDRESSING QUALITY ASSURANCE AND QUALITY CONTOL FAILURES, DISCREPANCIES OR NONCONFORMANCE

a. Corrective action is initiated when any situations become apparent which may affect data quality (i.e., consistent QC parameter failure, or failure of a PT sample). When it has been determined that a corrective action is needed the analyst documents the cause in the corrective action log (see example in table 6).

Table 6. Example format of corrective action log.

Describe the problem. Reference data and specific analytical runs. Note if problem reported on DMR	
Describe corrective action initiated	
Date and Initials	
Describe quality of data after corrective action implemented. Reference data and specific analytical runs	
Has the situation improved to acceptable level?	
Will further corrective action be needed?	
Date and Initials	

b. The action taken in hopes of fixing the problem are documented in the log

c. Data affected over time by the corrective action are referenced in the log. The situation is monitored for improvement and notes are made in the corrective action logbook. If the situation does not improve as expected, a new corrective action is undertaken and documented in the same manner as the initial attempt. This cycle continues until the situation has reached a state of acceptability. The process is graphically depicted in figure 1.

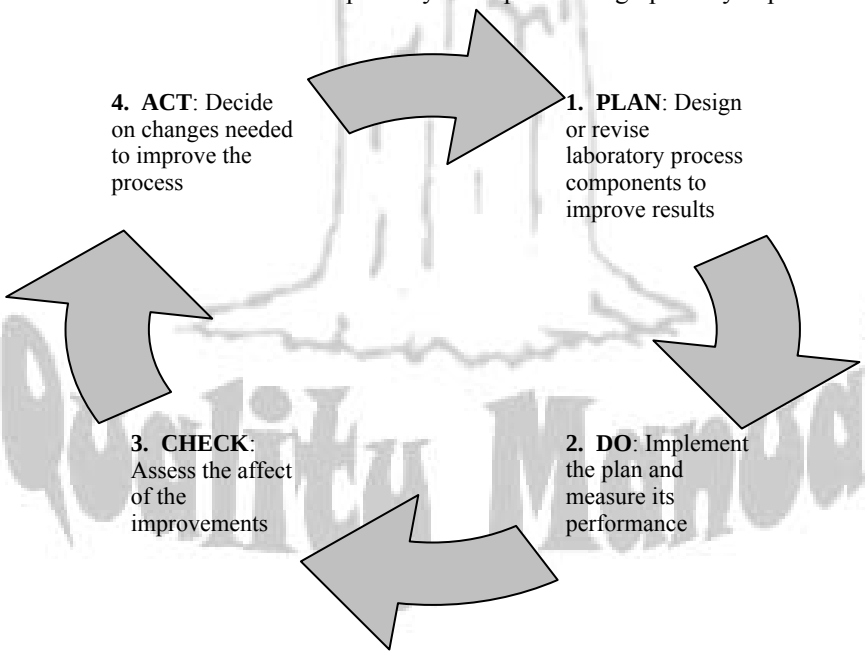


Figure 1. Graphical depiction of corrective action process.

d. Operators report those analytical results that are associated with any analytical run in which one or more of the quality control samples failed to meet acceptance criteria. These data are flagged on the DMR reports by placing a capital " Y " in the "QC Exceedance" box for any column that has been referred to in the "Laboratory Quality Control Comments" box. The date or dates of the analysis which had a quality control exceedence are also documented in the "Laboratory Quality Control Comments" section of the electronic DMR (eDMR). Comments include a narrative that describe which date or dates of the analyses are affected, and specific details regarding the reason for qualification of the data. The operator must also decide whether or not to include the analytical results when calculating weekly or monthly average values. If the decision is made to exclude the values in question from calculating weekly/monthly averages, an explanation for the exclusion(s) also is to be provided.

10. PROCEDURES FOR REVIEWING ANALYTICAL DATA AND REPORTING ANALYTICAL RESULTS

a. A number of reports are required to be filed to document compliance with the requirements of the WPDES permit. In each case, the data which must be included in the specific report is subject to appropriate review to ensure the accuracy of the data and compliance with effluent limitations (Table 5). Generally, when analytical results are completed by a Lab Technician, the data is reviewed by another individual familiar with the analysis, such as another Lab Technician, or the superintendent who as a supervisor of the laboratory operations. Once incorporated into the report, the superintendent (or director of the facility) reviews the compiled data against permit requirements and provide any necessary qualifications to the data, such as results that exceed permit limits or results for which the associated QC sample(s) failed to meet control limits. Monitoring reports are signed by the Village President (or principal executive officer, a ranking elected official, or other duly authorized representative).

b. All DMR reports are electronically submitted to the Department.