\_\_\_\_\_\_\_\_\_\_WWTP Laboratory Quality Manual

\*\*Update the manual to reflect your procedures (pay extra attention to bolded/red items)

\*Make sure to include your current practices, revise it if they change and inform staff they must follow any requirements

Revision # \_\_\_\_, Dated: \_\_\_\_\_\_\_\_

**Lab Organization and Responsibility**

1. The laboratory at the \_\_\_\_\_\_\_\_WWTP performs analyses necessary for both compliance with the requirements of the plant’s WPDES permit and process control. Quality Assurance (QA) is critical in producing sound, defensible data. The manual is intended to outline QA performed and to fulfill the requirements found in NR 149.37.
2. The lab personnel consist of a **laboratory director** who performs procedures on a daily basis **and two operators to fill in during vacancies and weekends on a rotating schedule**.  **In addition, the Wastewater Superintendent directly oversees day to day operations and personnel.**
3. Operators fulfill the I.D.C. requirements by reading the S.O.P. (Standard Operating Procedure) for each test. The operator must also pass the test-specific requirements such as blanks and standards as stated in the MWWTP LAB I.D.C manual. See the applicable SOPs for the required QC limits.

**Procedures for recordkeeping, retention, control and maintenance of documents**

1. All records of equipment calibration and maintenance, QC tests, sampling and reagent preparation are kept for at least three years (five years for sludge tests) at the plant **in a steel file cabinet on site.**
2. All raw data is kept, and when corrections are made on the bench sheet, white out/scribbling out are not allowed, instead a single line is drawn through the error, and the correct value written next to the error. It is recommended the change is initialed and dated. All observations are recorded in ink.
3. Records are stored in a secure manner to ensure their permanence and are only available to authorized personnel. Records are kept that pertain to all compliance samples, these include information but may not be limited to: purchased and prepared standards and reagents, sample handling, calibration, verification and equipment maintenance, sample analysis results, support equipment verifications, corrective action records, PT records, IDC and personnel training records and the laboratory accreditation certificates. Records and documents must be able to be retrieved to demonstrate compliance with NR149.
4. **If the records are retained only electronically (not printed out) the procedure used to maintain these records is….**
5. **After the workday the operator and the front gate is locked via key code entry by the operators or Superintendent. Only authorized personnel are granted entry to the facility after working hours.**

**Analytical Records**

1. The laboratory maintains all records containing raw data and calculations which are needed to reconstruct all results reported on the DMR for which the lab is accredited.
2. The lab has developed and adapted bench sheets for all routine analysis and documentation. Where possible, Wisconsin DNR suggestions are implemented and/or adapted to fit. The lab documents the following: sample ID, analysis time, preservation status, analyst, and analytical procedure, chemical used and data.
* Sample ID: Samples are identified (influent, effluent, etc.) and collection date.
* Analysis date and time: Grab samples are identified by date/time collected. Composite samples have both date/time sampler was started and date/time sample was collected.
* Analyst: The bench sheets indicate who is performing the test and the test being run.
* Analytical procedure: All tests are run according to the S.O.P. in use.
* Chemicals used: All standards and reagents used are recorded in the logbook to provide for direct traceability with the analytical records.
* Data: Raw data generated for samples and standards are recorded.

**Traceability for Reagents and Standards**

1. Reagents and standards are of analytical grade and currently purchased from **NCL labs or HACH Chemical Company (or equivalent)**. Container labels must include the expiration date, chemical name and concentration. This applies unless there is no applicable expiration date.
2. The laboratory needs to document the following (not on the label): Lot#, manufacturer, chemical name, concentration, expiration date (if applicable). Certificates of Analysis must be maintained.
3. When standards and reagents are prepared in the laboratory, the preparation details must be documented. The records must be linked to the stock chemicals used. Include also the preparer, preparation date and expiration date (if applicable).
4. The reagent name is logged on the appropriate inventory sheet the day it is received. Lot number, date opened, expiration date, and date discarded are recorded.  **Reagent water is produced within the lab using the Thermo Scientific and Barnstead ACS MegaPure System.**

**Procedure for handling samples**

1. **Sample carboys and all sample containers are washed daily** with hot tap water and non-phosphate detergent. Sample containers are then placed in the lab dishwasher and ran through on a sterilization setting also while using commercial grade non-phosphate detergent. Monthly the containers are rinsed with 10% household bleach (not the bleach that has additives). Follow the cleaning by hot tap water, rinse until all suds disappear, then rinse well with DI water. Samplers themselves are subjected to general washing by cleaning the sample carboy container(s) and any internal sampler parts which come into contact with the wastewater. SOP is as follows: 1. vigorously scrub all parts with water and a non- phosphorus detergent with a brush. 2. Triple rinse 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. Triple rinse using distilled water*. Optional: A one-time QC check may be performed for BOD, TSS, Phosphorus and Ammonia Nitrogen on carboys and sample containers. If results indicate contamination (not <2.0 mg/L for BOD and TSS or below LOD requirements for NH3 and TP) the source of the contamination must be addressed.*
2. All samples are identified by collection site and date. Flow composite samples are collected for both influent and effluent from the samplers located in the **headworks building and near the clarifier building by the effluent channel outfall respectively.** During collection they are refrigerated at ≤6ºC but not frozen.
3. Samples for phosphorus and ammonia are preserved with sulfuric acid to a pH of <2 immediately after collection (~15minutes) and refrigerated at ≤6ºC but not frozen if not analyzed immediately. Sampling frequency, sample type, parameters, and location are listed in the **WPDES permit binder**. Grab and composite samples for the day’s testing are normally collected between 7:30 a.m. and 8:30 a.m. Sample dates, times, and test times are recorded on the bench sheet. *Optional:* Plant sample composite carboys are tested for cleanliness at least one time – retesting is needed if the cleaning procedure or frequency changes.
4. **Samples for sludge analysis** are collected in containers supplied by the commercial lab contracted to do the testing. Any needed preservatives are already added to the plastic bottles. Samples are refrigerated at ≤6ºC (but not frozen) immediately after collection. Samples are to be placed on ice when collected and then transported to the contract lab by WWTP personnel or commercial carrier on ice (ice cubes or crushed ice) as soon as possible after collection. Samples are not allowed to exceed the NR219 holding times before sending them to the contract lab. All samples are submitted to the commercial laboratory with an appropriate chain-of-custody or sample form.

**Table 1. 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 | **SM 5210B-2011** |
| Total Suspended Solids | 24-hr composite [flow proportional] | Cool, < 6°C | Polyethylene  | 7 days | **SM 2450D-2011** |
| Ammonia-Nitrogen | 24-hr composite [flow proportional] | Cool, < 6°C; H2SO4 to pH <2  | Polyethylene  | 28 days | **EPA 350.1** |
| Total Phosphorus | 24-hr composite [flow proportional] | Cool, < 6°C; H2SO4 to pH <2 | Polyethylene  | 28 days | **SM 4500P E-2011 (preparation by B)** |
| pH (not accredited) | Grab | n/a  | Polyethylene | Immediately(same day) | **SM 4500pH 2011** |
| Subcontracted tests |  | Cool, < 6°C if cooling is required at time of collection | Polyethylene | Per NR219 | Per contract laboratory, compliance samples must be analyzed per NR219 approved methods |

NOTES: @ From time of completed sampling

 **\***Standard Methods for the Examination of Water and Wastewater, American Public Health Association, **22ndedition**

**Major analytical instruments and support equipment**

The lab has all the equipment required to correctly perform the tests for which it is certified. All equipment is kept in working order by following routine and preventative maintenance suggested by the manufacturer. Non-routine maintenance is done when corrective action warrants it. Many resources are used when O&M manual directions are insufficient to resolve the issue. Vendors, manufacturing representatives and other operators are consulted to find a solution. All maintenance activity is documented in the lab equipment log.

1. **The lab instruments currently used includes**:
* LBOD101 luminescent dissolved oxygen sensor with HQ440d Multi benchtop meter
* Orion VersaStar advanced electrochemistry meter and Ross SureFlow pH probe
* HACH DR3900 spectrophotometer
1. **Support equipment currently includes:**
* Ohaus analytical balance.
* Precision Scientific oven.
* Precision incubator for BOD
* Thermo Scientific incubator for drinking water bacteria testing.
* Transferpette S mechanical pipettes (100-1000µl, 0.5-5.0 ml, 1-10 ml)
* Two composite sampler units. One American Sigma sampler for influent and one Hach sampler for effluent.
* Maytag Refrigerator.
* Desiccator filled with color indicating desiccant.
* Hach DRB 200 heating reactor
* NCLabs desktop incubator for coliform.
* IDEXX Quanti-Tray Sealer Model 2X

**Procedures for calibration, verification, and maintenance of major analytical equipment and support equipment**

1. The effluent sampler tubing is replaced on an as needed basis (e.g., when it becomes discolored). As with the effluent sampler, the influent sampler tubing is replaced on an as needed basis. Maintenance is recorded on daily lab equipment log in the remarks section.
2. A file folder is maintained on each piece of equipment in the lab. The file contains O&M manual, vendor information and a record of maintenance done outside the normal routine.
3. The analytical balance is checked monthly when in use with at least one ASTM Class I or 2 weight and results are recorded and kept in the near the balance. The balance may not be used if the monthly check is outside acceptable tolerance limits listed on the form.
4. Weights used for balance verifications are sent out for verification or replaced every 5 years. If weights are not equivalent to ASTM Class 1 or 2 they are replaced.
5. The balance may be serviced once per year by an outside company. The results of that check are documented on a tag in the balance. The report is also maintained on file in the laboratory.
6. Barometric readings are checked at least annually using an official source (currently we use **www.weatherunderground.com**). Any suggested changes as calculated are entered into the meter. The procedure is documented in the BOD S.O.P.
7. The pH electrode is replaced as needed, calibration buffers are purchased as needed. The pH meter/probe is calibrated with at least 2 buffers than bracket the samples (7 and 10 or 4 and 7 or all three). Read back of the 7 buffer must be within 6.9-7.1 and the slope requirements by the manufacturer must be met and documented. No pH measurements are made until the problem is corrected.
8. The desiccator door or lid is greased when desiccant is in need of changing (indicated by color change from blue to pink).
9. The refrigerators, incubator, samplers and oven temperatures are recorded daily (on working days) on the applicable log sheet.
10. Thermometers are calibrated annually against a NIST traceable thermometer or replaced accordingly to manufacturers calibration date. Calibration reports on each NIST traceable thermometer are kept in a file in the lab. Correction factors are applied to thermometers if they deviate from the NIST traceable thermometer by more than the allowed tolerance. The date, correction factor (if needed) and analyst are recorded on a tag and attached the each thermometer. The thermometer calibration records are documented in the calibration log.
11. Mechanical pipettors need to be verified quarterly when in use. See the pipet verification log for criteria.
12. The calibration for the spectrometer and DO meter are in the applicable SOPs.

**Procedures for evaluating quality control samples**

1. Routine analysis of blanks and standards are performed and documented on bench sheets. If any of the QC fails, corrective action must be taken. When a corrective action does not resolve the QC failure, a qualifier note is made on the DMR in the Lab QC Comment field. Corrective actions to QC failure are documented on a separate log sheet. Outcomes of the action are documented and its value to the resolution of the problem. All corrective action must include the following:
* What the problem was or what failed?
* What was done to attempt to resolve the problem?
* Did corrective action resolve the problem or was it necessary to qualify the test results on the DMR?
* How you knew if the problem was resolved? In other words, were you able to document the problem was resolved?
* The analyst’s initials and date(s).
1. A GGA test is run weekly for the BOD test since the laboratory **has no more than 20 compliance samples to analyze per week.** A value of 198 ± 30.5 mg/L must be achieved. Results are recorded on the bench sheet. If the GGA results are outside the acceptable range, corrective action must be taken and a new GGA sample analyzed as soon as possible. Sample results must be qualified on the DMR back to the last valid GGA result.
2. Method blanks (reagent water run through the entire analysis process) are run with the phosphorus, ammonia and BOD tests. Method blanks are required with each batch up to a maximum of 20 samples. They are run under the same conditions as the samples associated with the batch. If a blank fails, it is rechecked and if it still fails, the sample results are qualified on the DMR. NR 149 states that the method blank is below the highest of the following:
* LOD,
* 5% of the permit limit for that analyte, or
* 10% of the sample concentration. BOD blanks must not deplete more than 0.24 mg/l.
1. Sample LODs must be able to meet regulatory limits. If the sample is diluted, the LODs and LOQs are adjusted by the sample’s dilution factor. The adjusted LODs and LOQs are reported.

The LOD is determined according to the approved procedure in 40 CFR Part 136 (revision 2). The LOQ is set to 10/3 the LOD, however the laboratory may also use the low calibration standard as the LOQ for TP and NH3.

BOD and TSS do not need an LOD study.

1. **The laboratory voluntarily purchases quarterly blind standards (QCS) from NCL or the State lab of Hygiene. We receive blinds for phosphorus, ammonia, BOD and TSS. The results are kept in a folder in the lab and are only known by the plant Superintendent. The results are only available to the analysts after all testing is done. In the case of unacceptable results, corrective action is taken to determine the source of the problem and a new blind standard would be ordered and analyzed.**
2. Annual proficiency samples are purchased from the **State Lab of Hygiene** and are analyzed and reported by the study deadline. In the case of unacceptable results, another set is ordered and analyzed. An acceptable result in each matrix the accredited is required for renewal of certification, and the correct method code must be reported. The PT is analyzed between January and August (analyzing a PT well within the allowed time frame is a good practice). The lab receives a report listing the range of acceptable values for each test. Refer to NR149 if the second PT does not pass.
3. Method codes for typical WW methods available in the 2021 updated code NR149 training documents available on the website.

**Procedures for initiating, following up on and documenting corrective action**

1. Corrective action is initiated when any situations become apparent which may affect data quality (QC failures, deviation from normal operating procedures, failure of a PT sample, etc.). When it has been determined that a corrective action is needed, the operator/analyst documents the cause in the corrective action log. The actions taken in hopes of fixing the problem are documented in the log.

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.

1. Analysts report those test 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 exceedance 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.

The Superintendent will ensure that the compiled data meets 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. Only authorized representatives are allowed to submit compliance reports to the Wisconsin DNR.

1. The Quality Manual is an overview of the Quality System. The information included in this document supplement the preparation and analytical SOPs. Refer to the SOPs for specific testing requirements accredited by WDNR, including training/IDCs.
2. All staff involved with laboratory activities play an important role to ensure data generated is usable. Staff are encouraged to let others know if there are problems. NR 149 includes the following information concerning ethics in the laboratory:

**NR 149.365 Laboratory ethics.** All the following practices are prohibited and may result in enforcement action under s.NR 149.10:

**(1)** Fabrication, falsification, or misrepresentation of data.

**(2)** Improper instrument clock setting, termed time traveling, or improper recording of date or time.

**(3)** Unwarranted manipulation of samples, software, peak integration, or analytical conditions.

**(4)** Concealing or failing to report a known improper or unethical behavior or action associated with sample analysis.

Quality Manual prepared by: