*Highlighted items are not requirements but indicate how some facilities manage their labs—the lab needs to edit this template for how they actually operate.*

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| **TITLE:** | **Laboratory Quality Manual** |
| **FACILITY:** | **Acme WWTP** |
| **DATE OF ISSUE:** | **4/10/20** |

1. **Procedures for Retention, Control, and Maintenance of Documents**
2. **General Information**

This Quality Manual (QM), standard operating procedures (SOPs), and all other laboratory related documents are available to all laboratory staff at all times. The QM and SOPs are reviewed each year and revised as needed. All revisions must be approved by the Laboratory Supervisor.

A copy of the approved *Standard Methods for the Examination of Water and Wastewater* is available in the lab. Wisconsin NR 149 and NR 219 are accessible on the internet. Any other necessary regulations, methods, or documents will be accessible to lab staff.

All records including sample collection, equipment calibration and maintenance, thermometer and weight certificates, quality control (QC) tests, standard and reagent preparation, bench sheets, logs, and any other laboratory records and documents are kept for at least three years (five years for sludge tests) at the laboratory. Records are saved and filed in labeled folders by year in the filing cabinets in the laboratory.

Records of laboratory personnel qualification, experience, and training must be kept. Initial demonstrations of capabilities (IDCs) must be retained as long as the employee works at the facility or for three years after the laboratory worker is no longer employed at the WWTP.

Records are stored in a secured manner and are only available to authorized personnel. The lab building is locked after the workday by the operator. The lab building is also locked when authorized personnel leave the facility. Records are stored in labeled banker boxes or file cabinets in a separate, locked storage room. Files are labeled by month and year. Electronic files are also backed up on the main server daily.

All handwritten data are recorded in ink. Pencils are not allowed in the laboratory. The use of correction fluid (white-out) is not allowed under any circumstances.

All raw data (original data) is kept. All data must be legible. The original data must not be obliterated—do not scribble out or overwrite. When corrections are made on bench sheets or other records, a single line is drawn through the error, the correct value is written next to the error, and the operator/analyst initials and dates those corrections.

All data is recorded at the time it is observed. Do not record dates, times, volumes, or other data ahead of time. Do not record data days or even hours later either.

1. **Administrative Records**

The WI DNR certificate of accreditation will be kept on the wall in the laboratory. The certificate is the property of the DNR.

1. **Reagent and Standard Records**

For reagent and standard records, see section, “Procedures for Traceability of Standards and Reagents.”

1. **Analytical and Technical Records**

The WWTP maintains all records containing raw data and calculations which are needed to reconstruct all results reported on the eDMR for which the lab is certified. The laboratory maintains all hard copy and electronic records.

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. All bench sheets have the analytical procedure printed on the form; all tests are run according to the method referenced in the SOP. The order of analysis must be run in the order on the bench sheet, which is typically top to bottom or left to right. See the attachments at the end for copies of the spreadsheets used for bench sheets. The lab documents at least the following: sample ID, analysis date/time, preservation status, analyst, chemicals used, and sample data.

* Sample ID: Samples are identified by collection date and location (influent, effluent, etc.).
* Analysis date/time: The analysis date and time is the time the test was started.
* Preservation status: For tests that require preservation, this is recorded on the preservation log or on the bench sheet.
* Analyst: The bench sheets indicate who is performing the test. For BOD and TSS, this could be two different analysts.
* Chemicals used: All chemicals used are recorded on the bench sheet to provide for direct traceability between the analytical records and the chemical records.
* Sample Data: Raw data generated for samples and standards are recorded. Raw sample data includes all measurements such as weight, absorbance, millivolts, dissolved oxygen, etc.
1. **Sample Collection Records**

The laboratory will maintain all sample collection records including all sample receipt logs. The lab must have access to records to determine if the samples meet all method requirements such as hold time. Records must be maintained that have both date and time the sampler was started, date and time the sample was collected, temperature of the sampler, and initials of the operator. Grab samples are identified by the date collected. Composite samples are identified by the date that the majority of sample was collected.

1. **Procedures for Traceability of Standards and Reagents**

Purchased reagents and standards: Purchased chemicals are of analytical grade and currently purchased from Pike Labs or another lab approved vendor. Upon receipt, all purchased standards and reagents are entered in the Chemical Log binder. Chemical name, lot number, manufacturer or vendor, concentration, expiration date, date received, and analyst initials are recorded for each chemical on the Purchased Chemicals Log. Each chemical will be assigned an ID number which must be recorded on each bench sheet for the tests in which it is used. See the attachments at the end for the Purchased Chemicals Log. Any certificates that come with a reagent or standard will be kept in the Chemical Log binder in the lab. All containers of chemicals must be labeled with the chemical name, expiration date, and concentration.

Prepared reagents and standards: Prepared chemicals from purchased reagents and standards are referenced to the original standard or reagent and expiration date from the bulk or original purchased stock. Date of preparation, date of expiration, and the laboratory analyst that prepared the reagent or standard are recorded for each chemical on the Prepared Chemicals Log in the Chemical Log binder. A new ID number will be assigned to each chemical, and these IDs are recorded on the bench sheet in which it is used. See the attachments at the end for the Prepared Chemicals Log. These containers are marked with the chemical name, expiration date, concentration, and the assigned ID number. The procedure to prepare these chemicals is described in the SOP for the test. If for any reason the recipe in the SOP is not followed, record the volume of source (purchased chemical or parent) used and the final volume of the solution (child) on the Prepared Chemicals Log.

No standard or reagent can be used past the expiration date. An expiration date is not required when one is not provided by the supplier.

Reagent water is purchased locally from the hardware store. The lab only uses Bass Brand distilled water.

1. **Procedures for Handling Samples**
2. **Procedures for all samples**

Samples with insufficient volume to complete the tests, samples beyond holding times, samples that were not properly preserved, samples in inappropriate containers, or samples received that show evidence that they weren’t collected correctly need to be rejected or appropriately qualified in the eDMR. Refer to each test SOP for additional reasons that sample results will need to be qualified.

Store all samples separately from all standards, reagents, food, and other potentially contaminating sources. A tightly lidded plastic storage container can be used to segregate chemicals from samples.

1. **Procedures for Acme WWTP samples**

All samples are uniquely identified by collection site and date. All sample containers must be labeled with the collection site and date.

Current sample locations, parameters, sampling frequency, and sample types are listed in the WPDES permits, which can be found in the permit binder. Also refer to the test SOPs for additional details on storage, preservation, and hold time. The following table lists the tests needed for ACME WWTP as of 4/10/20.

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|  | Type | Chemical Preservation | Thermal Preservation and Storage | Maximum Holding Time |
| BOD | Composite | None | If not run within **2** hours of collection, store at ≤ 6°C but not frozen | 48 hours from time of collection |
| TSS | Composite | None | If not run within **15** minutes of collection, store at ≤ 6°C but not frozen | 7 days from time of collection |
| TP | Composite | H2SO4, pH < 2 | If not run within **15** minutes of collection, store at ≤ 6°C but not frozen | 28 days from time of collection |
| NH3-N | Composite | H2SO4, pH < 2 | If not run within **15** minutes of collection, store at ≤ 6°C but not frozen | 28 days from time of collection |
| Cloride | Composite | None | None | 28 days from time of collection |
| pH | Grab | None | None | Immediately (within 15 minutes) |
| Temperature | Grab | None | None | Immediately  |

Acme WWTP grab and composite samples for the day’s testing are normally collected between 7:00 a.m. and 8:00 a.m. Composite samples are collected for both influent and effluent from the samplers. During collection they are refrigerated at ≤6°C but not frozen. The operator fills in the sampler log sheet noting the date, time, any remarks or unusual conditions, and his/her initials. For composite samples, this information and the temperature in the refrigerated sampler compartment are recorded for both the start and end of sampling. See the attachments at the end for the Sampling Log.

All samples for ammonia and phosphorus are preserved with sulfuric acid to a pH of <2 immediately after collection and refrigerated at ≤6°C but not frozen. Because the samples for ammonia and phosphorus need to be at room temperature for analysis, they likely cannot be run within 15 minutes of collection, so they are preserved. Check the pH at least quarterly with pH strips and record this on the Preservation Log. See the attachments at the end for the Preservation Log.

Carboys for composite plant samples are appropriately labeled and have been tested for cleanliness at least one time – retesting can be done to check cleanliness procedures.

Samples for sludge analysis, metals, and WET tests 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 ≤6°C (but not frozen) immediately after collection or as instructed by the commercial lab. Samples are transported to the 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 NR 219 holding times. All samples are submitted to the commercial laboratory with an appropriate chain-of-custody form.

1. **Procedures for client samples (sample acceptance policy)**

Samples from other facilities are submitted in their own sample containers and when preservation is required, preserved immediately after collection by the client. Sample bottles can also be provided to the client with the correct acid for preservation, when preservation is required.

When the laboratory is not responsible for sample collection and transport, it must verify that the paperwork, preservatives, containers and holding times are correct as required by the method or reject the sample.

Check all of the following:

* Check the pH on each total phosphorus and ammonia bottle and record it on the Sample Receipt Log. See the attachments at the end for the Sample Receipt Log. All samples for ammonia and phosphorus are preserved with sulfuric acid to a pH of <2 immediately after collection and refrigerated at ≤6°C but not frozen.
	+ If the pH is not <2, contact the client to see if the samples should still be run or if a new sample will be collected and submitted. If the client still wants the sample to be run, document that on the Sample Receipt Log, and be sure to qualify the result. This can be done by adding a note with the sample results that indicates that the sample was not properly preserved. This will need to be included with the eDMR data entry.
* Check the temperature of all samples with a traceable thermometer as soon as the samples arrive at the laboratory or as soon as practicable.
	+ The cooler melt water, the sample temperature, or a cooler temperature blank can be used to measure the temperature.
	+ The acceptable temperature range is ≤6°C but not frozen.
	+ If samples are received at a temperature of >6°C, they can be accepted and reported without a qualifier if all of the following are met:
		- the samples arrived at ACME WWTP the same day that they were collected,
		- the samples were immediately placed on ice, and
		- the samples were received on ice (not “blue ice”).
	+ If the samples are received at a temperature of >6°C and the above criteria were not met, qualify the sample results. This can be done by adding a note with the sample results that indicates that the sample was not received at the received at the correct temperature. This will need to be included with the eDMR data entry.
	+ Record the temperature on the Sample Receipt Log.
* Check that the records are complete. Records must be kept for all of the following:
	+ The identity of the client
	+ The dates of sample collection and the time of sample collection
	+ A unique sample ID was given to each sample; Sample IDs are given a letter based on the first letter or two of the client followed by the collection date and location. For example, a sample from Bluegill WWTP’s effluent collected on 7/21/21 is given an ID of B 7-21-21 EFF.
	+ Temperature and pH results were recorded on receipt
	+ Any comments were recorded if the samples did not meet the sample acceptance policies or if there is any reason to believe the samples weren’t collected correctly

If there are any problems or questions about the samples, contact the client and record this on the Sample Receipt Log.

1. **Procedures for Calibration, Verification, and Maintenance of Equipment**
2. **Equipment Maintenance**

The WWTP lab has all the equipment required to correctly perform the tests for which it is certified. All analytical instruments and support equipment are kept in working order by following routine and preventative maintenance suggested by the manufacturer. Non-routine maintenance is done as needed. All maintenance is documented on the Maintenance Log. See the attachments at the end for a copy of the Maintenance Log.

A file is maintained on each piece of equipment in the lab. The file contains the user manual, vendor information, and all maintenance records done outside the normal routine.

1. **Calibration, Verification, and Maintenance of Support Equipment**

Procedures for calibrating, verifying, applying correction factors, evaluating accuracy, and addressing failed equipment is discussed below where applicable (refer to the lab’s SOPs for calibration, verification, and maintenance of the analytical instruments).

Balance and Weights: The analytical balance is checked monthly with at least one ASTM Class 1 or 2 (or better) weights (one in the mg range and one in the gram range) and results are recorded and kept in the binder near the balance. The balance may not be used if the monthly check is outside acceptable limits listed on the form; check the level indicator, clean the pan, do an auto-calibration, or refer to the balance user manual. The weights are stored in their original containers. Weights should only be handled with plastic forceps and never be touched with fingers. The weights are sent out for calibration at least every 5 years or new weights must be purchased every 5 years. Keep all weight certificates in the binder near the balance. If the weights do not meet Class 1 or 2, the weights must be replaced. See the attachments at the end for a copy of the Monthly Balance Verification Log.

Barometer: Barometric readings are checked at least each year, but monthly is preferred, using an official source such as the nearest airport or on NOAA.com. The barometric pressure from this website is corrected to sea level, so the barometer at the laboratory needs a correction factor based on its elevation of 1650 ft. If the barometer is off by more than 5 mm Hg, the verification will be re-checked, and the meter is adjusted as needed. Note: to convert inches of Hg to mm of Hg multiply inches by 25.4. Example: 29.2 in Hg X 25.4 = 742 mmHg. Document the original barometric pressure, the sea level pressure, the source used (website), the corrected barometric pressure, initials of analyst, and date. See the BOD SOP and spreadsheet for additional details and calculations.

Block Digester: Each time the block digester is used, record the temperature reading on the bench sheet. The temperature for total phosphorus analysis must be 148 – 152°C. Every year, the block also needs to be verified with a traceable thermometer. To do the verification, take a used Hach vial, dispose of the contents appropriately, clean the vial well, and fill it with glycerin, sand, or a type of oil that doesn’t boil at 150°C. Put the thermometer in the tube, and then put the tube in the block. Start heating up the block, and let the thermometer stabilize for several minutes after the temperature has reached 150°C. If the thermometer reading is not 148°C – 152°C, adjust the block by following the instructions in the user manual. Record the temperature on the Temperature Log. See the attachments at the end for a copy of the Temperature Log.

Desiccator: The desiccator lid is greased. Desiccant is changed as needed (indicated by color change from blue to pink).

Mechanical pipettes: Pipettes are verified every quarter. Use the pipette to measure a volume of water into a beaker on a balance, record the weight, and repeat this four times. If the pipette can be adjusted for different volumes, test the pipette at the lowest volume, middle, and highest volume. The results are entered into the Quarterly Pipette Verification spreadsheet. If the results don’t meet the criteria in the spreadsheet, refer to the user manual to recalibrate, send the pipette out for service, or replace the pipette. A failed pipette cannot be used for compliance samples.

pH Meter: The pH electrode is replaced annually or as needed, and the storage buffer is changed weekly. The pH meter is calibrated daily with a pH 7.0 buffer and a pH 10.0 buffer. The slope is recorded on the bench sheet. If the pH meter fails to calibrate properly or if the slope is outside the manufacturers recommended range (typically -54 – -60), the electrode is cleaned and/or replaced. No pH measurements are made until the problem is corrected.

Refrigerators, samplers, incubator, and ovens: The temperatures of the refrigerator, samplers, incubator, and oven are recorded on the Temperature Log each day they are used. If the temperatures are outside of the required limits on the log sheet, make adjustments and place an up or down arrow next to the daily temperature reading. If samples were affected, take corrective actions and qualify the sample results as needed. See the attachments at the end for a copy of the Temperature Log.

Thermometers: All thermometers are replaced when they expire. The expiration date is listed on the thermometer certificates. If no expiration date is available, then it is one year from the calibration date. Certificates are saved in the Thermometer file, filed by year. Alternatively, thermometers are calibrated annually against a new NIST traceable thermometer. This is done by putting both the NIST traceable thermometer and the lab thermometer being calibrated in a beaker with ice and water. This can also be done by putting the NIST traceable thermometer and the lab thermometer being calibrated in the same refrigerator or the same oven. Allow both the NIST thermometer and the lab thermometer being calibrated to stabilize. Correction factors are applied to thermometers if they deviate from the NIST traceable thermometer. The date, correction factor (if needed) and analyst are recorded on a tag and attached to each thermometer. If the difference in temperature is more than 3°C, the thermometer is replaced. The thermometer calibration records are documented on the Thermometer Verification Log. See the attachments at the end for a copy of the Thermometer Verification Log.

Sampler tubing: The effluent and influent sampler tubing is replaced on an as needed basis (e.g., when it becomes discolored). Maintenance is recorded on the Lab Maintenance Log.

Glassware cleaning: Glassware is cleaned immediately after running all tests. Currently a 1% HCI solution is used for cleaning phosphorus and ammonia glassware. BOD bottles and glass pipets are cleaned with a non-phosphate detergent and a mild bleach solution. The glassware is then rinsed with tap water, then distilled water, and then allowed to dry on the rack above the sink. Glassware is always rotated (there are no dedicated method blank glassware). Glassware for phosphorus testing is kept separate, but is still rotated for the blanks, QC, and samples.

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| **Summary of Support Equipment** |
| **Equipment** | **Calibration and/or Verification Frequency** | **Criteria** | **Corrective Actions If Criteria Not Met** |
| Weights | Every 5 years | Class 1 or 2 (or better) | Purchase a new weight |
| Barometer | Annually (monthly preferred)  | ±5 mmHg | Refer to BOD meter manual; see BOD SOP |
| Thermometers | Annually (either replaced or verified) | No criteria, but correction factors must be applied if comparing to traceable thermometer | If the thermometers vary by more than 3°C from the traceable thermometer, replace the thermometer |
| Pipettes (mechanical) | Quarterly | <2% Inaccuracy,<2% Difference, and<1 %CV | Recalibrate per the manual, send the pipette out for service, or replace |
| Balance | Monthly | ≤10 mg: ±10%11 – 999 mg: ±5%≥1000 mg: ±0.5% | Check the level indicator, clean the pan, or repeat auto-calibration  |
| Digestion Block | Annually | 148°C – 152°C | See maintenance or troubleshooting in manual |
| Refrigerators (sample and chemical storage) | Each day of use | ≤6°C but not frozen | Defrost periodically; adjust to bring back in range; flag results if samples were affected |
| BOD Incubator | Each day of use | 19°C – 21°C | Adjust to bring back in range; flag results if samples were affected |
| TSS Oven | Each day of use | 103°C – 105°C | Adjust to bring back in range; flag results if samples were affected; see TSS SOP |
| Autoclave | Each day of use | 98 – 137 kPa (14.2 – 19.9 psi) | See maintenance or troubleshooting in manual |
| pH Meter and Probe | Each day of use | Slope of -54 – -60 or per probe manual | See maintenance or troubleshooting in manual |
| Desiccator | As needed | Color indicating desiccant must be blue | Replace desiccant |
| Sampler Tubing | As needed | Looks clean and method blanks pass | Replace tubing |

1. **Procedures for Evaluating Quality Control Samples**

Routine analysis of method blanks, laboratory control samples (LCS), initial calibration verification (ICV), continuing calibration verification (CCV), and any other method required QC samples are performed and documented on bench sheets. Refer to each test’s SOP for which QC samples need to be performed, when and how often they are performed, what the acceptance criteria are, and what corrective actions should be taken if the QC fails. If any of the QC fail, corrective action must be taken. See section, “Procedures for Initiating, Following Up, and Documenting Corrective Action.”

Annual proficiency testing (PT) samples are purchased from Walleye Labs 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 WWTP is certified for is required for renewal of accreditation. PT samples must be run just like regular samples unless the preparation instructions with the PT sample specifically instruct otherwise. No one in the laboratory is allowed to share the results of the PT sample with any other lab. The method code reported to the PT provider must match the approved method being used by the laboratory, which is referenced in each SOP. Acceptable PT sample results need to be reported electronically to the department by the approved PT sample provider no sooner than January 1 and no later than August 31.

Method detection limits (MDL), also referred to as limits of detection (LOD), are calculated from data generated following the procedure given in the latest revision of 40 CFR Part 136, Appendix B. BOD and TSS use reporting limits instead of LODs—refer to the SOPs for determining these reporting limits. For total phosphorus and ammonia, two spiked blanks are run in separate batches every quarter and are run just like normal samples. Every year, these spiked blank results and routine method blank results are used to calculate the new LODs. The LOD needs to be below the permit limit. Anytime there is major maintenance performed on an instrument, the LOD needs to be redone. Refer to the Attachments at the end for additional information.

1. **Procedures for Initiating, Following Up, and Documenting Corrective Action**

Corrective action is initiated each time any situation become apparent which may affect data quality (QC failures, deviation from normal operating procedures, failure of a PT sample, etc.). Each SOP includes specific corrective actions that can be taken for some common issues associated with the test.

When it has been determined that a corrective action is needed, the operator/analyst documents the issue on the Corrective Action Log. See the attachments at the end for a copy of the log. All corrective action must include the following:

* Identify the problem: What was the problem or what failed?
* Determine the cause: What was the most probable cause of the problem? Some issues may not have a specific cause, but the analyst needs to attempt to find the root cause of the problem.
* Take corrective actions(s): What was done to attempt to resolve the problem?
* Verify that the action(s) worked: Did corrective action resolve the problem or was it necessary to qualify the test results on the eDMR?
* Monitor the effectiveness: How do you know if the problem is resolved? In other words, has the problem reoccurred?
* The analyst’s initials and date(s).

The corrective actions are monitored for improvement and notes are made in the 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 actually been resolved. If the problem can’t be resolved, the staff at the WI DNR LabCert Program should be contacted for technical advice.

When analysts report results with one or more QC sample failures, these data must be flagged on the eDMR reports. See the section, “Procedures for Reviewing Data and Reporting Results.”

1. **Lab Personnel**

Initial Demonstration of Capability (IDC): IDCs must be documented and maintained. See the attachments at the end for a copy of the IDC form. Operators fulfill the IDC requirements by doing all of the following:

* reading the SOP for each test,
* performing the test and passing the test-specific QC (see the SOPs for QC and limits), and
* passing a blind sample (either the annual PT sample or a QCS sample).

Ethics: All of the following practices are prohibited and may result in enforcement action:

* fabrication, falsification, or misrepresentation of data;
* improper recording of date or time;
* unwarranted manipulation of samples or analytical conditions;
* concealing or failing to report a known improper or unethical behavior or action associated with sample analysis.
1. **Procedures for Reviewing Data and Reporting Results**

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 limits. Generally, when analytical results are completed by an analyst, the data is reviewed by the lead operator or the superintendent before entering the data in the eDMR or other compliance report.

Only authorized representatives are allowed to submit compliance reports to the Wisconsin DNR.

The lead operator and/or 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. These data are flagged on the eDMR 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 QC exceedance are also documented in the “Laboratory Quality Control Comments” section of the 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.

Example qualifiers:

* The GGA analyzed on 5/4 failed low at 163 mg/L. The affected samples were from sample dates 5/4-5/5.
* The BOD blank analyzed on 9/8 failed at 0.29 mg/L. The affected sample was from the sample date 9/8.

Sample results less than the corresponding LOD or reporting limit are reported as “<” values. The LODs reported must be current.

Reporting Results to Clients: For each client, ACME WWTP will keep a signed agreement on file that indicates that not all of the required NR 149.47 elements needs to be reported to the client. (See the attachments at the end for an example client agreement.) The reports, either in hardcopy or by email, will contain the information needed by the client for the eDMR. This includes applicable LODs or RLs, and any qualifiers needed for sample results (failed QC samples, not meeting sample acceptance criteria, etc.)

Subcontracted Tests: The tests that are subcontracted (WET, metals, TKN, etc.) are entered by the subcontract lab into the eDMR. If the subcontracted lab does not enter the results in the eDMR, be sure to enter any qualifiers reported with the subcontracted results. Subcontracted samples must be to a lab that is WI DNR certified to NR 149 for the corresponding tests.

*The lab may choose to include any attachments—the following are provided as examples that may be helpful for the lab*

**LIST OF ATTACHMENTS**

**Lab Information**

* Common Lab Abbreviations and Acronyms
* Common Lab Definitions
* Lab Activities Reminder Table
* LOD Procedure

**Forms**

* Purchased Chemicals Log
* Prepared Chemicals Log
* Sampling Log
* Preservation Log
* Sample Receipt Log
* Lab Maintenance Log
* Monthly Balance Verification Log
* Thermometer Verification Log
* Temperature Log
* Corrective Action Log
* IDC Records Form
* Client Test Report Agreement

**Spreadsheets**

* Bench Sheets for BOD, TSS, TP, and NH3-N
* Monthly DO Meter Barometric Pressure Verification Log
* Quarterly Pipette Verification Log
* LOD Calculations

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| **Common Lab Abbreviations and Acronyms**  |
| ASTM | American Society for Testing and Materials |
| BOD | Biochemical Oxygen Demand |
| cBOD | Carbonaceous Biochemical Oxygen Demand |
| CCV | Continuing Calibration Verification |
| COC | Chain of Custody |
| DATCP | Department of Agriculture, Trade, and Consumer Protection |
| DI | Deionized |
| DNR | Department of Natural Resources |
| DO | Dissolved Oxygen |
| eDMR | electronic Discharge Monitoring Report |
| GGA | Glucose-Glutamic Acid |
| ICV | Initial Calibration Verification |
| ID | Identification |
| IDC | Initial Demonstration of Capability |
| ISE | Ion Selective Electrode |
| LCS | Laboratory Control Sample |
| LOD | Limit of Detection |
| LOQ | Limit of Quantitation  |
| MDL | Method Detection Limit |
| NH3 (or NH3-N) | Ammonia |
| NIST | National Institute of Standards and Technology |
| NOAA | National Oceanic and Atmospheric Administration |
| NR | (Department of) Natural Resources |
| PT | Proficiency Testing |
| QC | Quality Control |
| QCS | Quality Control Standard |
| QM | Quality Manual |
| SM | Standard Methods for the Examination of Water and Wastes |
| SOP | Standard Operating Procedure |
| TP | Total Phosphorus |
| TSS | Total Suspended Solids |
| WET | Whole Effluent Toxicity |
| WPDES | Wisconsin Pollutant Discharge Elimination System |
| WWTP | Wastewater Treatment Plant |

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| **Common Lab Definitions** |
| Acceptance Limits | Limits established that are used to determine if the laboratory has analyzed a quality control sample or proficiency testing sample successfully. |
| Accuracy | The closeness of a measured value to an accepted reference value or standard. |
| Analyst | The designated person who performs the hands-on testing and who is responsible for meeting the required laboratory practices. |
| Analyte | Chemical substance, physical property, or organism analyzed in a sample. |
| Batch | A set of samples prepared or analyzed together using the same process, personnel, and lots of reagents. |
| Calibration | The process used to establish an observed relationship between the response of an analytical instrument and a known amount of analyte, or the process used to determine, by measuring or comparison with a reference standard, the correct value of each scale reading in an instrument, meter, or measuring device. |
| Calibration Blank | An aliquot that consists of the same matrix as that used for the calibration standards, but without the analytes. In other words, it is processed using the same procedure as the calibration standards except that no stock standard was added. |
| Calibration Curve | The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. |
| Calibration Standard | Solutions used to calibrate the instrument response with respect to analyte concentration. |
| Chain of Custody | Unbroken trail of accountability that ensures the physical security of samples,data, and records. |
| Continuing Calibration Verification | A standard of known concentration of analyte used to ensure the calibration is still valid throughout the analysis. |
| Control Limits | See acceptance limits. |
| Corrective Action | Any measure taken to eliminate or prevent the recurrence of the causes of problems (nonconformities, defects, or undesirable conditions). |
| Holding Time | The maximum time that samples may be held prior to analysis and still be considered valid. |
| Initial Calibration Verification | A standard of known concentration, prepared using second source standards, analyzed following the initial calibration and prior to measuring any samples to ensure the calibration is accurate. |
| Initial Demonstration of Capability | The process to determine if an analyst is qualified to perform laboratory testing. |
| Instrument Blank | A clean sample (e.g., distilled water) processed through the instrument steps of the measurement process; used to determine instrument contamination. |
| Interference | The combined or individual chemical components of a sample that may or may not cause a false positive measurement by an instrument. |
| Laboratory Control Sample (LCS) | A sample of a matrix without the analytes of interest spiked with a known amount of the analytes of interest. The purpose of an LCS is to determine whether the method process is in control and whether the laboratory can make accurate and precise measurements. |
| Limit of Detection (LOD) | The lowest concentration or amount of analyte that can be identified, measured, and reported with confidence that the concentration is not a false positive value. The DNR considers the LOD to be equivalent to the method detection limit. |
| Limit of Quantitation (LOQ) | The lowest concentration or amount of an analyte for which quantitative results can be obtained. |
| Method Blank | A clean matrix that is treated and processed exactly as a sample including exposure to all glassware, equipment, solvents, and reagents to measure contaminants in the measurement process. |
| Method Detection Limit (MDL) | The minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results. The MDL is generated according to the procedure specified in the latest revision of 40 CFR Part 136, Appendix B. |
| Precision | The degree to which a set of measurements obtained under similar conditions conform to themselves. Precision is usually expressed as the standard deviation, variance, or range, in either absolute or relative terms. |
| Preservation | The refrigeration of and/or reagents added at the time of sample collection to maintain the chemical and/or biological integrity of the sample. |
| Proficiency Testing (PT) | A study where a sample is obtained from an approved proficiency testing sample provider to evaluate the ability of a laboratory to produce an analytical test result meeting the definition of acceptable performance. The concentration of the analyte in the sample is unknown to the laboratory at the time of analysis. |
| Qualify | A written statement accompanying test results to identify anomalies or issues that were encountered in generating the results. |
| Quality Control | The overall system of technical activities designed to measure and control the quality of test results. |
| Raw Data | Any original information from a measurement recorded in any form that allows the reconstruction and evaluation of the activity. Raw data include absorbance, emission counts, abundance, and millivolts. Raw data may be stored in hard copy or electronically. |
| Reagent Water | Water which has been treated to remove any impurities that may affect the quality of an analysis. |
| Sample Matrix | Collective inherent chemical, biological, and physical components and characteristics of a sample. |
| Standard Operating Procedures (SOPs) | A written document which details the method of an operation or analysis and which is accepted as the method for performing certain routine or repetitive tasks. |

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| **Lab Activities Reminder Table** |
| One Time | IDC for each analyst and backup analyst for each test |
| Carboy cleaning blanks for certified tests; update if cleaning procedure changes |
| 5 years | Weights must be sent out for external verification or replaced (ASTM 1 or 2) |
| Yearly | Thermometers must be replaced one year after the calibration date if the vendor has not provided a longer expiration date OR thermometers must be verified against a traceable thermometer |
| Verify the temperature of the digestion block |
| PT sample for each test must be run prior to August each year |
| Barometer must be verified against an external source (monthly is best) |
| Perform LOD calculations and verifications |
| Check that all records are filed properly in labeled areas and folders |
| Review the SOPs and QM for corrections and that any changes needed are officially updated |
| Quarterly | Run two spiked blanks for the ongoing LODs (and could compile method blanks for the quarter too) |
| Verify mechanical pipettes |
| Check the pH of preserved samples for TP and NH3 (if there is a large industrial component to samples or if the plant processes have changed significantly, check pH more frequently) |
| Monthly | Balances must be checked with weights and the weights must meet tolerances |
| File bench sheets and eDMR records in the month/year folder and put in labeled file cabinet |
| Daily | Record temperatures of refrigerators, ovens, and incubators |
| As it occurs | Corrective actions are documented |
| Maintenance is documented |
| Reagents received and reagents prepared are entered into the log; when new reagents are opened or disposed of, add dates  |
| Each day the pH meter is used, calibrate the meter |
| Each day the DO meter is used, calibrate the meter |

**LOD (MDL) Study Instructions**

*See the spreadsheet for additional notes and instructions*

**Initial LOD**:  You will need to do a new LOD if you start doing a new method, you only run a certain test a couple of times per year, or if your ongoing LOD doesn’t pass (see next section).  To do the initial LOD:

1. Prepare 2 or 3 spiked blanks on at least 3 different days so that you have at least seven spiked blanks.

* 1. For example, you can prepare two 0.1 mg/L total phosphorus solutions on Monday and run the samples on Monday.  On Wednesday, you can prepare two 0.1 mg/L total phosphorus solutions and run then on Wednesday. On Friday you can prepare three 0.1 mg/L total phosphorus solutions and run then on Friday.  You don’t have to prepare and analyze them the same day, but you do still need at least 3 days of prep and 3 days of analysis.
	2. If you happen to have blank spikes at 0.1 mg/L, say from a low-level standard check, you can use that data, but you need to use all of the data.
	3. Enter this data in the spreadsheet. Use the tab labeled “Initial LOD.” Enter all dates, results, and the spike concentration in the section labeled, “Spiked Blanks.”
1. If any of the spiked blanks give a negative result, you need to start the initial LOD again but at a higher concentration.
2. The LOD**s** is calculated the same way as in the old procedure (the spreadsheet will do the calculations).
3. Compile the most recent method blanks you’ve run to calculate the LOD**b**.

* 1. If this is a new method or rarely used method and you don’t have at least 7 method blanks, you will need to analyze a method blank with each of the spiked blanks you ran in step 1.
	2. Enter this data in the spreadsheet in the section labeled, “Method Blanks.”
	3. Be sure to enter all “-” with any negative values that were measured.
	4. The spreadsheet will calculate the LOD**b** based on the average, standard deviation, and t-value.
1. The LOD will be the higher of the LOD**s** or LOD**b** (the spreadsheet will automatically calculate it).

**LOD (MDL) Study Instructions (Continued)**

**Ongoing LOD and Verification**:  Once you have all of your initial LOD studies done, you will need to start collecting data for the ongoing studies.  To do the ongoing LOD and verification:

1. Each quarter, prepare and run at least 2 spiked blanks using the same concentration as that used for initial LOD.  These must be run in different batches each quarter. By the end of twelve months, you will have 8 spiked blanks.

* 1. If a method is rarely run, you only need to run the 2 spiked blanks in quarters that you run actual samples (PT samples aren’t considered actual samples).
1. If more than 5% of the spiked blanks are negative, the initial LOD must be redone at a higher concentration (the spreadsheet will alert you).
2. Calculate the LOD**s** with all data from the last two years, but only data with the same spiking level.  This may include the 7 or 8 spiked blanks from the initial LOD if it was done within the last 2 years.
3. Enter this data on the “Ongoing LOD” tab of the spreadsheet. Not all of the lines in the Spiked Blanks section need to be filled.
4. Compile all method blanks.  Ideally, use all method blanks from the last 2 years.
	1. If more than 100 method blanks have been run in the last 2 years, you have the option to use either the 99th percentile calculation or the standard deviation calculation. The spreadsheet will show what the results would be from both options (if applicable).

* 1. You have the option to use only the last 6 months or the 50 last method blanks, whichever yields the greater number of method blanks.
		1. The spreadsheet has a spot to enter how frequently a test is run—this will help determine if you need to use the last 6 months, last 50, or last two years of method blanks.
1. The spreadsheet will calculate the ongoing LOD.
	1. The LOD will be the greater of the LOD**s** or LOD**b**.
	2. If the LOD is within 0.5 – 2 times the existing LOD and less than 3% of the method blanks are above the existing LOD, then the existing LOD may be left unchanged (the spreadsheet will say if the LOD can be left unchanged).
	3. If the LOD does not meet that criteria, then the LOD must be set to the new LOD (the spreadsheet will say if the LOD must be changed).



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| **Purchased Chemicals Log – Lab ID Tracking** |
| **Lab assigned ID #** | **Chemical Name** | **Date Received** | **Initials** | **Vendor** | **Lot #** | **Concentration [include units]** | **Date Expires** | **Date Discarded** |
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| **Prepared Chemicals Log - Lab ID Tracking** |
| **Prepared Chemical Lab Assigned ID #** | **Chemical Name** | **Prep Date** | **Prep****By** | **Source (Parent) Lab ID #** | **Source Volume or Weight [include units]** | **New (Child) Final Volume [include units]** | **New (Child) Conc. [include units]** | **Date Expires** | **Date Discarded** |
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| **Sampling Log for Plant:** |  |   |
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| **Month and Year:** |  |   |
|   |   |   |   |   |   |   |   |
|   | **Sample Date** | **Collect Date** |   |
| **Raw/FinalCollectionType** | **Date / Time Samplers Started** | **Sampler Temp (°C)** | **Person who Started Samplers** | **Date / Time Samplers Stopped** | **Sampler Temp (°C)** | **Person who Stopped Samplers** | **Comments** |
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| **FPC = Flow proportional composite** | **TPC = Time proportional composite** |

**Preservation Log**

 **Ammonia and Phosphorus Samples**

(At least Quarterly – pH needs to be <2)

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| Date | Initials | Influent pH | Effluent pH |  |
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**ACME LABORATORY SAMPLE RECEIPT LOG**

**Client: Bluegill Sewage Treatment Plant**

|  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Date | Initials | Sample ID | Sample collection date and time provided? | Bottles OK? | TP pH | NH3 pH | Temp(°C) | Comments |
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Reject or qualify data if any of the following:

• Not enough sample, • sample past hold time, • TP or NH3 not pH<2, • Temp >6°C and not on ice, • Damaged or wrong bottles

**Lab Maintenance Log**

Instructions: Complete this form and save it. Whenever maintenance is performed on any piece of laboratory equipment record that information below. If you need more than one line you can just continue on the next line. Include as much detail as you can. You do not have to limit yourself to one line per entry.

*Common lab equipment which require maintenance (there are others):*

|  |  |  |  |  |
| --- | --- | --- | --- | --- |
| pH meter / electrode | Incubator | DO meter barometer | Analytical Balance | Composite Sampler Tubing |
| TSS oven | DO meter / probe | NH3-N meter / electrode | Desiccators | Thermometers |

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **Date** | **What equipment was worked on?** | **What maintenance or corrective action did you perform on this equipment?** | **Did Fix work (Y/N)** | **Other Comments** | **Initials** |
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**Monthly Balance Verification Log**

**Lab: ­­­­­­­­­­­­­\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ YEAR: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_**

|  |  |  |
| --- | --- | --- |
| **Weight True Value (indicate g or mg)** | **Control Limits** **[indicate ± 0.5% for ≥ 1 g, indicate ± 5% for 20 – 900 mg, indicate ± 10% for ≤ 10 mg]** | **Acceptable Control Limit Range (indicate range – i.e. 190-210 mg for a 200 mg true value weight)** |
| 0.2000 g | ±5% | 0.1900 – 0.2100 g |
| 1.0000 g | ±0.5% | 0.9950 – 1.0050 g |

Use one weight in the gram (g) range and one weight in the milligram (mg) range.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **Month** | **Date** | **Initials** | **Weight True Value** **(g)**  | **Weight Measured Value** **(g)** | **Pass? (Y/N)** |
| Jan |  |  | 0.2000 g |  |  |
| 1.0000 g |  |  |
| Feb |  |  | 0.2000 g |  |  |
| 1.0000 g |  |  |
| Mar |  |  | 0.2000 g |  |  |
| 1.0000 g |  |  |
| Apr |  |  | 0.2000 g |  |  |
| 1.0000 g |  |  |
| May |  |  | 0.2000 g |  |  |
| 1.0000 g |  |  |
| June |  |  | 0.2000 g |  |  |
| 1.0000 g |  |  |
| July |  |  | 0.2000 g |  |  |
| 1.0000 g |  |  |
| Aug |  |  | 0.2000 g |  |  |
| 1.0000 g |  |  |
| Sept |  |  | 0.2000 g |  |  |
| 1.0000 g |  |  |
| Oct |  |  | 0.2000 g |  |  |
| 1.0000 g |  |  |
| Nov |  |  | 0.2000 g |  |  |
| 1.0000 g |  |  |
| Dec |  |  | 0.2000 g |  |  |
| 1.0000 g |  |  |

**This form is just one example of how to document monthly balance verification. There are many ways that balance verification can be documented. If you have any questions on this form contact the Laboratory Certification and Registration Program.**

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| **THERMOMETER VERIFICATION LOG** |
| **FREQUENCY: Annually** |
|  |  |  |  |  |  |  |  |  |
| **Certified or NIST Traceable Thermometer Serial #:** |  |  | **Expiration Date:** |   |  |
|  |  |  |  |  |  |  |  |  |
| **Date** | **Location of Lab Thermometer** | **Serial # of Lab Thermometer** | **Certified Thermometer****Temp Reading (°C)** | **Certified Thermometer Correction Factor\* (°C)** | **Certified Thermometer Corrected Reading (°C)** | **Lab Thermometer Reading (°C)** | **Correction Factor\*\* (°C)** | **Initials** |
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| **\* Refer to the thermometer certificate. If using a digital reference thermometer, there will be no correction factor for the reference thermometer.** |
| **\*\* Label each thermometer with the date of the calibration, initials of the analyst, and correction factor. The correction factor is to be applied to the lab thermometer reading when recording daily temperatures in the laboratory logbook.** |

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| **TEMPERATURE LOG** |
| **FREQUENCY: Each Use** |
|  |  |  |  |  |  |  |  |  |
| **Date** | **Initials** | **Sampler 1 (0°C - 6°C)** | **Sampler 2 (0°C - 6°C)** | **Sample Refrigerator (0°C - 6°C)** | **Standard and Reagent Refrigerator (0°C - 6°C)** | **BOD Incubator (19°C - 21°C)** | **TSS Oven (103°C - 105°C)** | **Total Phosphorus Digestion Block (150°C)** |
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| *Some temperatures may be documented on benchsheets.* |

**Corrective Action Log**

Instructions: Complete this form and save it. If you need more than one line you can just continue on the next line. Include as much detail as you can. You do not have to limit yourself to one line per entry. Use as much space as you need to explain the situation clearly.

*Example QC failures (there are others):*

|  |  |  |  |
| --- | --- | --- | --- |
| Method blank | LCS | Replicate (duplicate) | Matrix spike |
| QCS samples | Proficiency Testing samples | Calibration | Lab policy or procedure not followed |

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Date** | **What was the problem (or what failed)? Did the results fail high or low? What test?** | **What was done to try and fix the problem?** | **Did the fix work? (Y/N)** | **Qualify data (Y/N)** | **How do you know the fix worked?** | **Initials** |
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**Initial Demonstration of Capability Records**

|  |  |
| --- | --- |
| **Analyst** |  |
| **Test** | TSS | BOD Set Up | BOD Read Out | TP | NH3 |
| **Read the SOP** |  |  |  |  |  |
| **Performed the test and passed the QC** |  |  |  |  |  |
| **Passed a blind sample** |  |  |  |  |  |
| *For each test, enter the date the SOP was read, the test was performed and the QC passed, and the blind was passed.* |

|  |  |
| --- | --- |
| **Analyst** |  |
| **Test** | TSS | BOD Set Up | BOD Read Out | TP | NH3 |
| **Read the SOP** |  |  |  |  |  |
| **Performed the test and passed the QC** |  |  |  |  |  |
| **Passed a blind sample** |  |  |  |  |  |
| *For each test, enter the date the SOP was read, the test was performed and the QC passed, and the blind was passed.* |

|  |  |
| --- | --- |
| **Analyst** |  |
| **Test** | TSS | BOD Set Up | BOD Read Out | TP | NH3 |
| **Read the SOP** |  |  |  |  |  |
| **Performed the test and passed the QC** |  |  |  |  |  |
| **Passed a blind sample** |  |  |  |  |  |
| *For each test, enter the date the SOP was read, the test was performed and the QC passed, and the blind was passed.* |

**Test Report Agreement**

***NR 149. 47 (2)(b) When tests are performed for internal clients, or when a laboratory has a written agreement with a client, laboratory reports may be issued by the laboratory without all the content elements specified in this section. However, the laboratory shall retain and make available to the department, upon request, records that include the content elements specified in this section.***

Dear Client,

NR 149 requires that the test reports we submit to you include the following elements:

1. The name of the laboratory where tests were performed.
2. The laboratory’s accreditation identification number.
3. The sample identifying information provided by the client or collector.
4. Identification of the methods used for preparation and analysis.
5. The collection date of the samples.
6. Collection, preparation, and analysis times for test with holding times expressed in hours.
7. The dates of analysis, extraction, or digestion, when a holding time has been established for the preparation step.
8. When non-aqueous sample results are reported, the laboratory shall indicate whether the non-aqueous sample results were reported on a dry weight or wet weight basis.
9. The LOD and LOQ for tests which the department requires reporting to the LOD.
10. Except for HRGC/MS analysis, for sample results requiring adjustments, and indication of whether the LOD and LOQ have been adjusted accordingly.
11. The units of measurement.
12. The date of the test report.
13. Any qualifiers with reported results.
14. The identity of the subcontract laboratory, for each reported result generated by a subcontract laboratory.

As a general practice, this laboratory does not provide all of this required information with our test reports. Instead we provide a “test report,” with your sample results like the one included as an attachment with this letter. Based on our discussions in the past, I believe this is acceptable. If this is acceptable to you, I need you to indicate as such in writing (below), and send it back to me so that I have this on file for the department. Be assured that the information listed above, required by the department, is kept on file in the laboratory in case it is ever needed.

Sincerely,

My Laboratory

I do not need Laboratory \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ to provide my sample results on a test report with all of the information required under NR 149. The manner in which they currently provide sample results satisfies what I require at this time.

Signed: Date:

Client Name: Facility:













