



**STOUGHTON UTILITIES WASTEWATER TREATMENT
FACILITY LABORATORY QUALITY MANUAL***

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Prepared by:

**Phillip O. Linnerud
Wastewater Operator and Laboratory Technician
Wastewater System Division
Stoughton Utilities
700 Mandt Parkway
Stoughton, WI 53589**

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EXECUTIVE SUMMARY

Stoughton Utilities Wastewater Treatment Plant Laboratory Quality Manual (is an example of the minimum requirements) 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.

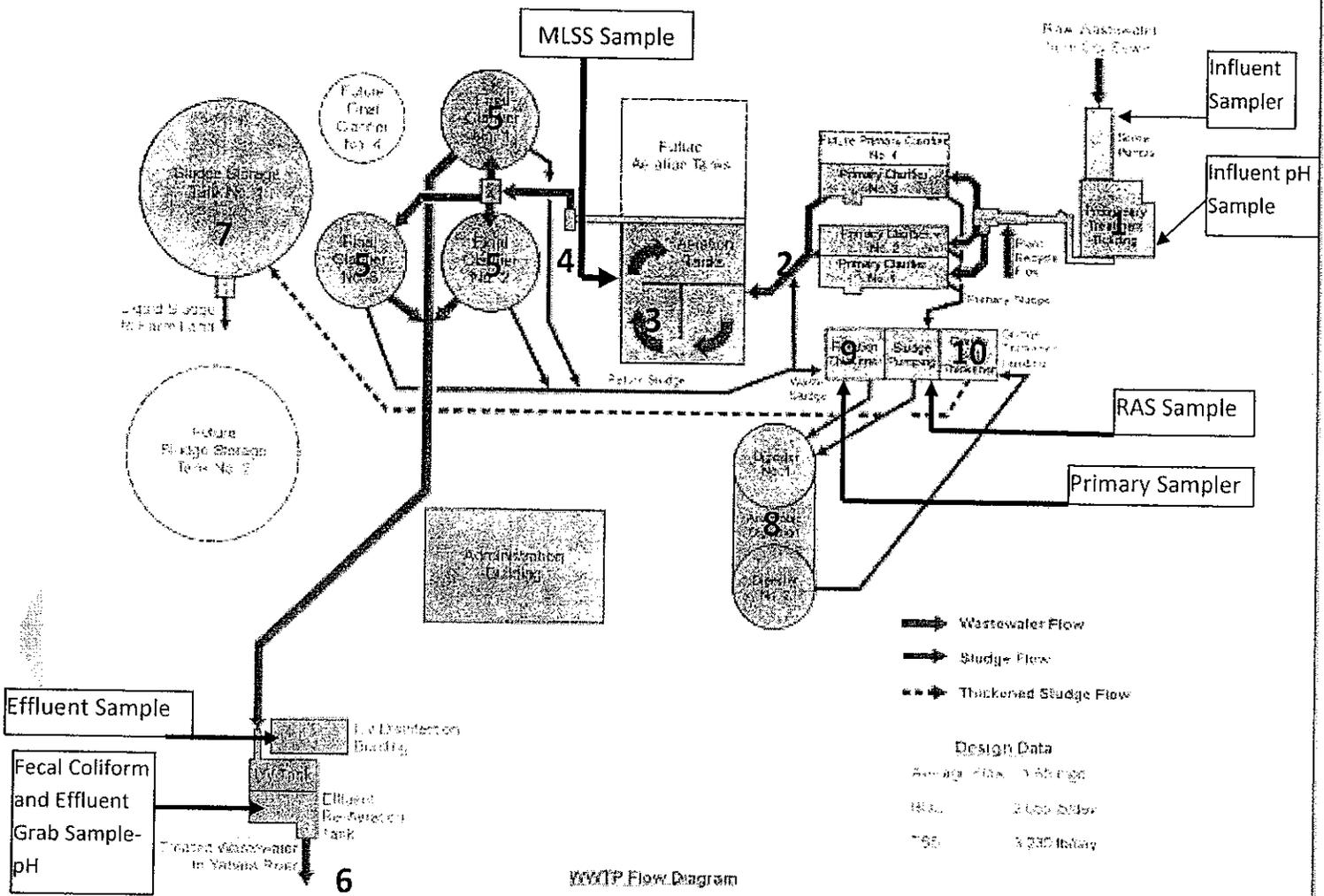
PREFACE

Presented here is a copy of the quality assurance manual for the laboratory at the Stoughton Wastewater Treatment Facility. This quality manual presented is required by NR 149.37 (1). They are valuable as a guide to maintaining analytical performance and assuring compliance with other requirements of NR 149.

This material is distributed as guidance for use by laboratory personnel regarding elements of a quality assurance program. The Stoughton City facility is a medium sized (about 1.5 mgd of combined domestic and industrial wastewater plant). Only the essential elements of a quality assurance document are included in this plan.

Please note that, where details are provided, they are specific to Stoughton Utilities Wastewater Division.

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.



Stoughton Wastewater Flow Schematic

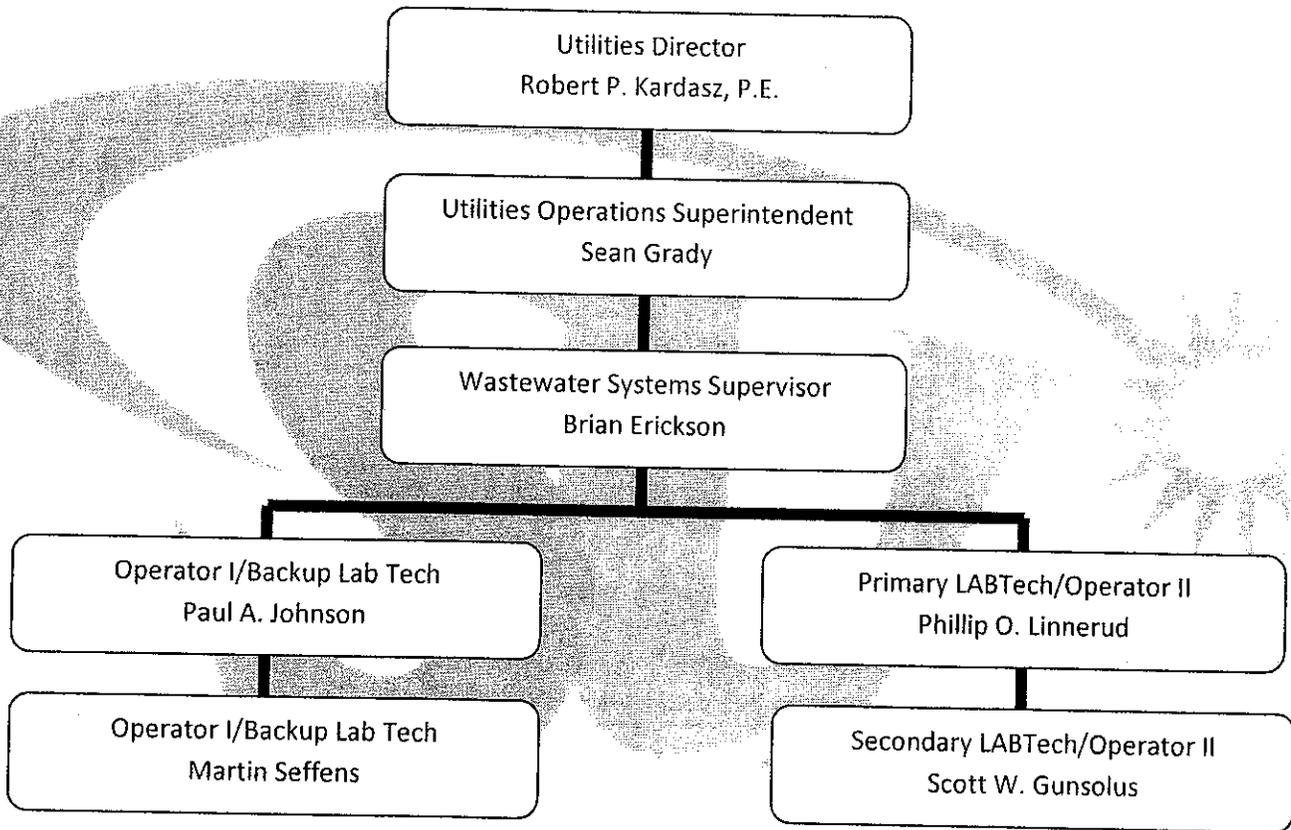
1. Influent flow, BOD, TSS, pH
2. Primary Clarifier, BOD, TSS
3. Aeration tank, DO
4. Aeration mixed liquor splitter, MLSS, TSS, VSS
5. Final Clarifier, Sludge Blanket
6. Plant effluent, CBOD, TSS, NH₃-N, T.Phos, pH, Fecal coliform, Mercury, Wet Acute & Chronic
7. Sludge storage tank, sample per permit for land application.
8. Digester, Settleability, TSS, VSS, pH, % solids
9. Dissolved Air Flotation Thickener, RAS, Primary sampler, DAF % solids
10. Gravity Belt Thickener, % solids

1. INTRODUCTION

The laboratory at the Stoughton 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. Quality Assurance (QA) is critical in producing sound, defensible data. This data provided the empirical evidence upon which decisions are based. The purpose of this manual is to outline QA performed in the lab and to fulfill the requirements set forth in NR 149.37. Permit is reviewed annually.

2. LABORATORY ORGANIZATION AND RESPONSIBILITY

Organizational Structure of the Stoughton 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. Stoughton Utilities Director, Robert P. Kardasz, received his Bachelor of Science in Civil Engineering Degree from the University of Wisconsin at Platteville and has completed graduate work at the Milwaukee School of Engineering and at the University of Wisconsin in Madison. As a professional engineer, he has served as a Civil Engineer with the City of Milwaukee Bureau of Engineers and as an Environmental Engineer with the Wisconsin Department of Natural Resources Bureau of Water Quality where he specialized in the requirement of Public Law 92-500 (Clean Water Act and Amendments). He has received water and wastewater certifications and was honored as a Life Member of the Wisconsin

Wastewater Operators' Association. He recently started his 32'nd year with Stoughton Utilities as Utilities Director. During his tenor, a number of wastewater treatment facility upgrades and collection system improvements have taken place.

Mr. Kardasz prepares the \$2+ Million Dollar Annual Budget, establishes user rates, facilitates the adoption of the Compliance Maintenance Annual Report, and reviews and signs the monthly Wastewater Discharge Monitoring Report. He is responsible for working with our wastewater professionals to comply with all local, State and Federal Regulations and to develop and facilitate their individual and divisional career paths.

- ii. Supervisor- 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 supervisor is trained and has extensive knowledge related to federal, state and local laws which regulate wastewater treatment and discharge.
- iii. 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.
- iv. Backup Laboratory Technician- The backup analyst does the laboratory suspended solids, BODs, CBODs and pH testing when the primary laboratory personnel is unavailable. The same requirements of the main analyst are required of any backup, weekend or fill-in analyst.

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 Stoughton Wastewater Treatment Plant are based do not contain specific IDC procedures, laboratory management has instituted the following IDC methodology: The IDC consists of documenting that 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 Stoughton Wastewater Treatment Plant, the results of the LCA 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 quarterly 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 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 the 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. The personnel records are located in the file cabinet on the north wall.

C. Analytical Records.

- i. The Stoughton Wastewater Treatment Plant 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 bench sheets 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 indentified 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 bench sheet.
 3. Preservation Status- Samples arrive to the laboratory immediately after collection from refrigerated auto samplers. Therefore, samples by Stoughton Wastewater Treatment Plant 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 bench sheet.
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 and the date disposed of purchased stock reagents are documented in a logbook upon receipt.

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

3. Follow corrective action logbook.

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 Stoughton Wastewater Treatment Plant 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 to their respective originating stocks or neat compounds. The material name, Stoughton Wastewater Treatment Plant 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 purchased from Badger Water in 5-gallon containers.

Table 3. Sampling Handling Guidelines

PARAMETER	SAMPLE TYPE	PRESERVATION	CONTAINER	@MAXIMUM HOLDING TIME	*ANALYTICAL METHOD
Biochemical Oxygen Demand C BOD	24-hr composite [flow proportional]	Cool, $\leq 6^{\circ}\text{C}$	Polyethylene	48 hours	5210 B
Total Suspended Solids	24-hr composite [flow proportional]	Cool, $\leq 6^{\circ}\text{C}$	Polyethylene	7 days	2540 D
Ammonia-Nitrogen	24-hr composite [flow proportional]	Cool, $\leq 6^{\circ}\text{C}$ H_2SO_4 to $\text{pH}<2$	Polyethylene	28 days	4500-NH ₃ F
Total Phosphorus	24-hr composite [flow proportional]	Cool, $\leq 6^{\circ}\text{C}$ H_2SO_4 to $\text{pH}<2$	Polyethylene	28 days	HACH 819 5 th Addition
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, $\leq 10^{\circ}\text{C}$	Glass jar 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 Addition

Table 1 – WPDES Permit Requirements

SAMPLE LOCATION	SAMPLE TYPE	SCHEMATIC REFERENCE	PARAMETERS TESTED	MONITORING FREQUENCY
Influent	Continuous	1	Flow	Totalized Daily
Influent	24-hour composite (flow proportional)	1	Biochemical Oxygen Demand Total Suspended Solids	<u>BOD</u> Wed Thurs Friday <u>TSS</u> Tue-Thurs
Effluent	24-hour composite (flow proportional)	2	Biochemical Oxygen Demand Total Suspended Solids Ammonia-Nitrogen Total Phosphorus	Daily SS & Ammonia & Phosphorus Tues- Thurs <u>BOD</u> Wed- Friday
Effluent	Grab	2	Dissolved Oxygen pH	3 x week
Effluent	Grab	2	Fecal Coliform [#]	Twice weekly

Seasonal Disinfection:

- Ultraviolet (UV) disinfection only required during the period from April 1st to October 15th of each 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.

2.2.1 Sampling Point (Outfall) 001

Monitoring Requirements and Effluent Limitations

Sample Location	Parameter	Limit Type	Limit and Units	Sample Frequency	Sample Type	Notes
Effluent	Flow Rate	-	MGD	Continuous	Continuous	
Effluent	CBOD ₅	Monthly Avg	25 mg/L	3/Week	24-Hr Flow Prop Comp	
Effluent	CBOD ₅	Weekly Avg	33 mg/L	3/Week	24-Hr Flow Prop Comp	
Effluent	CBOD ₅	Weekly Avg	40 mg/L	3/Week	24-Hr Flow Prop Comp	
Effluent	CBOD ₅	Weekly Avg	454 lbs/day	3/Week		
Effluent	Suspended Solids, Total	Monthly Avg	30 mg/L	3/Week	24-Hr Flow Prop Comp	
Effluent	Suspended Solids, Total	Weekly Avg	40 mg/L	3/Week	24-Hr Flow Prop Comp	
Effluent	Nitrogen, Ammonia (NH ₃ -N) Total		mg/L	3/Week	24-Hr Flow Prop Comp	Monitor Only
Effluent	Fecal Coliform	Geometric Mean	400 #/100 ml	2/Week	Grab	April 15 through Oct 15
Effluent	Dissolved Oxygen	Daily Min	6.0 mg/L	3/Week	Grab	May 1 through Oct 31
Effluent	pH Field	Daily Max	9.0 su	3/Week	Grab	Influent & Effluent
Effluent	pH Field	Daily Min	6.0 su	3/Week	Grab	Effluent & Influent
Effluent	Phosphorus, Total	Monthly Avg	1.5 mg/L	3/Week	24-Hr Flow Prop Comp	Effluent

5. SAMPLING PROCEDURES AND MAINTENANCE

A. Samples must be taken at 7:00 a.m. on the mornings of Tuesday through Friday.

Turn samplers on at 7:00 on Monday turn samplers off on Friday after samples are taken, rinse sample containers each day.

5 gallon sampler containers shall be cleaned each Friday with 10% Hydrochloric Acid and rinsed with distilled water. Let sample containers dry over the weekend. Then put back in samplers on Monday.

B. Maintenance on Samplers

To turn samplers off push the halt key

To turn samplers on push the resume key

To set sampler up for pumping sample

1. Press new program
2. Program delay press no
3. Flow mode press yes
4. Variable interval press no
5. Interval = 0005 counts press yes
6. Continuous mode press yes
7. Sample volume = 150 ml press yes
8. Calibrate volume press yes
9. Auto calibrate press no
10. Times calibrate press yes
11. Ready to pump press yes

The pump will begin pumping. Place a beaker under the discharge hose and measure around 150 mls then push stop pump key.

The try again will come up on the screen press no.

Ready to start will come up on the screen press start program.

C. Procedure for Handling Samples

1. Two flow-proportional samplers are used to obtain samples from the influent channel before screening and the effluent channel before disinfection (UV). The samplers received signals from the influent and effluent flow meter. Samplers have refrigerator that maintain a temperature of 0-6°C.

2. Temperature of samplers are recorded each day.

Samples are identified by sample date, sample time, sample type, influent, primary, effluent, MLSS and RPS, and initials of the person.

During the sampling, all samples from ammonia and phosphorous are acid preserved to a pH <2. 13 drops of 35.6N sulfuric acid and stored in refrigerator. Put date and time on sample bottle. Example: Inf 1/21 7:00 Eff 1/21 7:00

3. Samples are at room temperature 9.7-20.3°C before testing, usually between 8:00-10:00 a.m. is when testing is done. If they cannot be analyzed during this time they

are put into the refrigerator until they can be analyzed and warmed up at that time. Influent, primary and effluent are composite samples MLSS and RPS are grab samples. Wednesday through Friday influent and effluent composite samples are analyzed for pH.

4. Grab samples are collected in the morning by the Lab Tech in plastic bottles, pH samples for both influent and effluent are tested immediately Tuesday through Thursday.

For fecal coliform sample use a sterilized glass jar. To sterilize, wash jar with 10% Hydrochloric acid, rinse with distilled water, let dry. Put a drop of 10% sodium thiosulfate into the sample jar, then sample is taken.

5. Sludge report, toxicity and mercury testing is done by a Commercial Lab. For sludge report sampling, 2 areas of the sludge storage tank are sampled total of 7 grab samples. All sample jars are provided by the certified lab.

Samples are kept in refrigerator at 0-6°C.

When shipping, sample coolers are packed with ice on top and around samples. Stoughton uses Dunhams Express for transporting samples. Commercial Labs used by Stoughton are Test America phone 1-800-833-7036 and for sludge samples Northern Lakes Service phone 715-478-2777 for toxicity and mercury testing.

6. Sample bottles are permanently labeled for their use.

If sampler does not pump sample, a grab sample will be used and will be noted on testing Benchsheets.

Sample containers are 5 gallon containers, filled to 2-3 gallons. Container is capped and shaken many times and is poured into appropriate sample bottle 2 1000 ml for effluent, 500 ml for influent, primary, MLSS and RPS. After use sample bottles are washed with tap water and non phosphate soap. All soap is rinsed out with tap water and then re-rinsed with distilled water. Sample bottles for phosphorous are scrubbed with non-phosphorous detergent and rinsed with 10% hydrochloric acid, then re-rinsed with distilled water.

7. A one time test is done when sample containers are cleaned. Let container dry, then pour RO water in it and use for blanks on BODs, Suspended Solids, Phosphorous and ammonia.

D. Sampler Hose Cleaning

1. Sample tubing is replaced each month
2. Pump tubing is washed out weekly by soaking hose in 10% Hydrochloric acid scrubbed and rinsed out with distilled water
3. Strainer is washed out weekly with non-phosphate detergent and rinsed with distilled water

4. Sampler is cleaned weekly with non-phosphate detergent and rinsed with water
5. Sampler tubing is located in bottom shelf of island in the lab

A. Analytical Balance

1. Zero balance
2. Calibrate in house once per month. Use class 5 weights.
3. Calibrated annually by B&M Technical Services
4. Keep clean. Remove dust or spilled chemicals

B. Desiccator, used to dry SS crucibles

Replace desiccant when it turns purple.

C. Muffle furnace- used to burn off solids- total volatile solids test

D. Lab refrigerator

1. Keep clean
2. Thermometer calibrated by B & M Technical once per year

E. Influent sampler

1. Keep Clean
2. Thermometer calibrated by B & M Technical once per year

F. Effluent sampler

1. See Influent sampler

6. MAJOR LAB INSTRUMENTS

- ✓ Hach Odyssey
- ✓ DR2500 Spectrophotometer
- ✓ Orion Model 720 A+
- ✓ PH/Ammonia Meter
- ✓ PH Probe Orion 9157 BN
- ✓ Ammonia Probe Orion 9512 BN
- ✓ Oven Blue M Model OVI2A
- ✓ Balance Denver Instruments APX-100
- ✓ Muffle Furnace thermolyne Model 1500
- ✓ BOD Meter YSI Model 58
- ✓ BOD Probe YSI Model 5905
- ✓ Incubator Precision Scientific Model 815

7. EQUIPMENT MAINTENANCE

- A. Each piece of equipment has a file. The file includes owner's manual, preventative maintenance schedule and records of repairs and purchases. If a piece of equipment is repaired, document the

cause, the date of repair, who did the work and the cost. Also, record any breakdown that affects Lab results.

- B. The DO and Ammonia Probe membranes are replaced every 1 to 2 months or more frequently if readings are erratic.

8. LABWARE CLEANING

- A. All glassware is washed in dishwasher with a non-phosphate detergent.

Once per month, BOD bottles are acid washed using a 10% Hydrochloric Acid then rinsed with tap water and finally rinsed with distilled water two times.

Glassware for Phosphorous testing is washed with non-phosphate detergent then acid washed with 10% Hydrochloric Acid and finally rinsed with distilled water. Pipettes are acid washed and then stored in distilled water. After using pipettes, discard distilled water. All Phosphorous glassware is separated. The Shelf is labeled for phosphorous testing only. When phosphorous testing, do not use the same piece of glassware that is not used for the blank, influent and effluent sample each time.

BOD Bottles are stored dry. Sample bottles and 5 gallon carboys are rinsed daily and acid washed weekly using a 10% Hydrochloric Acid wash. They are then rinsed with tap water and distilled water.

9. INSTRUMENT CALIBRATION

- A. The pH meter, DO meter and ammonia electrode are calibrated each day that they are used. The balance is zeroed each day that it is used. The temperatures of the incubator, refrigerator and influent/effluent samplers are measured by Nist thermometers purchased from NCL and calibrated once per year. The temperatures are recorded on the log sheets. Correction factors are labeled on the thermometers. The temperatures for the Drying oven and Fecal Coliform Incubator are recorded each day they are used. If the temperature is out of range, adjust thermostat.

*Note: If thermometer liquid splits or if is + or - 2° CF, throw away and order a new one.

- B. Analytical balance is calibrated annually. Once per month two class S weights are used to check calibration.

10. PROCEDURES FOR EVALUATING QUALITY CONTROL SAMPLES

A. Quality Control

1. Analysis of Blank, Second Source Initial Calibration (QCS) standard, Continuing Calibration Verification (CCV) standards and Lab Control Samples (LCS) are performed. When corrective action on an LSC failure does not work, it is noted on DMR. Results for GGA's must be 167.5 - 228.5 mg/l. If they are not, record on DMR. Records of quality controls are kept on daily benchsheets.

2. Method Blanks

- a. Method Blank means a sample of clean distilled water. It is prepared under the same conditions as the samples in each test.

For phosphorus, a method blank is run without chemicals to zero spec then a second method blank is run with chemicals.

For an Ammonia blank, add ISA buffer. When the blank is above the detection limit, the lab evaluates the nature of the interference and its affect on each sample of a preparation batch. A sample in a batch associated with a method blank that fails is reanalyzed or qualified on the DMR. The method blank must be below the highest of LOD.

b. Initial calibration verification ICV.

i. A second source ICV standard is used when phosphorus test is run. Stoughton uses a .5 standard first. An ICV is run then another second source ICV is run. Two different lots are used. When a calibration curve is run, a .5 ICV from a different source is run to verify that the calibration is valid. The .5 ICV must fall within .45 - .55 mg/l + or - 10% of true value. A second source standard does not have to be used for SS, BOD or Ammonia.

c. Continuing calibration verification for phosphorus

i. A CCV/LCS is analyzed on days other than calibration days. The concentration used at Stoughton is .5 mg/l and must be within + or - 10% of value. If the CCV does not pass another CCV is run. If the second CCV does not pass the lab takes corrective action. After Correction action is taken, the lab must take two CCV's in a row. If these CCV's do not pass a new calibration curve must be run and analysis of all samples is repeated. All tests that do not pass are noted on DMR. CCV's are not required for Ammonia.

ii. Lab Control Samples (LCS)

These standard s are known standards that are purchased from North Central Labs. They are used to verify the accuracy of the results.

iii. Proficiency testing PT samples

PT samples are purchased from the State Lab of Hygiene once per year. Stoughton purchases samples for BOD, CBOD, Suspended Solids, Phosphorous and Ammonia. If one of the standards fails the lab will automatically receive one from the Lab of Hygiene. If all the tests pass, they automatically load into the lab certification computer system.

11. MAJOR LAB INSTRUMENTS

A. YSI Model 5905 self stirring probe: Change membrane when readings become erratic or when there is slow drifting. Membranes are in the drawer labeled "DO Meter & Probe Equipment". Unscrew old membrane from probe, rinse with distilled water and take out new membrane cap. Fill the cap with oxygen probe electrolyte. Screw membrane cap onto probe. Probe is stored in a BOD Bottle with water in the bottom when not in use.

B. YSI model 58 DO Meter: Zero meter and check temperature on meter with the digital thermometer four times per year.

C. Orion 9512 BN Ammonia Probe:

1. Store in a 1000 mg/l standard
2. Change membrane one time per month.

To change membrane, go to page 4-7 of the thermo scientific users guide for Ammonia Ion Electrode under Electrode assembly.

D. pH Probe Orion 91-51 BN

1. A weekly inspection of the electrode to check for scratches, cracks or crystal deposits needs to be done.
2. Rinse off any salt build up with distilled water

3. Drain the reference chamber, flush with fresh filling solution and refill the chamber. For the filling solution, use Orion 9000II
 4. Store probe in NCL Electrode storage solution
- E. Odyssey Spectrophotometer: Used for phosphorous testing check four times per year for verification of different wave lengths.
- F. Millipore Incubator: Used for fecal coliform

12. STOUGHTON WASTEWATER TREATMENT PLANT WEEKLY LAB SCHEDULE

A. Monday:

1. Turn on influent, primary and effluent samplers
2. Read all thermometers, samplers, refrigerator, drying oven and incubator. Write temperatures on specified lab benchesheets.
3. Calibrate meters and read BODs and CBODs

B. Tuesday:

1. Collect influent, primary, effluent, MLSS and RDS samples
2. Effluent composite phosphorous sample
3. Same as #3 above
4. pH testing – grab influent and effluent
5. Set up suspended solids
6. Read BODs and CBODs

C. Wednesday:

1. Collect samples. Refer to Tuesday #1
2. Effluent composite phosphorous sample
3. Same as #3 above
4. pH testing influent and effluent – grab composite
5. Read and set up suspended solids
6. Read and set up BODs and CBODs

D. Thursday:

1. Collect samples. Refer to Tuesday #1
2. Effluent composite sample
3. Same as #3 above
4. pH testing – influent and effluent – grab and composite
5. Read and set up suspended solids
6. Set up BODs and CBODs, BODs for B&G Foods

E. Friday:

1. Collect samples – influent, primary and effluent
2. Turn off samplers
3. Read thermometers
4. pH testing influent and effluent composite
5. Read suspended solids
6. Set up BODs, CBODs and BODs for Colorcon

F. Notes:

1. April 15th – October 15th testing for fecal coliforms two times per week
2. January – March test for Influent phosphorous

3. Sample for mercury once per quarter influent and effluent
4. Sludge testing in April
5. Friday take influent and effluent 5 gallon sample jug to lab, clean with 10% Hydrochloric Acid and rinse three times with distilled water. Let dry over the weekend and put them back into samplers on Monday.
6. Rinse out sample bottles daily. Clean with 10% Hydrochloric Acid on Fridays, rinse three times with distilled water and let them dry out over the weekend.

13. MINIMUM QC REQUIREMENT

- A. Total suspended solids method 2540 D 21st Edition
 1. Balance calibrated yearly by an outside source
 2. Re-dries on influent and effluent quarterly
- B. BOD SM5210 21st Edition
 1. Meter calibrated daily
 2. Method blank is used with each batch of test
 3. LCS GGA one time per week
- C. Ammonia (NH₃95N) SM 4500- NH₃D 21st Edition
 1. Calibrate meter when testing
 2. Method blank when testing
 3. CCV after 20 samples
 4. LCS one per each batch of samples
- D. Total Phosphorous- SM 4500- p B 5 and PE 21st Edition
 1. Calibration curve is run at least yearly using a minimum of 3 standards a new curve be run when a lot of chemicals can no longer produce a 100 + or - 10% CCV
 2. Method blank when testing
 3. ICV is required when a full calibration curve is made
 4. LCS once per every batch
- E. Control Limits
 1. BOD/GGA 198 + or - 30.5
 2. Ammonia LCS + or - 10%
 3. Total Phosphorous ICV, CCV, LCS + or - 10%
 4. Suspended solids + or - 10 %

14. MEASUREMENT TRACEABILITY OF THE STOUGHTON LAB

- A. All results can be referenced to all standards and reagents used.
- B. The accuracy of the certified weights used to calibrate balance or verify the calibration of support equipment.
Calibration of balance weights and thermometers are calibrated by B&M Technical Service.

Calibration of standards must provide traceability.

C. No standards can be used after the expiration date unless the lab can verify in a defensible manner.

15. MEASUREMENT TRACEABILITY DOCUMENTATION

- A. Identify source and purity of all standards and reagents and keep records of invoices.
- B. Document lot #, manufacturer, receipt date and expiration date of standards and reagents.
- C. Document all Lot # standards and reagents on bench sheets for that particular test.
- D. Keep records for analytical support equipment.

16. PROCEDURES FOR INITIATING, FOLLOWING UP ON AND DOCUMENTING CORRECTIVE ACTION ADDRESSING QA AND QC FAILURES

A. Corrective action must be taken when any of the following fails:

1. Suspended solids' weight is less than .10
2. BOD blanks are less than .20, depletion samples are greater than .20 depletion and less than 1.0
3. Method blanks and/or LCS in phosphorous
4. Blanks and 3 standards are Ammonia
5. Failure of a PT sample

B. Lab reports results in which one or more of the QC samples has failed to meet acceptance. The data is flagged on the DMR report by typing in a "1" on the QC field. The date or dates of the testing are documented in the lab QC comments section of the electronic DMR. Comments include dates of the tests are affected, details of the failure.

C. All lab data results are completed by and reviewed by Phil Linnerud, then they are entered into DMR files onto the computer. They are printed out and given to Brian Erickson, Plant Supervisor. If any QC sample fails to meet control limits and/or exceed permit limits, they are also noted on DMR. After Brian Erickson reviews the DMR, Robert Kardasz, Director of Public Works, reviews the DMR and signs the DMR.

Brian Erickson sends DMR electronically to the DNR.

Corrective Action Log	
Describe the problem. Note if reported on DMR	
Describe corrective action	
Date and Initials	
Describe data after corrective action is taken	

Is the improvement at an acceptable level	
Will further corrective action be needed	
Date and Initials	

** Copy for lab manual and bench sheet

D. The action taken in fixing the problem is documented in the log. If the action taken does not improve, a new corrective action is taken and documented the same as the first attempt. This continues until test is acceptable.

The following are run quarterly by Scott Gunsolus and Paul Johnson.

Total SS Influent and Effluent control limits from '08 are used.

BOD, CBOD Influent and effluent control limits from '08 are used.

Glucose, Glutamic Acid- Control limit 167.5 - 288.5

The following tests are run side by side with the primary Lab Tech - Phil Linnerud.

IDs for Total Phosphorous and Ammonia

1. Must pass 4 PT Sample
2. QCS
3. LCS
4. ICV
5. Calibration Curve for Total Phosphorous
6. CCV for Phosphorous
7. MDLs for ammonia and Phosphorous
8. Method Blank



Minimum Required QC - New NR 149*

Test	ICAL	ICV	CCV	Method Blank	LCS	QCS (blind)	LOD	IDC ^①	PT
*No Matrix Spike or replicate required for these 4 parameters in Code or Standard Methods									
BOD cBOD	<input checked="" type="checkbox"/> Daily	NA	NA	<input checked="" type="checkbox"/> 1/batch	<input checked="" type="checkbox"/> <20 smpls/week else 1 per batch of 20	NA	NA	<input checked="" type="checkbox"/> Once/ analyst	<input checked="" type="checkbox"/> 1/Year
TSS	Monthly balance check -gm range -mg range	NA	NA	NA	NA	NA	NA	<input checked="" type="checkbox"/> Once/ analyst	<input checked="" type="checkbox"/> 1/Year
NH3	<input checked="" type="checkbox"/> Daily	NA	<input checked="" type="checkbox"/> IF > 20 samples	<input checked="" type="checkbox"/> 1/batch	<input checked="" type="checkbox"/> 1/batch	NA	<input checked="" type="checkbox"/> Yearly	<input checked="" type="checkbox"/> Once/ analyst	<input checked="" type="checkbox"/> 1/Year
Total P	<input checked="" type="checkbox"/> If CCV fails; at least annually	<input checked="" type="checkbox"/> 2nd source/ after Cal	<input checked="" type="checkbox"/> Daily + IF >20 samples	<input checked="" type="checkbox"/> 1/batch	<input checked="" type="checkbox"/> 1/batch (replaces MS/replicates)	NA IF use 2nd source; Else 3/yr	<input checked="" type="checkbox"/> Yearly	<input checked="" type="checkbox"/> Once/ analyst	<input checked="" type="checkbox"/> 1/Year

① IDC requires that lab have some means of documenting that each analyst that will be performing a particular test is capable of performing the test. Offers flexibility in compliance.

17. TOTAL PHOSPHORUS

Hach Test 'N Tube Method: PhosVer 3 with Acid Persulfate Digestion
Hach Products for Analysis 8048 & 8190
(equivalent to standard methods 4500-p, B,5 and P.E. 21st Edition)

A. Test Procedure:

1. Make sure all glassware used for test has been properly acid cleaned and rinsed with distilled water.
2. Turn on COD vial reactor to warm up before setting up test tubes. Reactor temperature should be at 150 + or - 1° C for proper digestion.
3. Set up a label test tubes and place in test tube rack. Set up a zero blank for chemicals with no digestion and a method blank (distilled water with all chemicals and digestion), effluent samples, and a standard within the range of the curve as a CCV/LCS accuracy check. (a. 0.5 std is normally used - 2 ml of a 50 ppm diluted to 200 ml distilled water), (open standards are refrigerated): 5 ml is added to each tube using the 5 ml volumetric.
4. Effluent wastewater samples must be diluted to obtain a more accurate result from phosphorous curve. Use a 10 ml volumetric pipette. Pipete 10 mls of effluent sample into a 50 ml volumetric flask. Fill to volume with distilled water. Dilution factor is 5. Shake flask well before transferring sample to Test 'N Tube.
Influent wastewater samples and pipette 10 mls of Influent sample into a 100 ml volumetric flask fill to volume with distilled water dilution factor is 10. Shake flask well, before transferring sample to Test 'N Tube.
Fill each Test 'N Tube with 5 mls of diluted sample with a 5 ml volumetric pipette.
*Note: Always use a new pipet when pipetting different chemicals for samples so as to prevent contamination of the chemicals or samples.
5. All sample test tubes should be at 5 ml before moving on to the next step of the test procedure.
6. Using the small glass funnel, add one potassium persulfate powder pillow to each test tube.
7. Screw a cap tightly onto each test tube and shake vigorously to mix well and to dissolve all the chemicals.
8. Place all the test tubes into the COD vial reactor, and set the timer for 30 minutes at 150°C.
9. At the end of the 30-minute digestion period, remove all the test tubes and place back into the test tube rack for approximately 30 minutes of cooling back to room temperature.
*Note: Turn on the spectrophotometer and allow to warm up for 30 minutes while the test tubes are cooling down. Make sure the cell holder is empty when turning on the machine so no interference occurs during the set-up mode.
10. Take the caps off of the vials and add 2 ml of 1.54N sodium hydroxide to each test tube with the tensette pipet.
11. Screw the caps back on to each of the test tubes and shake vigorously to mix.
12. Remove the caps from the test tubes and add a PhosVer3 Phosphate reagent powder pillow to these tubes.
13. Cap the test tube and shake vigorously again to dissolve the reagent. Set the timer for FOUR minutes for color development before reading. Read after four minutes.
14. Once the proper time has elapsed for color development, turn the wavelength up on the spectrophotometer to 880nm. Make sure the readout window is set up to display in ABS for absorbance.

15. Take the reagent Blank and use a Kimwipe to clean the outside of the test tube. Also check for any bad scratches that might affect the readout.
16. Place the zero blank in spectrophotometer first and press zero.
17. Remove the zero blank and place the 0.5 standard LCS in read out in absorbance, then place method blank in read out in absorbance. If method blank is greater than MDL or LCS is out of the 100 + or - range, test will have to be done over. Save samples until calculations are done.
18. Repeat the above steps (16-18) for each of the test tubes set up that particular day.
19. Take the ABS results read from the spectrophotometer and go to the Strand P Excel file on the computer for the linear regression curve. Next enter in the ABS results in order to get the results interpolated to concentration in mg/L. Remember to multiply the diluted sample results by 5, or whatever the dilution factor was.

B. Phosphorous Curve

Run 5 standards (i.e. .1, .2, .5, 1.00). Enter the absorbance of each standard in Excel Work, Strand P, Curve data. Click on Run Regression. A correlation coefficient of at least .995 generally indicates acceptable characterization of the curve. If this degree of correlation is not obtained, the reason for the lack of linearity should be investigated, any necessary corrective action taken, and a new calibration curve must be constructed. A new curve should be run at least quarterly, or whenever reagent lots are changed.

C. Limit of Detection (LOD) or MDL

1. Determine a spike concentration, (1 to 5 times the est. Detection Level) - 1.
2. Prepare 7 or 8 replicate of reagent water spiked at an appropriate level.
3. Analyze the replicate spikes.
4. Calculate the LOD using Excel Work MDL.
5. Perform the "5-point" check of the LOD. (See Below)
6. Repeat the LOD determination at least yearly, and if there are changes in analyst or method.

D. "5 Point Check"

1. Does the spike level exceed 10 times the LOD? If so, the spike level is high.
2. Is the calculated LOD higher than the spike? If so, the spike level is too low.
3. Does the calculated LOD meet regulatory requirement (i.e. Permit limits)?
4. Is the Signal/noise (s/n) in the appropriate range?
5. Are the replicate recoveries reasonable?

E. Sample Storage

If a phosphorus sample needs to be stored the pH must be reduced to <2. Do this by adding 12 drops of 35.6N Sulfuric Acid to 150 ml of sample. Shake and refrigerate. The holding time of the sample is 28 days.

Notes:

1. CCV/LCS: Every 20 samples use a .5 mg/l must be within 100 + or - 10%
2. ICV second service immediately after calibration. When curve is generated use a .5 standard must be within 100 + or - 10 %.
3. PT one time per year

4. 35.6N Sulfuric Acid, 5 and 50 ppm standards are kept in refrigerator.
5. Test N Tube kits are located in south east corner cabinet
6. Test N Tube kits are purchased from Hach (phone #: 1-800-227-4224) catalog #308869198, account #809644.
7. ACS is run three times per year.
8. If bulb is changed in spec, a new curve is run.
9. Change bulb one time per year, then set up new phase curve.

Suggested Way to Prepare Stock Total Phosphorus Standards

mL Stock Std. Diluted to 1000 mL	Conc. Stock Std to use (ppm)	Final Conc. Stock Standard (ppm)
5	1000	5
50	1000	50
100	1000	100
20 (diluted to 200mL)	50	5

*Note: **Always** use class A volumetric pipets to prepare standards.*

Suggested Way to Prepare Working Total Phosphorus Standards

mL Stock Std. Diluted to 200 mL	Conc. Stock Std to use	Final Conc. Working Std.
4	5	0.1
8	5	0.2
2ml	50	0.5
3ml	50	.75
4ml	50	1.0
5ml	50	1.25
6ml	50	1.50
7ml	50	1.75

*Note: **Always** use class A volumetric pipets or air displacement pipets (Ependroff, Rainin, Gilson, etc.) to prepare standards.*

18. DETERMINATION OF FECAL COLIFORM

Membrane Filter Fecal Coliform Test (MFFCC)

Reference: Standard Methods, 21st Edition, Procedure 9222 D

A. PRE-SAMPLING PREPARATION:

1. Select a sample container of either hard glass (Pyrex) or nontoxic plastic (polypropylene), preferably with a wide mouth and at least 100 ml in size. Deliver 0.1 ml. of 10% sodium thiosulfate solution to sample container for each 100 ml. to be collected. This will neutralize any chlorine present. Cap container loosely and sterilize.
2. Sterilize the funnel unit of the filtering assembly.
3. Sterilize all pipettes and graduated cylinders to be used.

B. SAMPLING:

1. Sample must be a grab sample. Sample must be collected directly into the sterile sample bottle. The sample bottle should not be filled to the top so it can be shaken and mixed well during the test. Also, if overflow occurs the sodium thiosulfate may be lost and chlorine will not be dissipated.
2. The test should be set up within 6 hours of sample collection. If samples must be sent out for testing they must be collected as described above and must be iced and/or refrigerated during transit to the lab. The maximum time between sample collection and testing is 24 hours.

C. TESTING: Millipore Incubator

1. Disinfect work area with alcohol solution.
2. Label each sterile petri plate with sample volume to be filtered.
3. Choose sample volumes based on expected number of fecal coliform. Volumes should cover a range which results in at least one plate having 20-60 colonies.
4. Handle sterile pads and filters with a forceps dipped in alcohol and flamed off in a Bunsen burner or alcohol lamp. Only the outer 1/8 inch of the filter should be touched with the forceps.
5. Place a sterile absorbent pad in each plate. Tap the contents of an MFFCC ampoule onto the pad and pour off any excess liquid.
6. Place the sterile filter on the funnel base. Carefully put the funnel in place.
7. Pour 20-30 ml of the buffer water onto the filter.
8. Shake the sample very vigorously before withdrawing each volume. Add the smallest volume to be filtered to the buffer water in the funnel. Apply a suction of not more than 20 psi. in 2 successive additions of the buffer water (20-30 ml) rinse down the sides of the funnel. Suction until dry.

9. Carefully remove the funnel and lift membrane into petri dish with sterile forceps. The membrane filter should be in contact with the pad with no air bubbles underneath. If air bubbles occur, lift the membrane on one side and lower slowly. Do not touch the filtered area with the forceps.
10. Repeat with next largest sample volume.
11. To incubate, place petri dishes in a water proof plastic bag ("Ziploc") and seal. Invert the bag and secure under water in a water bath at 44.5 + or - 0.2 degrees Celsius. The thermometer in the bath must read to a tenth of a degree.
12. Incubate the plates for 24 + or - 2 hrs. Remove the plates from the bag. Select those plate(s) that contain 20-60 blue colonies. Average those plates in this range or, if only one plate has 20-60 use that. If no plates fall in this range count those which are nearest to 20-60.

D. CALCULATION:

$$\text{Fecal Coliform/100 ml} = \frac{100 * \# \text{ of colonies counted}}{\text{Number of ml filtered}}$$

19. STANDARD OPERATING PROCEDURES FOR pH

- A. Turn on pH ammonia meter by pressing the standby options key.
 - B. Use pH buffers 7 - 10 to calibrate.
 - C. Put pH⁷ buffer solution in a 100 ml beaker then place beaker on magnetic stirrer. Place pH probe in the beaker. To calibrate the meter press #2 (calibrate key) it will say "enter # of buffers", press 2 then "yes". When the meter says ready, calibrate as (example 7.010) enter in 7.010 then press "yes". Do the same for the pH 10 buffer. Now the meter is calibrated.
 - D. The Influent and Effluent can now be analyzed for Ph.
Pour the Influent sample into the beaker and place probe into the beaker with it and measure the pH. When the meter says "ready" that is the pH of the samples. Do the same for the Effluent sample.
 - E. To shut off the meter, push the standby options key.
- Note:
1. Plug the probe into input 2 on the meter.
 2. Store probe in NCL's Electrode storage solution
 3. Use fresh 7 + 10 buffer solution. Buffer solutions are stored in the Incubator
 4. pH meter is an Orion 720 A⁺
 5. Manuals for probe and meter are stored in drawer labeled "pH Meter Manual" on the east wall of the lab.
 6. Filling solution is drained and filled once per month. Filling solution is purchased from NCL Orion 9000II
 7. When doing the pH test, uncover the fill hole.

20. LAB SUPPORT EQUIPMENT

- A. Precision Scientific BOD incubator

- Check temperature daily- 20°C thermometer checked for proper temp by Gary Venden once per year.
- Keep clean
- B. Drying Oven
 - Check temperature daily- 103°C – 105°C
 - Thermometer checked for proper temperature by Gary Venden once per year
- C. Hach COD Reactor
 - Check temperature when used for phosphorus testing.

1 Influent Requirements

1.1 Monitoring Requirements

The permittee shall comply with the following monitoring requirements.

1.1.1 Sampling Point 701

Monitoring Requirements and Limitations						
Influent Sample Location	Parameter	Limit Type	Limit and Units	Sample Frequency	Sample Type	Notes
Sampler Screen Building	Flow Rate		MGD	Continuous	Continuous	
Sampler Screen Building	BOD ₅ Total		mg/L	3/Week	24-Hour Flow Prop Comp	
Sampler Screen Building	Suspended Solids, Total		mg/L	3/Week	24-Hour Flow Prop Comp	Report on DMR for months of Jan – Mar
Sampler Screen Building	Phosphorus Total		mg/L	Weekly	24-Hour Flow Prop Comp	

21. STANDARD OPERATING PROCEDURE FOR TOTAL SUSPENDED SOLIDS

Dried at 103-105° C in drying oven

Reference: Standard Methods, 21st Edition, Procedure 2540 D

- A. Apparatus
 - i. Samplers Influent + Effluent 0-6°
 - ii. Glass Fiber Filters, NCL 934-AH 3.7cm
 - iii. Fiter Funnel and Suction Flask

iv. Gooch Crucibles

v. Drying oven 103-105° C Blue M

vi. Desicator – Drierite should be blue, when it turns pink replace it

vii. Vacuum Pump

viii. Balance- Denver Instrument

B. Procedure

i. Preparation of Filter and crucible

1. Place a 3.7cm filter into a Gooch Crucible making sure you do not touch the filter or crucible. Use a forceps or rubber gloves. Mark crucibles with an Identifying number (i.e. R1- Influent, F1- Effluent)

2. Rinse under suction with 3 25ml portions of distilled water.

3. Place crucible with filter into the drying oven and let dry at a temperature of 103-105°C overnight.

4. Removed crucibles from oven and place them into the Desicator. Let them cool for 30 minutes.

5. Zeroing the Balance: check the balance Bulls-eye level to make sure bubble is inside the target. Then press the zero Button on balance to zero. Make sure doors are shut when doing this or any other measurements.

6. Remove crucible from Desicator and weigh. Record the weight of the crucible with the Identifying symbol. Crucible may then be returned to Desicator until all the crucibles are read.

ii. Sample Preparation

1. Place weighed crucible onto Suction Flask. Turn on vacuum at 20 psi (vacuum switch is located on south wall and left of the BOD meter.) Then wet down filter with about 10mls of distilled water 3 times to seat filter.

2. Typical amounts of sample in milliliters to be used are:

a. Influent – 25mls – use 25 ml pipet

b. Primary – 25mls – use 25 ml pipet

c. MLSS – 10mls – use 25ml pipet

d. RPS – 5mls – use 25ml pipet

e. Effluent – 300 – 500mls use 100ml Graduated Cylinder.

If sample is really clear you will use 500mls and if it's cloudy use 300 or 400mls. You want to make sure the dry weight difference is greater than .0010g. For instance on Wednesday you set up an Effluent ss using 300mls, the weight of the crucible and filter is 17.4967. The next day you weigh the same crucible with sample and the weight is 17.4973; a difference of .0006g. This means your next ss sample will require 500 mls. Also note on DMR the date of the sample weighed that read .0006g. Also note on DMR that a larger sample was used the next day.

3. For cloudy samples measuring a difference of less than .0010g must be flagged and noted on DMR. For the above example, you would note on DMR "300ml of Effluent was used and came up with < .0010g. Corrective action for the next Effluent sample, 500mls was used".
4. Once all of the samples have all been filtered, place in oven and dry at 103-105°C for 24 hours. The next morning take out the crucibles place in desiccator and let cool for at least 30 minutes.
5. Remove from desiccator and weigh the sample. Record the weight in grams.
6. Calculate Suspended Solids mg/L using the following formula

- a. crucible and sample weight – crucible weight
- b. 100 / sample volume used

Example: Crucible and Sample Weight = 17.8493
 Crucible Weight = 17.8391
 Volume of Sample = 25ml

$$17.8493 - 17.8391 = 102 \text{ mg/L}$$

Influent:	Given	$\frac{100}{25} = 4$	$4 * 102 = 408 \text{ mg/L}$
	Volume of sample Used	25	
Effluent:	Given	$\frac{100}{400} = .25$	
	Volume of sample Used	400	

Quality Control

- i. Balance Verification: **1 per month**, use 100 mg or 1 gram
- ii. Professional clean and calibrate: **1 per year**. This is completed by B&M.
- iii. PT: At least **1 per year**
- iv. Keep weights in original storage box when not in use. Handle with plastic forceps.
- v. Send weights into Northern Balance every **5 years** to be calibrated.

vi. Re-dries on Influent and Effluent Samples: **4 times per year**

Take initial weight with sample then let dry 24 hours than weigh again. The difference in weight should be no more than + or - .0003grams. If the difference is more than + or - .0003grams, dry again and weigh.

vii. Sample holding time: **24 hours**

22. BOD SEEDING PROCEDURE WITH GLUCOSE GLUTOMIC ACID TEST

(Revised 1-13-2009)

GGA standard – NCL 198 ppm BOD standard

Use 6mls per bottle

Must use seed

GGA's 1 per week

Known standard = 167.5 - 228.5

A. Preparation of Seed

- i. Collect a raw influent grab sample the day before performing the test.
- ii. Place sample in incubator (20°C) overnight.

B. Preparation of Seed Controls

- i. Take the incubated raw influent sample out of the incubator – **Do Not Mix!!**
- ii. Pipet 3, 6 and 9 ml of the clear supernatant into three BOD bottles respectively.
- iii. Fill these three bottles with dilution water.
- iv. Determine the initial dissolved oxygen on each of the three bottles.

C. Preparation of Seeded BOD Samples

- i. Fill the bottles approximately $\frac{1}{3}$ – $\frac{1}{2}$ with dilution water.
- ii. Pipet 1 ml of the supernatant into each of the BOD sample bottles that will require seeding. Use 1 ml volumetric pipet.
- iii. Add the appropriate amount of sample to each of the bottles.
- iv. Complete the filling of the BOD Bottles with dilution water.
- v. Determine the initial dissolved oxygen (IDO) on each of the bottles.

D. Preparation of GGA's – Use 6ml volumetric pipet

- i. Shake NCL BOD standard and pour some out into a small beaker to avoid contamination

ii. Add 6ml of GGA standard to the BOD Bottles where 1 ml of Influent supernatant was added.

iii. Fill BOD Bottles to volume with dilution water.

Filling out Bench Sheet, see attachment.

iv. Calculations are the same as BOD's and CBOD's



23. STANDARD OPERATING PROCEDURES FOR 5 DAY BOD AND CBOD TEST

A. APPARATUS:

1. 300 ml BOD Bottles
2. Incubator (20° C)
3. YSI Model 54 D.O meter
4. Pipetor Graduated Cylinder
5. YSI Model 5905 BOD Probe

B. NUTRIENT SOLUTIONS:

1. Phosphate buffer 4ml
2. Magnesium Sulfate 4ml
3. Calcium Chloride 4ml
4. Ferric Chloride 4ml
5. Dilution water purchased from Badger Water

C. CALIBRATION:

1. Verify accuracy of DO meter-probe 1 time per month, using another thermometer. They should not be off + or - 1° C. If off by greater than .5° C then get probe fixed.
2. Calibrate barometer 1 time per month. Obtain local corrected barometric pressure from the internet. To calibrate barometer you need to un-correct the barometric pressure that was taken from the internet.
3. Example on how to un-correct barometric pressure.
 - a. Corrected barometric pressure is 29.65
 - b. The altitude in feet for Stoughton is 860
 - c. Determine the correction factor
$$CF = \frac{760 - (\text{Altitude} * .026)}{760} = \frac{760 - (860 * .026)}{760} = \frac{760 - 22.36}{760} = .971$$
4. The un-corrected BP = $29.65 * .971 = 28.79$
5. Set barometer at 28.79
6. Calibrating meter when doing CBOD analysis
 - a. Take barometer reading. Example: $\frac{29.65 * 25.4 - \text{given}}{760 \text{ given}} = \frac{753}{760} = .991$
~ .991 * room temp. (Example: 20.3° C) 60 to DO saturation table and look up what the DO saturation is for 20.3° C = 9.01
~ Final calculation is .991 * 9.01 = 8.93 – set meter to this value

Notes:

1. Make sure probe tip is dry when calibration is made
2. Make sure meter is zeroed
3. Room temp should be 19.7° C – 20.3° C when testing

YSI 5905 DO Probe

1. Screw on membrane caps. These should be replaced every 1 to 2 months or when there are erratic readings. Probe tips are purchased from NCL order #: YSI-5906
2. Silver anode and gold cathode need cleaning periodically (when they get tarnished). For the gold cathode use sanding disc provided in the 5906 membrane kit. Sand the gold cathode with a twisting motion about 3 times or until it is bright again.
For the silver anode: soak the probe in a 14% Ammonium Hydroxide solution for 2-3 minutes or overnight in a 3% Ammonium Hydroxide solution. Rinse with Distilled water.

3. To change membrane caps- remove the stir paddle from the probe by pulling it straight out. Then unscrew the old membrane cap from the probe and clean the probe tip with distilled water. Hold the membrane cap and fill it at least ½ full with the electrolyte solution. Screw the membrane cap on to the probe moderately tight. A small amount of the electrolyte should overflow. Rinse off probe with distilled water. Reinstall stir paddle and let probe sit overnight. Check membrane for air bubbles underneath wrinkles or tears.
4. Turn DO meter on and let probe warm up for at least 30 minutes before taking the first reading.
5. Store probe in a BOD bottle and fill with 1-2 inches of distilled water.
6. If having problems with DO probe, go to page 11 of the troubleshooting guide in YSI 5905 DO Probe Instruction Manual. The manual is located in the drawer marked DO Meter and Probe.

24. STANDARD OPERATING PROCEDURES FOR DETERMINATION OF BIOCHEMICAL OXYGEN DEMAND (BOD₅) AND CBOD

REFERENCE: STANDARD METHODS, 21ST EDITION, PROCEDURE 5210 B.

A. PREPARATION OF SAMPLE:

1. The diluted sample used to determine BOD must have a pH between 6.5 and 8.0 for municipal sewage for effluent, the pH range is generally between 5-9, but the buffering capacity of the phosphate buffer will often bring the pH of the diluted sample between 6.5 and 7.5. For unknown samples, check the pH of the dilution which uses the most sample to confirm that the dilutions lie in the proper pH range. As needed, neutralize samples with 1N sulfuric acid or 1N sodium hydroxide. Do not dilute the sample with the acid or base by more than 0.5% (1.5 ml in a 300 ml BOD bottle).
2. Take pH readings of composite Influent and Effluent samples, also, measure temperatures of both samples. The temperatures should be 20°C + or - .3°C.
3. Samples supersaturated with dissolved oxygen, over about 9.3 mg/l at 20°C, may be encountered during winter months or in localities where algae are actively growing (lagoons). To prevent loss of oxygen during incubation of these samples, the DO should be reduced by shaking the sample or aerating it with filtered compressed air. Air - RO Water ½ hour. Air-Effluent 10 minutes. Shake Influent for 2-3 minutes.
4. Samples of untreated industrial wastes, disinfected wastes (final effluent), high temperature wastes, or wastes with extreme pH values may not contain enough microorganisms to oxidize the biodegradable matter in the samples. Such wastes should be seeded.

B. DILUTION TECHNIQUE:

1. Typical dilutions for Stoughton WWTP:
 - a. Influent 5+6 mls
 - b. Primary 10+15 mls
 - c. Effluent 150-300 mls
2. Estimate the BOD of the sample and select suitable dilutions from the following table:

Estimated BOD5 (mg/l)	Suggested Sample Volumes (ml)
< 5	200, 250, 300
< 10	100, 150, 200
10-30	25, 50, 100
30-60	15, 25, 50
60-90	10, 15, 25
90-150	5, 10, 15
150-300	3, 5, 10
300-700	1, 3, 5
700-1500	0.5, 1, 3
1500-2500	0.25, .05, 1

- Using a large-tipped, volumetric pipette – for samples less than 50 ml – or a graduated cylinder for larger sample volumes, measure the proper amount of well-mixed sample into thoroughly cleaned and rinsed 300 ml bottles. Dilutions under 3 ml should be made by diluting the waste in a graduated cylinder before pipetting.
- Dilution water may be prepared immediately before use, or except for the addition of the phosphate buffer, days or weeks ahead of time. Add 1 ml for each nutrient solution per liter of dilution water. The phosphate buffer is the critical nutrient in stimulating contaminating growths so it must be added the day the water is to be used. Distilled water should be allowed to equilibrate with outside air for at least 24 hours preferably at 20°C before use. To avoid dust or dirt contamination, a paper towel or sponge should cover the bottle opening not the bottle cap.
- Each BOD bottle is filled by slowly adding sufficient dilution water so that the stopper can be inserted without leaving an air bubble but not so much that there is overflow. When volumes of sample used exceed 150 ml, additional nutrients should be added to the sample bottle. Use Hach nutrient bugger pillows.

C. INCUBATION AND DISSOLVED OXYGEN (DO) DETERMINATIONS:

- Calibrate DO meter each day of use and check membrane on probe. Calibration is done using a winkler titration.
- Determine the DO OD the two dilution water blanks and all sample bottles and record on data sheet as initial DO.
- Place the samples and the two dilution water blanks in a 20 ± or - 1°C incubator for 5 days. Fill water seals with dilution water and cap to reduce evaporation from seals. Check daily; add water to seals if necessary.
- Before removing the caps, pour out the water above the cap.
- After 5 days determine the DO of the two dilution water blanks and the sample bottles.

D. CALCULATIONS:

Using the data recorded:

$$\text{BOD mg/l} = (\text{initial DO} - \text{DO}_5) * \text{Dilution Factor}$$

$$\text{Dilution Factor} = \frac{\text{Bottle Volume (300 ml)}}{\text{Sample Volume}}$$

- Add nitrification inhibitor to all three bottles filled with effluent. Use two shots per bottle. Inhibitor from NCL
- Nutrient solutions from NCL
- Procedure for CBOD samples (Effluent). These samples need to be seeded.

- a. Set up three bottles. These are for the (control samples) (use settled Influent sample) pipette. Put 3 mls in bottle 1, 6 mls in bottle 2, 9 mls in bottle 3. Fill the bottles with distilled water.
- b. Set up three bottles. These are for the Effluent samples. Measure and pour the recommended amount of Effluent sample into each bottle. Then pipette 1 ml of settled Influent into each bottle. Lastly, fill bottles with distilled water.
4. Completely fill two bottles with dilution water to be incubated as blanks.
5. Label each bottle carefully as to sample and volume used. Record the data on the bench sheet.
6. Use two bottles for Influent pipette; 5 mls into the first bottle and 6 mls into the second bottle.

Use two bottles for Primary pipette; 10 mls into the first bottle and 15 mls into the second bottle.

Use three bottles for Effluent pipette. Use 150 mls – 300 mls depending on clarity of the sample.

For example: When the sample is clear, set up the bottles and use 250, 275 and 300 mls. Less clarity means using less an amount of the sample. Use your best judgment when setting up Effluent samples.

E. CALCULATION OF SEED CORRECTION:

1. Determine the 5 day DO concentration on each of the seed controls.
2. Use the same rule for DO depletion as in all other BODs (at least 2.0 mg/l DO depletion and at least 1.0 mg/l residual DO (after 5 days)).
(Standard Methods, Seventeenth Edition)
3. If none of the bottles attain a proper depletion, adjust the amount of seed addition accordingly in subsequent tests.
4. For each seed control dilution, the mg DO used per ml seed =

$$\frac{(IDO - DO_5 \text{ for seed control})}{\text{ml seed in seed control}}$$
5. If two seed controls meet the DO depletion criteria, calculate the average mg DO/ml seed for the two bottles.
6. Seed correction = (mg DO/ml seed in seed control) * (ml seed added to samples)
7. If the seed correction does not fall in the range of 0.6 – 1.0, but the seed controls met the DO depletion criteria, the amount of seed used in the sample bottles will have to be adjusted in subsequent tests.

F. CALCULATION OF BOD IN SAMPLE:

$$BOD_5 = BOD \text{ mg/l} = ((IDO - DO_5) - \text{Seed Correction}) * \text{Dilution factor} * \text{Dilution factor} = \frac{\quad}{300/\text{sample size}}$$

BOD Seed Dilution Guidelines

Estimated BOD of Seed	Dilutions for Seed Control	#ml Seed per BOD Bottle	#ml Diluted Seed (10ml seed + 90ml H ₂ O)
30	15, 25, 50	6 – 10	NA
50	15, 25, 50	4 – 6	NA
100	5, 10, 15	2 – 3	NA
150	5, 10, 15	1 – 2	NA
250	3, 5, 10	1	6 – 10
500	1, 3, 5	0.5	5

If the BOD of the seed is 150 mg/l or less, the seed may be added directly to the BOD samples without dilution. If dilution is necessary, use volumes noted in column 4. Set up the seed control dilutions as shown in column 2. Prepare seed controls with seed at full strength.



25. AMMONIA NITROGEN (NH₃ - N)

Standard Methods 21st Edition, Procedure 4500-NH₃ D

A. APPARATUS:

- a. Orion Analyzer Model 720A+
- b. Orion ammonia electrode model 9512
- c. Magnetic stirrer

B. REAGENTS:

- a. Ammonia free water: All reagents and dilutions must be made with ammonia free water.
- b. Ammonia standard 10ppm, 100ppm and 1000ppm
- c. Ammonia ISA buffer

C. PROCEDURE:

- a. The Orion 720a is set up for Ammonia N by pushing the 2nd and then the mv buttons until 2nd channel appears. Then push the mode key, until the mv appears.
- b. Three standards are prepared: .2, 2.0, 20.0 set up 3 200ml volumetric flasks. Fill them half way with distilled water. Use chart below for the .2, 2.0 and 20.0: Make sure 4ml volumetric pipettes are used. Be sure to rinse thoroughly after each standard is prepared. Dilute to 200 volume with distilled water. Shake well, then pour 100ml of the .2 standard and transfer it to a 150ml beaker. Do the same with the 2.0 and 20.0 standard.
- c. Bring pH up to 2.0

Final Standard Conc.	Vol. of Stock Standard	Conc. Of Stock Standard
0.2mg/l	4ml	10ppm
2.0mg/l /LCS 1.0mg/l	4ml /2ml	100ppm
20.0mg/l	4ml	1000ppm

- d. To calibrate meter, press calibrate, then it will ask you how many standards, press 3 and then yes.
- e. The 0.2 ppm standard is put on the stir plate with a magnet added, stirred slowly with the electrode put into the solution at a slight angle to avoid trapping air bubbles on the membrane. At this point 2 mL of ISA buffer is added and a timer is set at 10 minutes. After 10 minutes or when the reading stabilizes press the read button and record millivolts then punch in .20 for first standard then push the yes button. The readout will automatically go to 2nd standard. To get from concentration back to millivolts, press 2nd and the mv.
- f. Remove probe from the 0.2 std. and rinse with filtered water and blot dry with a clean paper towel. Add probe to the 2.0 ppm std. and repeat steps done for the 0.2 ppm std. The 20 ppm std. is run in the same way.

- g. Next a blank is run using only 100 mL of filtered water and 2 mL of ISA buffer. The timer is set for at least 15 minutes. After 15 minutes and after the reading stabilizes, or if you are below the LOD, record in mv and concentration. 100 mL of effluent composite sample to a 150 mL beaker, run the same as the 2.0 and 20.0 meaning 4 or 5 min to wait for concentration readout, if the sample is below 2.0 you will have to wait longer. Record results in concentration and mv.
- h. Make sure you save the sample for each day until you have all of the results. Any concentration over 20mg/l will have to be done again. When this happens take 20mls of sample, dilute with distilled water to 100ml volume. Run test again record concentration and multiply times 5 to get final concentration and mv.

Note: Electrode is stored in a 1000ppm standard solution. If sample does not turn blue after ISA addition, add 1 more ml, until blue. Reagents are never drawn directly from bottle. Shake reagent bottle, pour into a clean beaker then draw off standard.

1. Note: Ammonia Nitrogen sample must be preserved immediately upon entering the lab. The pH must be adjusted to <2 by adding 13 drops 35.6N sulfuric acid to a 250ml sample. Refrigerate to 0-6* C. this is for same day samples and up to 28 days. On day of testing, take samples out of refrigerator and let to warm up to room temperature before doing ammonia procedure. Put month and Date on 250mL
2. Change membrane as needed (when response time gets too slow)
3. Blank each analysis day
4. Calibration each analysis day
5. LCS every 2 weeks 1.0 mg/l standard
6. PT once per year
7. MV should be between 54-60

26. LIMIT OF DETECTION (LOD)

At least once per year

- A. Determine a spike concentration, (1 to 5 times the estimated detection level) 0.2.
- B. Prepare 7 to 8 replicates of reagent water spiked at an appropriate level.
- C. Analyze the replicate spikes.
- D. Calculate the LOD using Excel Work - MDL.

LIMIT OF DETECTION (LOD)

- A. Determine a spike in concentration, (1 to 5 times the est. detection level).
- B. Prepare 7 to 8 replicates of reagent water spiked at an appropriate level.
- C. Analyze the replicate spikes.
- D. Calculate the LOD using Excel Work - MDL.
- E. Perform the "5-point" check of the LOD. (See below)
- F. Repeat the LOD determination at least yearly, and if there are changes in analyst or method.

“5-Point Check”

1. Does the spike level exceed 10 times the LOD? If so, the spike level is high.
2. Is the calculated LOD higher than the spike? If so, the spike level is too low.
3. Does the calculated LOD meet regulatory requirements (i.e. Permit Limits)?
4. Is the signal/noise (s/n) in the appropriate range?
5. Are the replicate recoveries reasonable?

