NH Department of Environmental Services
Wastewater Engineering Bureau
Permits & Compliance Section

WASTEWATER LABORATORY
QUALITY ASSURANCE PROGRAM GUIDELINES

March 2010

NEW HAMPSHIRE DEPARTMENT OF Environmental Services

WD-10-10

WASTEWATER LABORATORY
QUALITY ASSURANCE PROGRAM GUIDELINES

Prepared by:
Stephanie Larson
Thomas J. Croteau
Roy Gilbreth

Wastewater Engineering Bureau
Permits & Compliance Section

STATE OF NEW HAMPSHIRE
DEPARTMENT OF ENVIRONMENTAL SERVICES
29 HAZEN DRIVE; PO BOX 95
CONCORD, NH 03302-0095
Revised April 30, 2009
(September 12, 2005)

TABLE OF CONTENTS

Forward

These guidelines have been developed to assist Wastewater Treatment Facilities in New Hampshire in developing and implementing Laboratory Quality Assurance. Every effort has been made to cite information that can be found in Standard Methods for the Examination of Water and Wastewater and/or EPA references. Considerable time has been put into conversations with EPA quality control specialists in Chelmsford, Mass., including the State Lab, to assure that the items contained herein are required. These guidelines are meant to assist facilities in developing a minimum program, and therefore are assumed to constitute minimum requirements for NPDES analyses. Several examples are noted herein to help the facility to write a Quality Assurance/Quality Control (QA/QC) manual. Please do not simply copy the examples and enter them (as is) in your QA/QC manual. They should be modified to fit your application.

Those facilities wishing to implement a more comprehensive program than outlined in this guideline are certainly encouraged to do so. A facility may produce its own quality assurance baseline, if acceptable documentation can be provided to demonstrate that the program is valid.

An acceptable "Quality Control Frequency Table" (see page 7) has been included in these guidelines, at the request of numerous wastewater treatment plant operators. This section is similar to plans reflected in EPA documentation (40 CFR 122.41(e) and industry standard quality control frequency plans. As with any QC frequency, the QC frequency may not guarantee defensible data, but will be considered adequate quality control by the NH Department of Environmental Services NPDES monitoring program. This frequency guidance table is only applicable to the NPDES program and should not be used to satisfy other regulatory agency requirements for quality assurance.

This guideline should not be used to satisfy QA requirements for drinking and wastewater laboratory certification in New Hampshire. Those interested in laboratory certification should contact the Quality Assurance Officer of the Laboratory Services Unit of NHDES.

When referencing Standard Methods, do not rely solely on the test method for guidance. There are other sections in the Standard Methods that should also be referenced such as Part 1000. Sections 1010 – 1100 are considered as part of the laboratory QA/QC program. In addition, 40 CFR 122.41 (e) and NPDES permit Part II, Section B.1. mentions that proper operation and maintenance of a treatment facility also includes adequate laboratory controls and appropriate quality assurance procedures.

Remember: The more effort you put into your laboratory QA/QC manual, the more reliable and defensible the analytical data will become.

Definitions:

Batch (Lab)

One or more samples analyzed at the same time for the same parameter.

Batch (Lot)

The quantity of product produced at one operation. Also known as a batch lot.

Chain-of-Custody

A record of each person involved in the possession of a sample from the person who collected the sample to the person who analyzed the sample in the laboratory and to the person who witnessed disposal of the sample.

Discharge Monitoring Report (DMR)

An EPA uniform national form, including any subsequent additions, revisions, or modifications for the reporting of self-monitoring results by permittees.

Document

Any written, recorded information that is subject to change over time. Procedures, plans, policies, and records are documents. Documents may be controlled.

Duplicate samples

Samples that are separate samples taken from the same source at the same time. These samples provide a check on sampling equipment and precision techniques.

Flow-Paced Composite Sample

A composite sample consisting of a mixture of aliquots (a minimum of eight grab samples) continuously collected proportionally to flow during an 8-, 16- or 24-hour period (dependent upon the permit requirements).

Flow-Weighted Composite Sample

A composite sample consisting of a mixture of individual aliquots collected at a constant time interval, where the volume of each aliquot is proportional to the flow rate of the discharge.

Grab Sample

A single sample collected at a particular time and place which represents the composition of the waste stream only at that time and place.

Records

A completed document that provides objective evidence of an item or process and is not subject to change over time – unlike a document. Records may include log book entries, MORs, DMRs, bench sheets, photographs, drawings, magnetic tape, or other data recording media. See documents.

Replicate

A sample that has been divided into two containers and analyzed for the same parameter.

Spiked samples

Spiked samples are samples to which a known quantity of substance has been added. They provide a way to verify the accuracy of the analytical procedures.

Split sample

A sample that has been divided into two containers for analysis by separate laboratories. Analysis of these samples provide an excellent means of identifying discrepancies in the permittee's analytical techniques and procedures.

Standard Methods

A joint publication of the American Public Health Association (APHA), American Water Works Association (AWWA), and the Water Pollution Control Federation (WPCF) which outlines accepted laboratory procedures used to analyze the impurities in water and wastewater.

Standard Operating Procedures (SOPs)

A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps that is officially approved as the method of performing certain routine or repetitive tasks.

Time-Weighted (Sequential) Composite Sample

A composite sample consisting of a mixture of equal volume aliquots collected at a constant time interval.

It is essential that all laboratories, analyzing wastewater compliance samples, adhere to defined **quality assurance** procedures. Laboratory **quality assurance** is "the total program for ensuring data reliability by using administrative procedures and policies to evaluate and maintain the desired quality of data"[1]. To accomplish these goals, each laboratory must implement a quality assurance program[2]. This program should be tailored to your facility, be concise, easy-to-understand and be approved by the Department of Environmental Services (DES). It should also be made available to all appropriate facility personnel.

At a minimum, the following items must be addressed. Each item listed below is an integral part of the quality assurance program and must be outlined in a written QA manual.

A) Laboratory Water Quality.

[1] NPDES Compliance Monitoring Inspector Training Manual; Laboratory Analysis, September 1988.
[2] Pursuant to Title 40 of the Code of Federal Regulations, Part 122.41(e).

B) Reagent Quality.
C) Quality Control.
D) Sample Collection Procedures.
E) Sample Handling Procedures.
F) Instrument or Equipment Calibration.
G) Analytical Procedures.
H) Data Manipulation and Record Keeping.
I) Preventive Maintenance Procedures and Schedules.
J) Corrective Action Contingencies.

Following is an elaboration of the above items. Note that the entire monitoring program, from sampling to Discharge Monitoring Report (DMR), **must be documented** and easily verifiable.

Laboratory Water Quality[3,4]

Laboratory pure water is used for rinsing, diluting, making standards and media. It may be purchased in batch lots from a reputable source or prepared in-house. At the end of this section, there's a lab water flow chart for guidance.

A) Prepared In-house: The quality of this water must be tested to assure it meets minimum requirements of the test being performed.

1) General Reagent Water

a) The tests listed below are to be performed on general reagent water used for rinsing, dilutions and making standards (not agar, urea substrate or BOD dilution water – see Section 2).

Tests	Monitoring Freq.	Limit
Conductivity	monthly	<2 umhos/cm @ 25°C
Chlorine	daily	< detection limit

Total chlorine residual must be analyzed to insure that the lab water used for Biochemical Oxygen Demand (BOD_5) and total chlorine residual analyses are acceptable. The residual value obtained from the chlorine test should be less than the detection limit (generally in the range of 0.00-0.05 mg/L).

b) Reagent water used in microbiological analyses as well as general use.

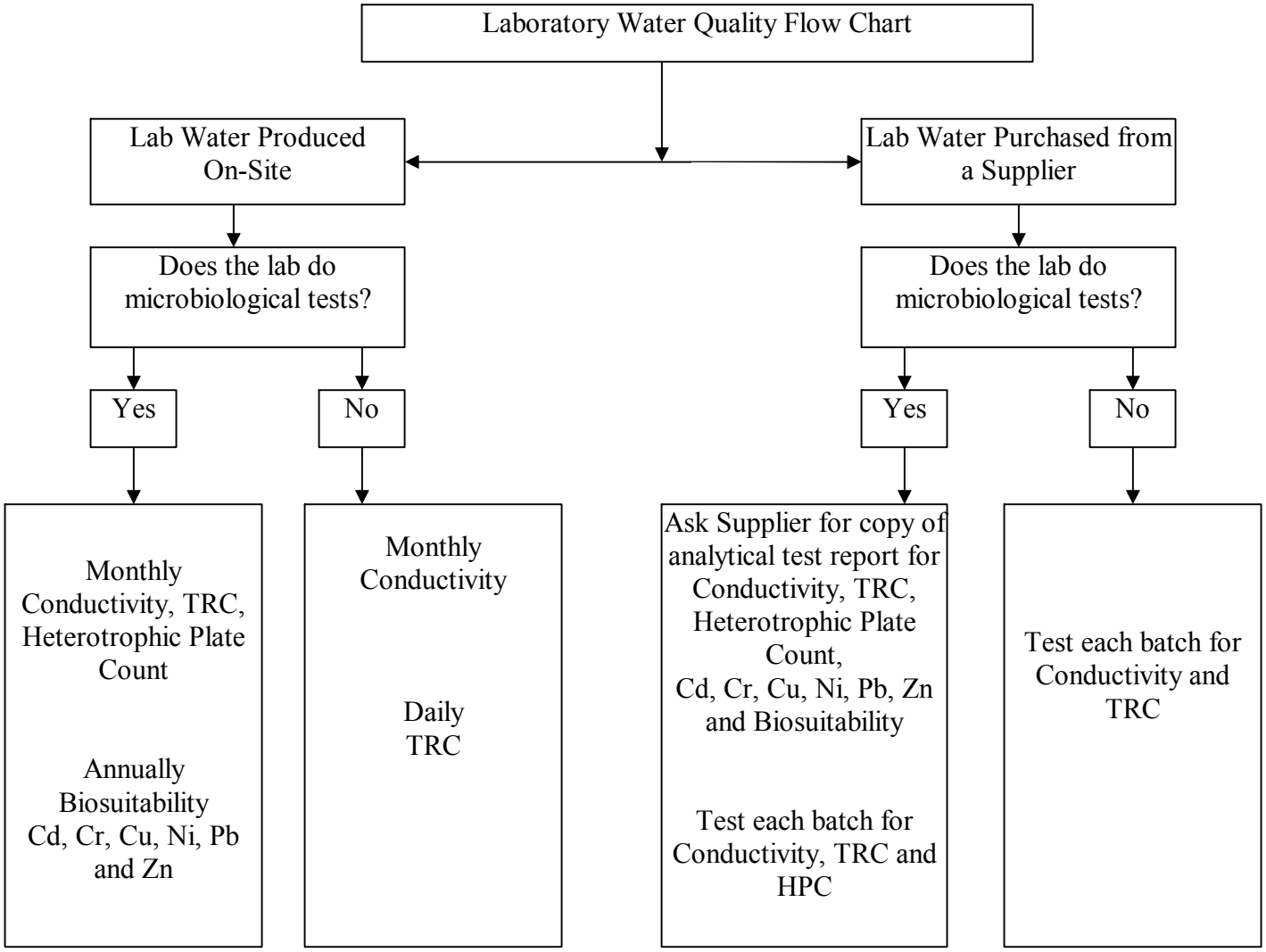
Tests	Monitoring Freq.	Limit
Conductivity	Monthly	<2 umhos/cm @ 25°C
T. Residual Chlorine	Monthly	< detection limit
Heavy Metals, single[5] (Cd, Cr, Cu, Ni, Pb, Zn)	Annually	<0.05 mg/L
Heterotrophic Plate Count	Monthly	<500 CFU/mL
Water Quality Test[6] (Biosuitability)	Annually	0.8 - 3.0 ratio

[3] Manual for the Certification of Laboratories Analyzing Drinking Water, EPA/570/9-90/008, April 1990.
[4] See Standard Methods 19th edition, Section 1080
[5] Totally, no greater than 0.1 mg/L
[6] See Standard Methods 19th Edition, Section 9020B 3(c), for more details.

B) <u>Purchased Reagent Water</u>:

1) If the facility purchases a batch lot (all containers have the same package lot number) of reagent water, you must ask the supplier to submit a copy of the test results for your record. The quality of this water must be tested for the parameters noted above in A.1.a. and/or A.1.b.to assure it meets minimum requirements of the test being performed. The analyses are typically performed each year by the supplier and the facility must have the most recent analytical report kept on file. **If there are parameters not tested by the supplier, then the facility must test the water for those missing parameters described above.**

Attach a copy of your purchase invoice with the analytical report.


Laboratory Water Quality Flow Chart
Lab Water Produced On-Site
Lab Water Purchased from a Supplier
Does the lab do microbiological tests?
Does the lab do microbiological tests?
Yes
No
Yes
No
Monthly Conductivity, TRC, Heterotrophic Plate Count
Annually Biosuitability Cd, Cr, Cu, Ni, Pb and Zn
Monthly Conductivity
Daily TRC
Ask Supplier for copy of analytical test report for Conductivity, TRC, Heterotrophic Plate Count, Cd, Cr, Cu, Ni, Pb, Zn and Biosuitability
Test each batch for Conductivity, TRC and HPC
Test each batch for Conductivity and TRC

Reagent Quality

A) Reagents are the chemicals, liquid and solid, used in the laboratory.

1) Reagents usually display an expiration date on the box or bottle. Expired reagents must not be used.
2) As part of the reagent inventory control, received and opened dates should be recorded on the box or bottle. This can be helpful should trouble-shooting become necessary.
3) Use only the highest quality chemical reagents unless a particular method states otherwise. Only order chemicals for which the American Chemical Society (ACS) has published specifications in the "ACS grade." If ACS grade is unavailable, order chemicals that are "analytical reagent grade".[7]

Quality Control

"Documentation is a system that produces unequivocal, accurate records that document all program activities." Documentation is a general term referring to all printed information recorded periodically as part of the laboratory's recording keeping and reporting conditions specified in all NPDES permits.

"Quality Control is the routine application of procedures to control the accuracy and precision of sampling and analytical measurement process(es)."[8] A minimum laboratory quality assurance program must include quality control for each parameter as described below.

QC Data

QC data are the results of analyses done on QC standards, sample duplicates, spikes and blanks. With each batch (one or more samples analyzed at the same time for the same parameter) of analyses, the following tests must be run:

1) One spike or QC standard

2) One sample duplicate

3) One blank on water

A) QC Standards

1) QC standards are substances that are a known concentration or value; a "known" quantity. They are used to determine the accuracy of sampling and analysis.

2) For each batch of samples analyzed, one QC standard must be performed.

   a) If the constituents of the waste stream analyzed vary in type or strength, spikes must be analyzed with each batch, rather than QC standards.

3) The QC standard must be of the same chemical constituents, and be between the lowest and highest calibration standards or near the expected value of the sample.

4) They are in addition to and, ideally, should be from a different source, vendor or lot than

[7] Standard Methods 18th Edition, Section 1070C, "Reagents"; Section 1070C has been removed in SM 19th and 20th Editions – refer to the method for reagent quality.

[8] NPDES Compliance Monitoring Inspector Training Manual; Laboratory Analysis, September 1988.

calibration standards.

5) QC standard results should not be used to manipulate instruments or data, only recorded and compared to the known, "calculated" value.

6) As there is no readily available QC Standard for bacteria, a split of the sample must be analyzed quarterly by another laboratory, preferably certified.

7) The QC standard must be within 20% of the known "calculated" value (0.2 su for pH) or within the manufacturer's range. If the standard falls outside of this range, then the batch results are invalid and the sample result cannot be used in the DMR calculations. If desired, control charts may be used to determine the acceptable QC standard range, in lieu of the given value of 20%.

   Use the following formula to determine percent error (% error) from the known "calculated" value:

$$\frac{\text{observed value - known value}}{\text{known value}} \times 100$$

   Written explanation as to QC standard failure must be included in a log book or on the bench sheet and on the DMR, and Corrective Action Contingencies must be implemented.

B) Duplicates

1) Duplicate samples are **separate** samples taken from the same source at the same time. They are used to determine precision (consistency) of the sampling and analysis technique.

2) One duplicate must be performed with each batch of samples.

3) For composite samples, a split of the whole composite sample (replicate) may be done in lieu of a true duplicate.

4) Duplicate and sample results **must not be averaged** together. Duplicate results are not used in calculating values for DMRs. Report only the sample results.

5) Duplicates should be within 20% of your actual sample result. If the duplicate falls outside of this range, the sample value is questionable and should be rechecked for validity. Use discretion when reporting failure, as 20% may be too stringent (tight) a range for tests that yield low results. If desired, control charts may be used to determine the acceptable duplicate result range, in lieu of the given value of 20%.
   Use the following formula to determine percent error(% error) from the sample value:

$$\frac{\text{observed value - known value}}{\text{known value}} \times 100$$

   Written explanation of duplicate "failure" must be noted on a bench sheet or log book and on the DMR, and Corrective Action Contingencies must be implemented.

C) Blanks

1) Blanks consist of dilution or general lab water. Blanks are performed as a rough check of water quality and a check of the analyst's test preparation and technique.

2) Blanks must be analyzed with each batch, unless the method states otherwise. Refer to the approved method for acceptable blank values.

3) If the blank varies from the approved method accepted value, the result is questionable but not invalid if a successful QC standard was performed.

   Written explanation as to significant blank variation from approved method accepted values must be noted in a log book or on a bench sheet and on the DMR, and Corrective Action Contingencies implemented.

D) Spikes

1) Spikes are samples (i.e., wastewater) that have had a known amount of standard added to them. They are used to determine whether there is a substance in the sample that would interfere with test results.

2) For each batch of samples analyzed, one spike must be performed.

   a) If the constituents of the waste stream analyzed do not vary widely in type or strength, QC standards may be analyzed with each batch, rather than spikes.

      1) If QC standards are analyzed in lieu of spikes with each batch, **spikes must still be analyzed for all parameters once per year and when the constituents of the waste stream vary in type or strength, for example; influent change, seasonal flow change, or process change**.

3) Spike results must be within 20% (80% to 120% recovery) of the known "calculated" value. If the percent recovery is out of the 80% to 120% range, the contents of the sample may be interfering with the method of measurement for that test. If desired, control charts may be used to determine the acceptable range for recovery, in lieu of the given value of 20%.

   Use the following formula to determine percent recovery of the standard in the spike:

$$\frac{\text{observed spike value - observed sample value}}{\text{known "calculated" standard value}} \times 100$$

Written explanation as to spike failure (what is causing the interference) must be noted in a log book or on a bench sheet and on the DMR, and Corrective Action Contingencies must be implemented.

Quality Control Frequency

A) The following table summarizes QC Control Frequency for a variety of parameters.

Parameter	Permit Frequency[9]	QC Standards	Duplicates	Spikes	Blanks	
Ammonia as N	> 1/week	1/wk	1/wk		1/yr	1/ea
	≤ 1/week	1/ea	1/ea		1/yr	1/ea
BOD	> 1/week	1/wk	1/wk		1/yr	1/ea
	≤ 1/week	1/ea	1/ea		1/yr	1/ea
Cl Res (Total)	> 1/week	1/wk	1/wk		1/yr	1/ea[10]
	≤ 1/week	1/ea	1/ea		1/yr	1/ea[10]
Bacteria						
HACH/1603	> 1/week	1/qtr[11]	1/ea		N/A	1/ea
	≤ 1/week	1/qtr	1/ea		N/A	1/ea
IDEXX	> 1/week	1/qtr	1/wk		N/A	N/A
	≤ 1/week	1/qtr	1/mo		N/A	N/A
Nitrate Nitrogen	> 1/week	1/wk	1/wk		1/yr	1/ea
	≤ 1/week	1/wk	1/ea		1/yr	1/ea
pH	> 1/week	1/ea	1/wk		N/A	N/A
	≤ 1/week	1/ea	1/ea		N/A	N/A
Phosphorus (Total)	> 1/week	1/ea	1/wk		1/yr	1/ea
	≤ 1/week	1/ea	1/ea		1/yr	1/ea
Temperature	> 1/week	N/A	1/wk		N/A	N/A
	≤ 1/week	N/A	1/ea		N/A	N/A
TSS	> 1/week	1/wk	1/wk		N/A	1/wk
	≤ 1/week	1/ea	1/ea		N/A	1/ea

Sample Collection Procedures[12]

Documenting sampling procedures ensures that all parties involved in sample collection would correctly and consistently obtain wastewater samples. Whether or not the sample is valid has much to do with how, when, and where the sample was taken. An exact procedure should be written for each parameter, and should be tailored to your facility and plant conditions. Sampling procedures (better known as Standard Operating Procedure or SOPs) should be in a written format:

---

[9] > 1/week: measurements required more than once per week, including continuous monitoring
≤ 1/week: measurements required once per week or less than once per week (bi-monthly)
1/ea: performed once each time analysis is conducted
1/yr: performed once each year or when processes or conditions change
N/A: not applicable to this parameter

[10] Must be performed when making chlorine standard solutions and in-house reagent water

[11] Split with another laboratory

[12] Standard Method 18th, 19th and 20th Editions Section 1060 B., "Collection of Samples"

1) Container descriptions
2) Cleaning procedures for sample containers
3) Volume of sample required to perform analysis
4) Preservation and storage of samples
5) Representative sampling times
6) Consistent and representative sampling locations
7) Collection techniques
8) Holding time
9) Storage requirements if not analyzed immediately

Sample Handling Procedures

Once a sample has been collected, it must be transported to the laboratory and analyzed within the required holding time. Items to include for sample handling procedures in a written format are:

1) Labeling of sample bottles
2) Chain of custody forms (if outside lab is contracted)
3) Date(s) and time(s) sample collected
4) Sampler(s) initials
5) Sample location
6) Type of sample (grab or composite)
7) Temperature of sample
8) Preservation and storage of samples

Instrument or Equipment Calibration

All instrument calibrations (both in-house and vendor) must be documented in a notebook.

Included in a written format:

1) Frequency of routine instrument calibration;
2) Frequency of professional instrument calibration **(only balances and thermometers require annual calibrations; calibration of all other instruments is left to the lab's discretion)**.
3) Calibration dates and the person performing the calibration.
4) Calibration procedures

Analytical Procedures (Standard Operating Procedures (SOPs))

A simplified, step-by-step procedure outlining the permit parameter analyses will help your analyst follow correct steps during testing. The currently approved version of Standard Methods or EPA method (40 CFR 136 lists the approved methods) may be used as a starting point and should be available for reference, but the goal here is a concise, easy-to-read procedure.

It is acceptable to have a copy of simplified procedures, but they must reference approved procedures and exactly reflect the manner in which you actually perform the test. Purchased or acquired procedures should be modified to reflect the actual procedures at a facility. Even though this type of written procedure is acceptable, it is preferred that they be written by persons that perform the tests at the facility. Please include in a written format:

1) Cleaning procedures for lab glassware
2) Preparation of any reagents needed
3) A brief description of the test to be performed - SOPs
4) The correct procedure
5) Reference method number

Data[13]

Once data has been correctly obtained, care should be taken to ensure proper documentation of the numbers. It is important to make sure that correct significant figures and units of measure are used. Discharge Monitoring Reports (DMR) must be generated and postmarked by the 15th of the month following the completed reporting period. In addition, the DMRs should be cross-checked for accuracy by at least one other person.

Include in written format:

1) A rule to ensure consistency in rounding off numerical results.
2) A chart or table that includes multipliers, divisors, and tips for changing units.
3) A procedure on how to handle invalid results in the lab and on the DMR cover letter.
4) Transcription and calculations check system to include bench sheets, lab books, and DMRs.
5) Complete DMR correctly.
6) Include correct address (and section) of federal, state and local agencies for DMR submittal.

Preventative Maintenance Procedures and Schedules

All instruments for measurement and analyses have operation and maintenance manuals. Reference these manuals for instrument maintenance schedules, and follow their instructions. These manuals are available through the manufacturer and the distributor of the product (if they are not included with the product). Usually a customer service number is listed with or on the packaging. All adjustments and maintenance must be performed and documented to guarantee the instruments are in reliable working order. Please include in a written format:

1) A regular, comprehensive maintenance schedule
2) A list of employees responsible for performing maintenance
3) A list of duties, with check-off areas to ensure completion

Corrective Action Contingencies

These contingencies will outline the course of action to be taken in the event that any areas of the quality assurance process become invalid or in question, as in the case of unacceptable QC results. They should include the required (see Part II of your permit)responses to federal, state, and local agencies by addressing what to do with unacceptable results from analyses of Quality Control standards, duplicates, blanks and spikes. Some areas you should include in your written corrective action plan are:

1) Reasons for unacceptable results
2) Estimate impact to receiving water
3) Steps to prevent recurrence
4) Will resampling and restesting be required
5) Whom to inform
6) Corrective action documentation

[13] Standard Method 18th, 19th , and 20th Edition Section 1050 B., "Significant Figures"

APPENDIX A

CONTENTS OF A QA/QC MANUAL

Contents of a QA/AC Manual

The specific items that must be included in the master QA/QC manual for your facility are:

1) Facility specific Laboratory Quality Assurance guidelines. Pages 1 through 9 of the NHDES Wastewater Engineering Bureau - Permits & Compliance Section's Wastewater Laboratory Quality Assurance Program Guidelines may be used as an outline.

2) Sample collection procedures.

3) Sample handling procedures.

4) Sample analysis procedures - SOPs.

5) A written outline of how often calibration standards, quality control standards, spikes, blanks and sample duplicates will be performed, including QC frequency.

6) Blank copies of forms used for recording data, incubation times, initials, temperatures etc. (i.e. bench sheets).

7) Data manipulation rules.

8) All maintenance and professional calibration schedules and procedures.

9) Corrective action contingencies.

10) Other items specific to your facility that would be included to assure data quality.

APPENDIX B

EXAMPLES OF PROCEDURES

The following are examples of procedures and bench sheets for an imaginary wastewater facility. Do not use these examples for your facility unless they reflect the exact conditions of your plant. Several of the examples are not complete and are only intended to help you through the more difficult parts of developing your own QA Manual.

Example of a SAMPLE COLLECTION PROCEDURE

Settleable Solids

1. Container Description – plastic is allowed. Use a 5 gal. plastic bucket.

2. Cleaning Procedure - wash with soap and water and rinse clean

3. Volume of Sample - 1000 mL required minimum volume, therefore grab a little over 1000 mL

4. Preservation Techniques - none required if analyzed immediately. If sample cannot be analyzed immediately, refrigerate at 4°C.

5. Representative Sampling Times - morning, 7 days per week

6. Sampling Location - end of chlorine contact chamber

7. Collection Technique - single grab sample; lower bucket into effluent approximately 1/2 depth of chamber and let fill. Avoid large floatable particles. Pull bucket out of the waste stream, being careful to ensure that the 1000 ml+ volume is retained. Carry to the lab.

8. Holding Time - Sample must be analyzed within 48 hours of the grab.

Example of an on-site SAMPLE HANDLING PROCEDURE

Settleable Solids

1. Labeling of Sample Bottles = N/A, bucket as container

2. Chain of Custody = N/A, samples will always be analyzed on site and documented on the lab bench sheet.

3. Date and Time sample collected = Record date and time sample was collected and sampler's initials on the settleable solids bench sheet.

4. Date and time analyzed = Record date and time sample was analyzed and analyst's initials on the settleable solids bench sheet.

Example of an off-site SAMPLE HANDLING PROCEDURE

METALS

Whenever samples are shipped off-site to be analyzed the following procedure must be followed.

1. Label the Sample bottles with the following information:

   a) date and time
   b) location of sample taken
   c) preservative
   d) list of analytes
   e) in-house sample number
   f) sample type
   g) facility name

2. Chain-of-Custody - Obtain a blank form from the contract lab and make sure it is complete. Send it along with the sample. If the contract lab does not have forms, use the one included herein.

3. Date and Time Sample Collected - Record date and time sample collected on the chain-of-custody form or if in-house, lab bench sheet.

Example of Chain 0f Custody Form

NH DES LABORATORY SERVICES LOGIN AND CUSTODY SHEET

(Laboratory Policy: Samples not meeting method requirements will be analyzed at the discretion of the NH DES Laboratory.)

LAB ACCOUNT (Billing) _____ One Stop Project: _____ NHDES Site Number_____
Description:_____ Town:_____ Temp. °C._____
Collected by:_____ Contact & Phone# _____

Sample Location / Station ID	Date/Time Sampled	# of Containers	Matrix											Misc ID	*Sampler Comments* (RESIDUAL CHLORINE LEVEL)	Lab Login #

Relinquished By_____Date and Time_____Received By_____

Relinquished By_____Date and Time_____ **Received For Laboratory By_____**

Matrix: A= Air S= Soil AQ= Aqueous (Ground Water, Surface Water, Drinking Water, Waste Water) ❒Other: _____

Section No.: 22.0
Revision No.: 5
Date: 11-2-06
Page 1 of 1

Page _____ of _____ Data Reviewed By_____ Date_____

I:\wlocke\New login sheet 1103\Login Form Rev 11 06 rev 5.doc3/15/07 2:01 PM

Example of INSTRUMENT OR EQUIPMENT CALIBRATION

Analytical Balance

1. Frequency of Calibration - daily before use

2. Frequency of Professional Calibration – once per year

3. Dates, Times and Analyst Performing the Work - Record all information noted on the form below with each check.

4. Calibration Procedure – At minimum, perform a monthly calibration check on the balance, by weighing a 1.000 g weight. If weight varies by +/- 0.001 g, calibrate as per manufacturer's instructions. If the calibration cannot be achieved, arrange for a professional to calibrate the instrument as soon as possible.

Date	Time	Initials	Weight Used	Weight Read	Level	Check By	Comments
1/20/91			1.000g				

Professional calibration performed by: _____

Example of an ANALYTICAL PROCEDURE

Settleable Solids
Detection Limit = 0.1 mL/L

1) Cleaning Procedures – Wash with hot water and soap and rinse Imhoff cone prior to use

2) Preparation of Reagents - none for this analysis

3) Description of Test - Settleable solids is the term applied to the material settling out of suspension within a 60 minute period.

4) Analytical Procedure –

a- Assure that the sample has not exceeded holding time of 48 hours.
b- Mix sample well before pouring.
c- Fill Imhoff cone to the 1 liter mark.
d- Set timer for 45 minutes.
e- After settling for 45 minutes, gently stir sides of cone with a glass rod.
f- Allow to settle 15 minutes longer.
g- Record volume of solids in mL/L on the bench sheet.

note: If the settled matter is not compacted, estimate actual settled volume.

5) Reference Method - Settleable Solids - Standard Methods 18th, 19th, and 20th Editions, Section 2540 F.

Example (partial) of BOD[14] Set-up Procedure

Note: Check the pH of all samples prior to set-up. The pH of sample must be in the range of 6.5 to 7.5. If not within the range, adjust the pH using either sulfuric acid (2:1 ratio) or sodium hydroxide (1.5N) solutions. It will probably only take a drop or two to adjust; use the magnetic stirrer when adding the chemicals. Record the adjusted pH readings on the BOD_5 Lab Sheet in the box next to each individual group of samples.

A) To Determine Influent Sample Volume

1) Influent Sample Expected BOD = 200 mg/L

a) Perform 3 dilutions of the sample[15]

Use the following formula to determine sample sizes:

$$\text{mL sample added to 300mL BOD bottle} = \frac{\text{(most valid depletion, mg/L)(volume of BOD bottle, mL)}}{\text{(expected BOD, mg/L)}}$$

For example:

1) 8 mL sample added to 300 mL BOD bottle = (4 mg/L)(300mL) / (150 mg/L)

2) 6 mL sample added to 300 mL BOD bottle = (4 mg/L)(300mL) / (200 mg/L)

3) 4.8 mL sample added to 300 mL BOD bottle = (4 mg/L)(300mL) / (250 mg/L)

b) Fill each sample bottle with dilution water.

B) To Determine Effluent Sample Volume

1) Effluent Sample Expected BOD = 20 mg/L

a) Perform 3 dilutions of the sample

[14] "A discharger whose permit requires reporting the traditional 5-day BOD may not use a nitrogen inhibitor in the procedure for reporting results." Federal Register; Part VIII, October 26, 1984.

[15] Standard Methods 18th, 19th and 20th Editions, Section 5210 B.

Formula:

$$\text{mL sample added to 300mL BOD bottle} = \frac{(\text{most valid depletion, mg/L})(\text{volume of BOD bottle, mL})}{(\text{expected BOD, mg/L})}$$

1) 120 mL sample added to 300 mL BOD bottle $= \frac{(4\text{ mg/L})(300\text{mL})}{(10\text{ mg/L})}$

2) 60 mL sample added to 300 mL BOD bottle $= \frac{(4\text{ mg/L})(300\text{mL})}{(20\text{ mg/L})}$

3) 40 mL sample added to 300 mL BOD bottle $= \frac{(4\text{ mg/L})(300\text{mL})}{(30\text{ mg/L})}$

b) Add an amount of acceptable seeding material[16] (see D 5 & 6 for determination of acceptable seeding material) to the 300 mL BOD bottles. The depletion attributable to the seed should be .6 to 1.0 mg/l per bottle.

c) Fill each sample bottle with dilution water.

C) To Set-up a Dilution Water Blank

1) Fill a BOD bottle with dilution water.

D) To Determine Seed Volume in a Seed Blank (seed plus dilution water)

1) Set the seed blank up as you would a regular sample.

2) Add enough seed to:

a. deplete the DO by at least 2 mg/L after 5 days and
b. insure that at least 1 mg/L DO remains in the bottle after 5 days.

3) The seed volume you should use is derived from trial and error.

All seeds are different.

a) Try the following seed volumes: 3 mL, 6 mL and 9 mL. At least one of these seed volumes should fit the above criteria (2a & 2b). If not, adjust accordingly.

4) Follow this example:

a) DO depletion of bottle with 6 mL of seed plus dilution water after 5 days.

[16] Only add seed material if the effluent sample has been disinfected.

initial DO = 8.5 mg/L
final DO = 4.0 mg/L (does this meet criteria 2a above? - yes)

4.5 mg/L (does this meet criteria 2b above? - yes)

E. To Determine Volume of Acceptable Seed in sample bottles.

1) Since the two criteria above have been met, this bottle may be used to determine how much of the seed will be needed and added to a BOD bottle to produce a depletion of 0.6 to 1.0 mg/l. The strength of the seed should be such that a small amount (1 to 2 ml) will be needed.

Use the following formula:

$$\text{mg/L DO depletion caused by 1 mL of seed} = \frac{\text{5 day depletion}}{\text{mL(s) of seed}}$$

$$\text{0.75 mg/L DO depletion caused by 1 mL of seed} = \frac{\text{4.5 mg/L}}{\text{6 mL}}$$

The value 0.75 mg/L is the depletion caused by 1 mL of seed.

Likewise, if the value had been 0.40 mg/L depletion caused by 1 mL of seed, 2 mL of seed would have to be added to the BOD bottle to achieve a DO uptake of between 0.6 and 1.0 mg/L (2mL x .40 mg/L = 0.8 mg/L).

E) To Determine Seeded Standard (standard plus seed) Volume

1) Standard Known Value = 200 mg/L

a) For our example, we have chosen a full strength standard of 200 mg/L.

b) Use the following formula to determine how much of the standard to use in the BOD bottle.

Formula:

$$\text{mL standard added to 300mL BOD bottle} = \frac{\text{(most valid depletion, mg/L)(volume of BOD bottle, mL)}}{\text{(known standard BOD, mg/L)}}$$

$$\text{6 mL standard added to 300 mL BOD bottle} = \frac{\text{(4 mg/L)(300mL)}}{\text{(200 mg/L)}}$$

c) Add 1 mL of acceptable seeding material (see D 5 & 6 for deter-

mination of acceptable seeding material) to the 300 mL BOD bottle.

d) Fill the BOD bottle with dilution water.

F) To Prepare Spike (standard plus sample plus seed)

1) The DO depletion of the standard plus the sample plus the seed after 5 days, should be in the 3 to 6 mg/L most valid depletion range. The following formula is used in an attempt to keep the depletion within this range. Since the value of the seed is known, (from Section D5 & 6) it may be added to the answer after the calculation is performed.

a. Standard Known BOD = 200 mg/L

$$\text{mL standard added to 300 mL BOD bottle for spike} = \frac{(\text{1/2 most valid depletion, mg/L})(\text{BOD bottle vol, mL})}{(\text{known standard BOD, mg/L})}$$

$$\text{3 mL standard added to 300 mL BOD bottle for spike} = \frac{(2\ \text{mg/L})(300\ \text{mL})}{(200\ \text{mg/L})}$$

b. Effluent Sample Expected BOD = 20 mg/L

$$\text{mL sample added to 300 mL BOD bottle for spike} = \frac{(\text{1/2 most valid depletion, mg/L})(\text{BOD bottle vol, mL})}{(\text{expected sample BOD, mg/L})}$$

$$\text{30 mL sample added to 300 mL BOD bottle for spike} = \frac{(2\ \text{mg/L})(300\ \text{mL})}{(20\ \text{mg/L})}$$

c. Add 1 mL of acceptable seeding material (see D 5 & 6 for determination of acceptable seeding material) to the 300 mL BOD bottle.

d. Fill bottle with dilution water.

2) Therefore, you know by using this formula that the standard (3 mL) plus the sample (30mL) plus the seed (1 mL) in the BOD bottle should have a depletion of approximately 4.6 to 5.0 mg/L (2 mg/L + 2 mg/L + 0.6 to 1.0 mg/L), and will not over or under deplete the bottle.

3) Perform the tests as follows:

a. Analyze the spike.
b. Analyze a sample with seed as normal. Put the same amount of seed in this bottle as in the spike.
c. Analyze seed controls as usual.

4) Determine the following information.

a. Depletion of standard plus sample plus seed bottle (spike). This is the direct 5 day depletion from a. above.

b. Determine expected depletion of sample in the spike by calculation as follows:

1. Find out the part of the depletion from the sample in the "sample with seed bottle" as follows:

   Total depletion from seed and sample - depletion from seed only

2. Use this result to determine the depletion per ml of sample:

$$\text{sample depletion per ml.} = \frac{\text{depletion from sample}}{\text{ml. of sample}}$$

3. Multiply the depletion per ml. of sample by the amount of sample that was put into the spike.

c. Determine the amount of standard put into the spike bottle (from pg 24, F.1.a. In this case 2.0 mg/l)

5) Calculate the percent of standard recovered as follows:

Formula:

$$\%\ \text{Recovery} = \frac{\text{Depletion of 3a above - Depletion of 3b3 above - seed depletion}}{\text{Depletion of 3c above}}$$

The recovery value (%) must be between 80% and 120% depending on the value of the sample.

Example of some DATA MANIPULATION RULES

Detection Limit - The detection limit for each procedure is at the top of the appropriate procedure - Do not use values smaller than the detection limits.

1. Calculating Results - Round data to significant figures* using these examples:

   10.55 becomes 10.6
   10.65 becomes 10.6
   10.75 becomes 10.8
   10.85 becomes 10.8

   If number underlined is an odd number, round up. If number underlined is an even number, round down.

2. Unit Conversion

   1 liter (L) = 1000 milliliters (mL)
   1 gram (g) = 1000 milligrams (mg)
   1 milliliter (mL) = 1000 microliters (uL)
   1 gallon (gal) = 3.675 litres (L)

   Units - Always carry along units when manipulating data.

   4135 mg
   - 4045 mg
   0090 mg

   $\frac{90 \text{ mg}}{100 \text{ mL}} = 0.90 \text{ mg/mL} \times 1000 \text{ mL/L} = 900 \text{ mg/L}$

3. Invalid Results - Any analysis in which the quality control standard value measured varies more than +/- 20% from the known "calculated" value, is considered invalid and must not be used in the DMR calculations. If this is the case, use all results from any resampling/retesting performed, in the calculations on the DMR.

   If there are no resampling/retesting values, or not enough to satisfy the permit requirement, a cover letter, attached to the DMR, must explain why requirements were not met. Remember, you must not use invalid results for reporting.

4. Check System - Have someone in-house review at least one calculation for each parameter from start to finish and document their initials under "check by" in the log book.

5. DMR Completion - Ensure that the reviewer reviews the DMR for transcription errors.

* Round off by dropping digits that are not significant. If the digit is 6, 7, 8, or 9 is dropped, increase preceding digit by one unit; if the digit 0, 1, 2, 3, or 4 is dropped, do not alter preceding digit. If the digit 5 is dropped, round off preceding digit to the nearest even number: thus 2.25 becomes 2.2 and 2.35 becomes 2.4.

6. Addresses

State Agency

NH Department of Environmental Services
Water Division
Permits and Compliance Section
29 Hazen Drive; PO Box 95
Concord, New Hampshire 03302-0095

Federal Agency

DMRs:

Water Technical Unit
US Environmental Protection Agency
OES4-SMR
5 Post Office Square
Suite 100
Boston, Massachusetts 02109-3912

5-day letters and other correspondences:

Ms. Joy Hilton
Water Technical Unit
US Environmental Protection Agency
OES4-3
5 Post Office Square
Suite 100
Boston, Massachusetts 02109-3912

Example of Preventative Maintenance Program

YSI DO Meter and Probe

1. Check probe for bubbles under membrane or membrane damage.

   IF YES: Change membrane according to DO meter manual and note (write in 'yes' near the date) on chart below. At this time also check probe cathode for tarnishing. Clean cathode in accordance with the manufacturer's instructions. If not able to clean, return for service.

   IF NO: Check when last membrane was installed. Replace every 4 weeks regardless of its condition.

2. If applicable, check if "red line" can be obtained (depending on what model you have).

   IF NO: Replace battery. Battery should be changed at least once per year regardless, or more often if "red line" cannot be obtained (if older model) or if you have a low battery indicator signal.

3. Address for more technical information.

Yellow Springs Instrument Co., Inc.
Customer Service Department
PO Box 279
Yellow Springs, Ohio 45387

tel. 513-767-7241

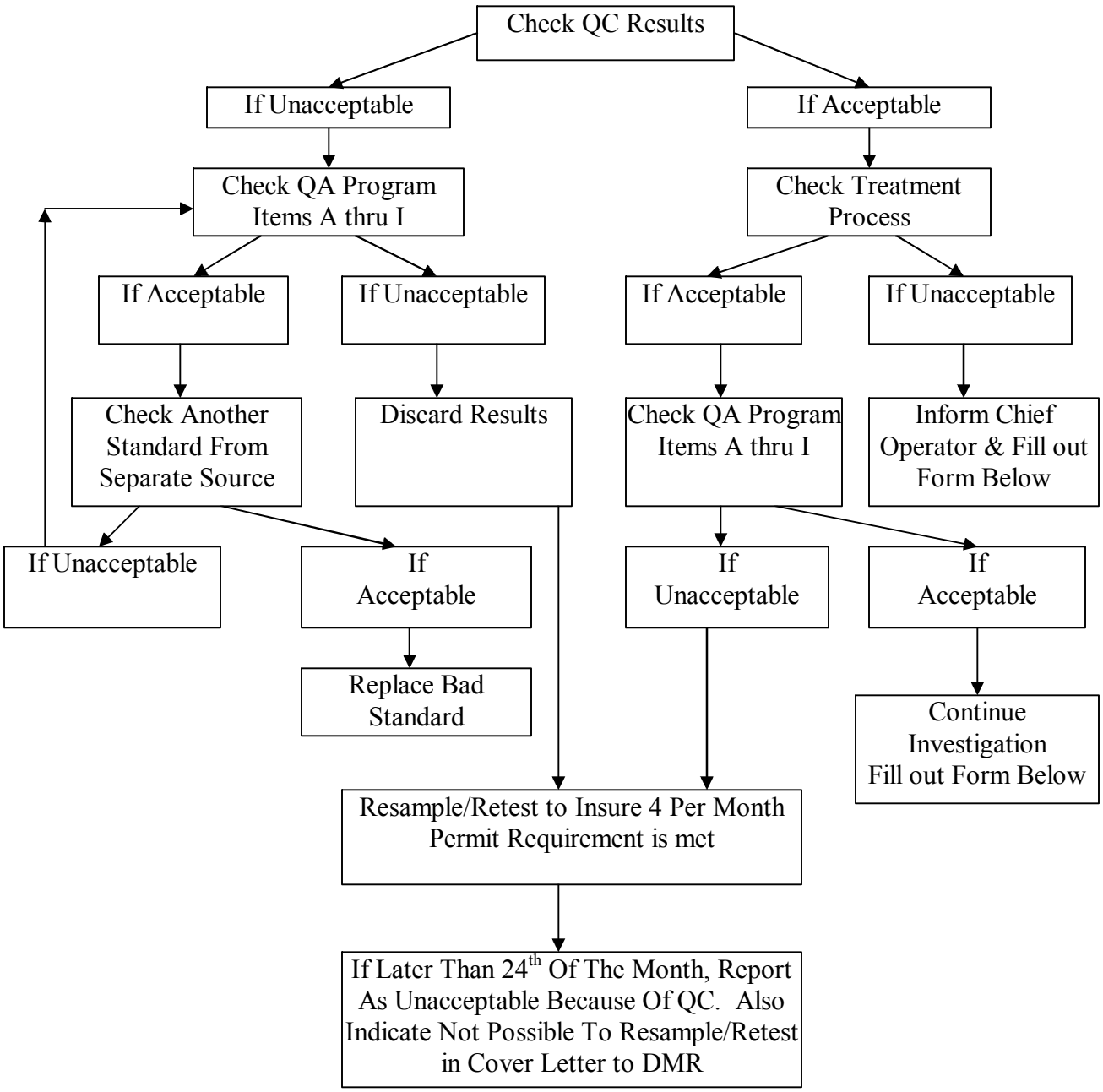
Check/Change Dates	Initials (MSG, MAO, CMB)	Comments
Jan 1/		
Jan 8/		
Jan 15/		
Jan 22/		
Jan 29/		

EXAMPLE of a CORRECTIVE ACTION/CONTINGENCY PLAN

Total Suspended Solids

1. Reasons for Unacceptable Sample Results.

Ascertain reasons per flow chart below. Fill out the included form in all cases.



2. Estimate Impact to Receiving Water:

3. Steps to Prevent Reoccurrence: Call 603-271-3503 and ask for NHDES Wastewater Engineering Bureau's Operations Section if you need assistance.

4. Who to Inform:

   Permits and Compliance Section – Contact Your Inspector

   US EPA – Joy Hilton at 617-918-1877

   Town A - (contact name and address, if receiving water used as a water supply)
   Town B - (contact name and address, if receiving water used as a water supply)

5. Corrective Action Documentation

   Problem:

   Reason:

   Action:

APPENDIX C

PREPARING SPIKES

Preparing Spikes and Calculating Percent Recovery

The following guidelines are included to assist you in understanding the general process used in the preparation of spikes, and the calculation of the percent error to determine if there are any interferences present in the wastes that will affect the test procedure. The specific case of the BOD test is covered under the example BOD set-up included in Appendix B.

SPIKE - For this guideline, a spike is defined as known standard plus sample.

A. Preparing the spike.

1. Decide approximately what you want your spike to yield for a result.

- A good rule of thumb is to use about double the value that you expect your sample to be, while remaining within the measurement limits of the test.

For example:

a. My chlorine residual is usually about .5 mg/L, therefore I want to create a spike of 1.0 mg/l. Thus, the concentration of the standard in the spike should be .5 mg/l.

2. Ascertain the volume of sample and known standard that will need to be combined to prepare the spike, as follows:

a. Choose a volumetric flask in which to prepare the spike. A 100 ml flask is usually a convenient size to work with. (If your test requires more than 100 ml, then you may wish to use a larger flask).

b. Calculate how much of the known standard that you will need to use to give the desired concentration of standard in the spike (from step 1), as follows:

standard concentration	X	volume of standard to add to the spike	=	desired concentration of the standard in the spike	X	volume of a flask in which the spike is prepared

Using our example, the calculation will be as follows: (Assume that the purchased standard is 50 mg/l).

(50 mg/l) (X) = (.5 mg/l) (100 ml)

therefore, (X) = 1 ml.

Notes:

1. The result (X) will change if your purchased standard has a different value than the 50 mg/l in our example.

2. The result (X) will change if the volume of the flask is different than the 100 ml. in our example.

3. Purchased standards that are very concentrated, such as 1000 mg/l, may need to be diluted once before introducing sample to obtain a value for (X) that can be accurately dispensed with the equipment available at the facility (pipettes, etc.).

3. Combine Standard and sample.

a. Measure the amount of standard calculated in step # 2b above into the flask that was chosen in which to prepare the spike.

b. Bring flask to volume with the sample.

- Using our example would yield the following:

  Measure 1 ml of the 50mg/l standard into a 100 ml volumetric flask. Add enough sample (99ml) to bring the flask to volume.

Once the standard and sample have been combined into the flask, this is the "spike."

B. Analyze

Note that at this time you should have two portions to analyze. One is the spike, containing sample and standard, and the other is sample only.

1. Determine the result of the spike by using the same method normally used.

2. Determine the result of the sample

- For purposes of our example, assume that the results were as follows:

  - spike = 1.1 mg/l

  - sample = .45 mg/l

C. Determine the Following Results

1. Determine the concentration of the sample in the spike with the following formula:

measured sample concentration from B 1.	X	volume of sample in spike	=	concentration of sample in spike	X	volume of the spike flask

Using our example, the calculation will be as follows:

(.45 mg/l) (99 ml) = (X) (100 ml)

therefore: .4455 mg/l = (concentration of sample in spike)

2. Determine the concentration of the standard in the spike. Known from step A and B (the amount that was decided upon and prepared).

   In our example the amount is .5 mg/l.

D. Calculate Percent Recovery.

Use the following formula to determine the recovery of the standard in the spike:

$$\frac{\text{(observed spike result)-(sample in spike ,see 1 above)}}{\text{(standard value, see A.1a.)}} \times 100$$

The recovery of the standard should be 80-120%

APPENDIX D

PREPARING STANDARDS

Preparing Standards

This guideline is included to assist you in understanding the general process used in the preparation of quality control standards of known concentrations, from purchased standards of known concentrations.

The following general concentration formula is indispensable in calculating amounts of purchased standard needed to prepare QC standards of known concentrations.

$$(C1)(V1) = (C2)(V2)$$

or

(initial concentration)(initial volume) = (final concentration)(final volume)

A. Preparing the Standard

1. Decide what value the standard will be.

   For example:

   My sample is usually about .5 mg/l, therefore the desired QC standard is about .5 mg/l.

2. Decide if the purchased standard will need to be diluted to yield the desired value from # 1 above as follows:

   Most standards are of a value that is too high to use directly, therefore it is likely that a dilution will be needed. This will be determined on an individual basis for each test and standard. For example:

   A purchased chlorine standard has a value of 45 mg/l. Most facilities will measure chlorine of less than 2 mg/l, therefore dilution of the purchased standard is needed.

3. Dilute standard to yield the desired result.

   Use the general concentration formula as follows:

$$(C1)(V1) = (C2)(V2)$$

where: C1 = concentration of known purchased standard.
V1 = volume of known purchased standard required
C2 = desired final concentration.
V2 = volume of a flask in which to dilute the standard.

From our previous examples:

C1 = 45 mg/l
C2 = .5 mg/l

Convenient volumetric flasks to use can be 50, 100, or 200 ml.

For this example a 100 ml flask will be used.

Therefore, the formula will be as follows:

$$(45 \text{ mg/l})(X) = (.5 \text{ mg/l})(100 \text{ ml})$$
$$(X) = 1.11 \text{ ml}$$

Therefore,

1. place 1.11 ml of the purchased standard into a 100 ml flask.
2. Bring to volume with lab water.

This yields 100 ml of .5 mg/l chlorine standard that can be used in the performance of QC.

It is important to note that the volume determined above (1.11 ml) may not be able to be accurately dispensed with the equipment available. If this is the case a second calculation of the formula may be needed. V1 in the second calculation would be an amount that could be measured with the equipment available, that is close to the V1 determined in the first calculation. This will result in a slightly different standard concentration than originally chosen. For example:

Assume that the equipment could measure 1.1 ml accurately, then the second calculation would be as follows:

$$(45 \text{ mg/l})(1.1 \text{ ml}) = (X)(100 \text{ ml})$$

$$.495 \text{ mg/l} = (X)$$

Therefore, 1.1 ml of standard is diluted into a 100 ml. flask to yield a standard in the range of the normal test results (Normal = about .5 mg/l, prepared standard = .495 mg/l).

Appendix E

Laboratory Bench Sheets

Year:_____ **CHLORINE RESIDUAL - BENCH SHEET, NPDES ANALYSIS**

Date	Day	Time Sampled	Sampler	Analysis Start Time	Analyst	Blank AM Setup only	(**) Standard AM Setup only	PM Final Effluent Grab	Duplicate PM Final Effluent Grab	Zero Check	Absorbance Check	Final Composite Effluent/ Eff Spike
	Sun											
	Mon											
	Tue											
	Wed											
	Thu											
	Fri											
	Sat											

** Blank and standard must be made prior to use. Standard: pipette 1 ml of chlorine standard (top shelf of refrigerator) into a 100 ml volumetric flask. Fill with DI water to the line, cover with the cap and mix by inverting 5 times. Then fill 2 cuvettes with standard and add a Cl_2 packet to one. Wait 3 minutes (set timer) before using. Blank: fill 2 cuvettes with DI water and add a Cl_2 packet to one.
Note: All samples are taken with a polypropylene bucket. Chlorine standard lab lot#:_____

Sampling Point: Final effluent weir.

Standard Method 19th Edition Section 4500-Cl G.

Year:_____ pH - Bench Sheet, NPDES Analysis

Date	Day	Time Sampled	Sampler	Analysis Start Time	Analyst	Calibration Buffers			QC* Standard	PM Final Eff. grab	Dup.PM Eff. Grab	Comments
						4.0	10.0	7.0				
	Sun											
	Mon											
	Tue											
	Wed											
	Thu											
	Fri											
	Sat											

* Hydrion is used for a pH QC standard Lot#:_____; pH 4 Lot#:_____; pH 7 Lot#:_____; pH 10 Lot#:_____

Note: All samples are grab samples taken with a polypropylene bucket.
Sample Point: Final effluent weir. Method Reference: Standard Methods, 19th Ed. Section 4500-H

BOD LAB SHEET

D.O. METER
CALIBRATED:(1,2,3) _____ SAMPLING DATE:___/___/___
(Circle no. of times calibrated)
SAMPLES COLLECTED BY:_____ on_____/_____/_____

SET-UP BY:_____ Time in:_____ Date in:_____ Inf. pH:_____
Adj:_____

READ BY:_____ Time out:_____ Date out:_____ Eff. pH:_____ Adj:_____

RESULTS CHECKED BY:_____

pH[1]	Sample Source	Bottle No.	Sample Volume Ml	% Sample	Initial D.O. mg/l	Final D.O. mg/l	Depleted D.O.[6] mg/l	5 Day BOD mg/l	Avg. Result mg/l
	Blank							H L OK	
	Blank							H L OK	
	Blank							H L OK	
	Influent		10	.033					
	Influent		8	.027					
	Influent		5	.017					
	Alphatrol[2]		5 ml	.017				Valid yes/no	
	Primary		15	.05					
	Primary		7.5	.025					
	SEED[3]		100						
	Outfall[4]		100*	.33					
	Outfall[4,5] Dup.		100*	.33					
	Outfall[4]		75*	.25					
	Outfall[4,5] Dup.		75*	.25					
	Outfall[4]		50*	.167					
	Outfall[4,5] Dup/Spike[4]		50*	.167					

* Add two (2) doses of Hach Nitrification Inhibitor to each outfall bottle. Hach N.I. Lot#:_____ Hach Nutrient Pillow Lot#:_____

% Removal =

pH[1] - adjust pH of samples if not within the range of 6.5 - 7.5.
Alpha-trol[2] - add 10 ml of 2^0 eff. seed to this bottle; Alpha-trol Lot#:_____
Seed[3] - add mls noted of 2^0 eff. seed; **Outfall[4]** - add 10 ml of 2^0 eff. seed to each outfall bottle;
Spike[5] - add 2.5 ml of Alpha-trol for spiking outfall on Thursdays
Depleted D.O.[6] - D.O. depletion for Blanks should be no more than 0.20 mg/l.

Standard Method 18th, 19th and 20th Editions Section 5210 B

Bench Sheet/NPDES Analysis
TSS Sampling Information

Sample Source And ID#	Collection		Type of Sample		Flow Prop.		Analyst	Analysis Start Time	Sampling Date	Collected By
	Date	Time	Comp	Grab	Yes	No				
Inf.-HW Bldg										
Pri. Eff. Bldg										
Outfall Weir										
Outfall – UV Eff.										

Sample Source And ID#	Blank	Alpha Trol	Outfall #1	Outfall #2	UV Eff.	Pri. Eff.	Inf.	MLSS #1	MLSS #2	RAS	WAS	
Final Wt. #2												
Final Wt. #1												
Dry Paper Wt. #2												
Dry Paper Wt. #1												
Dry Solids Wt.												
ml of sample	100	100	500	500	500	250	250	25	25	25	10	
Multiplier	10,000	10,000	2,000	2,000	2,000	4,000	4,000	40,000	40,000	40,000	100,000	
TSS Wt. Mg/l												

Alpha-trol Lot#:_____ Alpha-trol acceptable limits:_____ - _____ mg/l Alpha-trol TSS: _____mg/l

Test Valid? Yes or No

Inf TSS _____ Outfall TSS _____ % Removal _____ % Removal = $\frac{\text{Influent - Outfall x 100}}{\text{Influent}}$

1,000 mg = 1 gram TSS, mg/l = Sample weight, mg x $\frac{\text{1,000 ml/l}}{\text{ml Sample}}$

Standard Method 18th,19th and 20th Editions Section 2540 D

Method 1603 - e. Coli Bench Sheet/NPDES Analysis

Sample Source	Collected By		Sampled By	Sample Type	Analyst	Filtering Start Time	Incubation Start Time @ 35.5 °C	Incubation Start Time @ 44.5 °C	Incubation End Date & Time
	Date	Time							
Outfall - UV Eff.				Grab					

	Blank	Dilution #1	Dilution #2	Dilution #3	Dup.	Comments
Number of Colonies						
Sample Size, mL						
Counts/100 mL						

Notes:

1/quarter, send a split sample to another lab: _____ in-house: _____

Low incubator temp: _____ time checked:_____ Checked by: _____

High incubator temp: _____ time checked:_____ Checked by: _____

Results = $\frac{\text{number of colonies}}{\text{volume of sample in mL}}$ x 100 = _____ cnts/100 mL

EPA Method 1603

HACH mColiBlue-24 – e. Coli Bench Sheet/NPDES Analysis

Sample Source	Collected By		Sampled By	Sample Type	Analyst	Filtering Start Time	Incubation Start Time @ 35.5 °C		Incubation End Date & Time
	Date	Time							
Outfall – UV Eff.				Grab					

	Blank	Dilution #1	Dilution #2	Dilution #3	Dup.	Comments
Number of Colonies						
Sample Size, mL						
Counts/100 mL						

Notes:

1/quarter, send a split sample to another lab: _____ in-house: _____

Low incubator temp: _____ time checked:_____ Checked by: _____

High incubator temp: _____ time checked:_____ Checked by: _____

Results = $\frac{\text{number of colonies}}{\text{volume of sample in mL}}$ x 100 = _____ cnts/100 mL

HACH mColiBlue-24

Instrument: _____ Temperature Recording Sheet

Year: _____

	January	Initials	February	Initials	March	Initials	April	Initials	May	Initials	June	Initials	Comments
1													
2													
3													
4													
5													
6													
7													
8													
9													
10													
11													
12													
13													
14													
15													
16													
17													
18													
19													
20													
21													
22													
23													
24													
25													
26													
27													
28													
29													

30													
31													

Instrument: _____ Temperature Recording Sheet

Year: _____

	July	Initials	August	Initials	September	Initials	October	Initials	November	Initials	December	Initials	Comments
1													
2													
3													
4													
5													
6													
7													
8													
9													
10													
11													
12													
13													
14													
15													
16													
17													
18													
19													
20													
21													
22													
23													
24													
25													
26													
27													
28													
2													

9													
30													
31													

Appendix F

QA/QC Supplemental Requirements for use of Colilert and Enterolert

Background

The New Hampshire Department of Environmental Services has added additional quality control requirements for the Colilert and Enterolert bacterial analytical methods to supplement the Quality Assurance/Quality Control (QA/QC) provided by IDEXX Laboratories. Permittees using the IDEXX Colilert or Enterolert methods for bacteria analyses need to implement the QA/QC measures listed below and include them in the facility laboratory QA/QC document. The headings as appropriate follow the NHDES "Wastewater Laboratory Quality Assurance Program Guidelines" dated March 2004 and revised September 12, 2005.

Proper Selection of Sample Tray

a) For NPDES permits having a maximum daily discharge limit for E. coli of 406 colonies/100 ml, the Quanti-Tray/2000 must be used. As an alternative, Quanti-Tray, which provides counts of up to 200 colonies/100 ml, may be used if dilutions are run that have the capability of producing counts greater than 406 colonies/100 ml.

Laboratory Water Quality

a) Dilution water, if used, must be sterile, non-buffered and oxidant-free.

Reagent Quality

a) Positive and negative controls must be run on each lot of substrate powder.

    (1) Colilert

        (a) Cultured positive control, e.g. E. coli, and negative controls, e.g. *Klebsiella pneumoniae*, *Enterobacter aerogenes* and *Pseudomonas aeruginosa*

        or

        (b) Quanti-Cult – a positive/negative QC Colilert kit manufactured by Remel, Inc.

    (2) Enterolert

        (a) Cultured positive controls, e.g. *Enterococcus faecium* and negative controls, e.g. *Serratia marcescens* and *Aerococcus viridans*. A positive and negative control QC Enterolert kit is not available.

Quality Control

a) QC standards – None.
b) QC duplicates – One every 10 analyses.
c) QC blanks – None. If dilutions are run - one every 10 analyses.
d) QC splits – One every 3 months.

Equipment Preventive Maintenance

a) Quanti-Tray sealers

    (1) Cleaning should be done after 100 samples sealed (refer to sealer counter). See IDEXX instructions for cleaning procedures.

b) Quanti-Tray rubber insert

(1) Clean rubber insert as needed. Autoclave or clean with isopropyl alcohol or household bleach.

c) UV light

(1) Clean UV lamp lens with a soft cloth moistened with ethanol or a product recommended by the manufacturer as needed if soiled or grimed.

Troubleshooting

a) Dilution water. If using dilution water, verify sterility by adding 50 ml of dilution water to 50 ml of a double strength non-selective broth (e.g. tryptic soy, trypticase soy, trypone or nutrient broth) and incubate for 24 hours. If growth occurs use another batch for analyses.
b) Reagent quality. Verify sterility by adding substrate powder to 50 ml of a double strength non-selective broth (e.g. tryptic soy, trypticase soy, trypone or nutrient broth) and incubate for 24 hours. If growth occurs use another batch for analyses.
c) Bottles. Verify sterility by incubating one bottle with a sterile, non-selective broth for 24 hours. If growth occurs use another batch for analyses.
d) Quanti-Trays. Verify sterility by adding approximately 100 ml of a sterile non-selective broth. Incubate for 24 hours and check for growth. If growth occurs use another batch for analyses.
e) If a check on the above four items proves negative, then a systematic check of equipment is recommended.

References

Standard Methods for the Analysis of Water and Wastewater
18th, 19th, 20th and online versions
Method 9223 B
"Enzyme Substrate Test"

American Society for Testing and Materials (ASTM)
Method D 6503-99
"Standard Test Method for Enterococci in Water Using Enterolert"

Manual for the Certification of Laboratories Analyzing Drinking Water
Criteria and Procedures Quality Assurance
EPA 815-R-05-004, January 2005

NH Department of Environmental Services
WWEB Permits & Compliance Section
Wastewater Laboratory Quality Assurance Program Guidelines
Revised 9/12/05

IDEXX Laboratories Inc.
One IDEXX Drive
Westbrook, ME 04092

Remel Inc.
12076 Santa Fe Drive
Lenexa, KS 66215

SAMPLE

Wastewater Treatment Facility Name

Sample/Analysis Date:			
Sample Time:			
Sampler:			
Analysis Start Time:		Analyst:	
Removal Date:		Time Out of Incubator	
		2nd Time Out of Incubator	
Read By:			

Sample volume	100 mL 1	Duplicate 2	Blank		Comments
No. Large positive wells					
No. Small positive wells					
Results(MPN/100 mL)					

Sample Type: Grab
Sample location:
Colilert Method: Ref: SM (18th, 19th, 20th, 21st and on-line versions) Method 9223, Enzyme Substrate Coliform Test -- Approved by EPA, March 26, 2007, Federal Register Vol. 72 No 57 Page 1422
Enterolert Method: Ref: ASTM Method D 6503-99 – Approved by EPA, March 26, 2007, Federal Register Vol. 72 No 57 Page 1422

Idexx Quanti-Tray	Lot #		Exp.	
Enzyme Sustrate Powder	Lot #		Exp.	
Sample Container	Lot #		Exp.	(NA)

Sample analysis must begin within 2 hours of arrival at lab.
Sample must be taken in sterilized container. If sample is chlorinated, then sample container must contain sodium thiosulfate powder/tablet.