DEQ Quality Assurance Guidance

Guidance for Self Monitoring Laboratories (NPDES and WPCF)

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1.0 Scope and Application

This guidance document is intended to provide assistance to self monitoring laboratories as applicable to NPDES (National Pollution Discharge Elimination System) permitting and WPCF (Water Pollution Control Facilities) applicable to requirements in *Standard Methods for the Analysis of Water and Wastewater* and EPA requirements in 40 CFR Part 136. This document does not supersede DEQ wastewater permit conditions. The information is provided for information purposes only.

This guidance is <u>not</u> intended to be used as guidance for compliance under national safe drinking water regulations (e.g. 40 CFR Part 141) nor as guidance for routine groundwater or surface water monitoring.

This guidance document is provided by DEQ as a summary of some of the basic method requirements and should not be considered a complete list of requirements. All laboratories should be familiar with the requirements of the analytical methodologies. **DEQ used the analytical methods and federal regulations as reference while preparing this document. In case of a discrepancy, the actual content of regulations and analytical referenced methods will supersede this document.**

2.0 Useful Resources

- Code of Federal Regulations: <u>https://www.ecfr.gov</u>
 - Test Methods: 40 CFR <u>136.3</u>
 - Alternate Test Procedure: 40 CFR <u>136.5</u>
 - Method Modifications: 40 CFR <u>136.6</u>
 - Quality Assurance Quality Control: 40 CFR <u>136.7</u>
- USEPA Handbook for the Analytical Quality Control In Water and Wastewater Laboratories (1979): <u>https://www.epa.gov/sites/production/files/2015-06/documents/QCHandbook.pdf</u>
- USEPA NPDES Compliance Inspection Manual (2004): https://www.epa.gov/sites/production/files/2013-09/documents/npdesinspect_0.pdf
- USEPA 2017 Method Update Rule: <u>https://www.epa.gov/cwa-methods/methods-update-rule-2017</u>

3.0 Documentation Requirements

3.1 Quality Assurance Manual

It is essential that all labs analyzing compliance samples adhere to defined quality assurance procedures. This is to insure that routinely generated analytical data are scientifically valid and defensible and are of known and acceptable precision and accuracy. To accomplish these goals, each laboratory should prepare a written description of its quality assurance activities (a QA manual). The following items should be addressed in each QA manual:

- a) a Table of Contents, and applicable lists of references and glossaries, and appendices.
- b) a quality policy statement, including objectives and commitments, by top management;
- c) the organization and management structure of the laboratory
 - a. its place in any parent organization and relevant organizational charts;
 - b. the relationship between management, technical operations, support services and the quality system;

- d) procedures to ensure that all required records are retained, as well as procedures for control and maintenance of documentation through a document control system which ensures that all standard operating procedures, manuals, or documents clearly indicate the time period during which the procedure or document was in force;
- e) job descriptions of key staff and reference to the job descriptions of other staff;
- f) identification of the laboratory's approved signatories; at a minimum, the title page of the Quality Manual must have the signed and dated concurrence, (with appropriate titles) of all responsible parties including the QA officer(s), technical director(s) (however named), and the agent who is in charge of all laboratory activities such as the laboratory director or laboratory manager;
- g) the laboratory's procedures for achieving traceability of measurements;
- h) a list of all test methods under which the laboratory performs its accredited testing;
- i) mechanisms for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work;
- j) reference to the calibration and/or verification test procedures used;
- k) procedures for handling submitted samples;
- 1) reference to the major equipment and reference measurement standards used as well as the facilities and services used by the laboratory in conducting tests;
- m) reference to procedures for calibration, verification and maintenance of equipment;
- n) reference to verification practices including interlaboratory comparisons, proficiency testing programs, use of reference materials and internal quality control schemes;
- o) procedures to be followed for feedback and corrective action whenever testing discrepancies are detected, or departures from documented policies and procedures occur;
- p) the laboratory management arrangements for exceptionally permitting departures from documented policies and procedures or from standard specifications;
- q) procedures for audits and data review;
- r) processes/procedures for establishing that personnel are adequately experienced in the duties they are expected to carry out and are receiving any needed training;
- s) processes/procedures for educating and training personnel in their ethical and legal responsibilities including the potential punishments and penalties for improper, unethical or illegal actions; and,
- t) reference to procedures for reporting analytical results;

The QA plan may be a separately prepared QA document or may incorporate, by reference, already available standard operating procedures (SOPs) that are approved by the laboratory director and that address the listed items. Documentation for many of the listed QA plan items can be made by reference to appropriate sections of the laboratories SOPs, or to other literature (e.g. Standard Methods for the Examination of Water and Wastewater, 40 CFR Part 136, etc.) The QA plan must be available for analysts and for inspection by authorities.

To assist labs if QA Manual development, DEQ adapted a QA Manual template, originally developed by the Virginia DEQ, for use by wastewater laboratories in Oregon. The template (in MS Word) can be found on DEQ's webpage @ www.oregon.gov/deq/FilterDocs/QAManualTemplate.docx. To use the manual modify the sections to reflect the labs' specific practices and remove areas that may not be applicable.

3.2 Procedures

Each facility/laboratory should have a procedure manual that includes all the SOPs used for their selfmonitoring program. The facility's SOPs should cover sampling, equipment calibration and maintenance, analytical methods, quality control activities and laboratory data handling and reporting. Facility SOPs should include enough detail to use this document as a training manual for new employees.

3.2.1. Sampling Procedures

- a) Sample collection and analysis schedules for parameters specified in permit and/or other tests not covered in permit, but are used to determine plant performance.
- b) Sample collection locations.
- c) Sample types such as grab, composite or flow proportioned composites including instructions on sampler setup.
 - a. The following parameters <u>CANNOT be analyzed from a composite sample</u>. Grab samples must be used instead: pH, temperature, cyanide, total phenols, residual chlorine, oil and grease, fecal coliform (including E. coli), and Enterococci, or volatile organics.
 - b. All other parameters may be run from a well mixed composite sample.
- d) Sample handling requirements such as sampling containers, preservatives (e.g. acid, thiosulfate, refrigeration etc.), and holding time.

3.2.2. Facilities and Equipment

- a) Operating instructions for equipment such as balances, meters, incubators, and samplers etc., which outline proper calibration procedures to be followed whenever equipment is used.
- b) Maintenance schedules on major equipment, which indicate what type of maintenance is to be performed. A certified repairman should service balances annually.
- c) Cleaning procedures to be followed for each type of equipment and glassware used by the facility.
- d) Instructions for ordering chemicals and reagents used by the laboratory, dating of all chemicals when received and opened, and preparation of laboratory reagents. Reagents or standards should be labeled for identity, concentration, date prepared, expiration date and preparers initials.
- e) An adequate supply of reagent water is required. Water must be free of the analyte tested and of interferences. Water may come from distilled/deionized, distilled, or Reverse-Osmosis systems.

3.2.3. Acceptable Methods

All methods that are referenced in 40 CFR part 136.3 are acceptable for reporting to Oregon DEQ unless otherwise specified in a facility permit. The references to *Standard Methods*¹ in 40 CFR 136.3 have changed to simply reference the year the method was adopted rather than referencing

¹ Refers to the compendium method book *Standard Methods for the Examination of Water and Wastewater*, published by the American Water Works Association (AWWA) and Water Environment Federation (WEF). For more information see <u>https://www.standardmethods.org/</u>

the multiple editions that contain a given method. The change was made so EPA would not have to continue adding all of the *Standard Method* Editions in the CFR.

For example: For BOD, the current approved method is SM5210B-2011, with 2011 being the year the method version was adopted. The method version is published in the edition published following 2011 (22st edition, published in 2012 and on-line)

Where a facility wants to utilize a new method or technology that is not listed in 40 CFR part 136.3, they may apply directly to EPA Region 10 for an Alternate Testing Procedure (ATP) approval. The Approval must be submitted to the permit inspector once obtained.

EPA published the final 2017 Method Update Rule in the Federal Register on 8/28/2017 with an effective date of 9/27/2017. All references to Standard Methods will be from the 22nd or 23rd editions or Standard Methods on-line (almost all approved inorganic chemistry methods were updated in 2011).

3.2.4. Analytical Procedures

- a) Standard Operating Procedures (SOPs) should be written outlining the test procedures to be followed by all personnel. The SOPs should conform to EPA approved methods contained in Standard Methods, ASTM, EPA Methods, or other methods listed in 40 CFR part 136.3. The SOP contains all specific analytical instructions from sample prep to calculations of final result; including calibration procedures, QC procedures, dilutions etc. SOPs should reference the approved method from which they were developed and describe the test as it is performed.
 - a. **Note**: as an Option to a formal written procedure, if the laboratory follows the referenced method to the letter, another written document is not necessary or the laboratory may list their specific variances from the method and attach this to a copy of the referenced method as their written procedure.
- b) Methods which deviate from the approved method must be demonstrated to be applicable by documenting that results obtained are comparable to those obtained using the approved method.

Method Reference	Description
ASTM	American Society for Testing and Materials (ASTM), Consensus Standard Organization – <u>www.astm.org</u>
AOAC	Association of Analytical Communities, International (AOAC) "Official Methods of Analysis of the Association of Analytical Chemists, Methods Manual, Sixteenth Edition, 4 th Revision 1998 <u>www.aoac.org</u>
USGS	Unites States Geologic Survey (USGS) <i>Methods for Analysis of Inorganic Substances is Water and Fluvial Sediments</i> , Book 5 1989 <u>www.usgs.gov</u>
Standard Methods	American Waterworks Association, Water Environment Federation – Standard Methods for the Analysis of Water and Wastewater. www.standardmethods.org

c) The Department of Environmental Quality and EPA must approve all nonstandard methods in writing.

4.0 Quality Assurance / Quality Control in the CFR

<u>40 CFR part 136.7</u> outlines the QA/QC requirements for permittees/labs that perform compliance testing under NPDES. Essentially there are 3 options depending on applicability:

- a) "Refer to and follow the QA/QC published in the "equivalent" EPA method for that parameter that has such QA/QC procedures;"
 - Some of the older EPA methods or other approved methods are lacking in QC detail, if you are are citing one of these methods, you need to try and identify and locate the latest equivalent version the EPA method that contains detailed QC procedures. If none are available, then you will need to go to the third option below (c).
- *b)* "Refer to the appropriate QA/QC section(s) of an approved part 136 method from a consensus organization compendium;"
 - As an example, rather than putting the QA/QC procedures in every method, Standard Methods has specific chapters for QA/QC procedures that apply throughout the book. So citing Standard Methods references, includes the QA/QC in these chapters. Examples below.
 - i. **1020** Quality Assurance / Quality Control General QA/QC for all <u>Chemistry</u> methods (note that all of the items do not apply to all methods (e.g. matrix spiking doesn't apply to pH or BOD).
 - ii. **2020 QA/QC** Applies to 2000 series methods (e.g. SM 2540 Solids TDS, TSS, etc)
 - iii. 3020 QA/QC Applies to the 3000 series methods Metals
 - iv. **4020** QA/QC Applies to the 4000 series methods Non-Metal Inorganics (e.g. Ammonia, Chlorine, pH, Nitrate, Phosphate, etc.)
 - v. **9020 QA QC** Quality assurance procedures for 9000 series microbiology methods.
- c) "Incorporate the following twelve quality control elements, where applicable, into the laboratory's documented standard operating procedure (SOP) for performing compliance analyses when using an approved part 136 method when the method lacks such QA/QC procedures. One or more of the twelve QC elements may not apply to a given method and may be omitted if a written rationale is provided indicating why the element(s) is/are inappropriate for a specific method.
 - *(i) Demonstration of Capability (DOC);*
 - *(ii) Method Detection Limit (MDL);* (**DEQ note**: see 40 CFR 136 Appendix B for applicability)
 - *(iii) Laboratory reagent blank (LRB), also referred to as method blank (MB);*
 - *(iv) Laboratory fortified blank (LFB), also referred to as a spiked blank, or laboratory control sample (LCS);*
 - (v) Matrix spike (MS) and matrix spike duplicate (MSD), or laboratory fortified matrix (LFM) and LFM duplicate, may be used for suspected matrix interference problems to assess precision;
 - (vi) Internal standards (for GC/MS analyses), surrogate standards (for organic analysis) or tracers (for radiochemistry);
 - (vii) Calibration (initial and continuing), also referred to as initial calibration verification (ICV) and continuing calibration verification (CCV);

- (viii) Control charts (or other trend analyses of quality control results);
- *(ix) Corrective action (root cause analysis);*
- (x) QC acceptance criteria;
- (xi) Definitions of preparation and analytical batches that may drive QC frequencies; and
- (xii) Minimum frequency for conducting all QC elements.

These twelve quality control elements must be clearly documented in the written standard operating procedure for each analytical method not containing QA/QC procedures, where applicable."

4.1 Basic Quality Control Procedures for the small lab

- a) Demonstration of capability for each analyst: This should reflected by the training and initially successful analysis of known QC samples or PT samples PRIOR to running real samples.
- b) A blank analysis on water and reagents should be run with each test.
- c) A blank spike/Laboratory control sample or QC reference reference standard should be run with each batch.
- d) Calibration consisting of at least a low, midpoint and a high standard. At least one calibration check standard should be run with each set-up.
- e) Duplicate an analysis with each set-up as a precision check.
- f) A sample spiked with target analyte matrix spike should be run with each batch as an accuracy check, and to identify interferences in the sample that may affect actual measurements. For those analyses where sample spiking cannot be performed, (e.g. turbidity, TSS, Residual Chlorine, etc) a QC reference standard should be analyzed with each batch to determine accuracy.
- g) Control limits should be established for duplicates and standards, or spike samples for data verification. Examples of establishing control limits are outlined in Appendix A. Note that there may be a minimum control limit required for the methods that must be met regardless of the control limits established by the laboratory.
- h) Routinely verify quality of reagent water. Reagent water must meet the requirements of the individual analytical methods. Testing the conductivity of reagent water at least daily should be performed as a check of water quality for inorganic (and metals) parameters. Laboratories should minimally use the criteria for medium quality water, however it is dependent on data quality objectives (DQOs). Conductivity checks do not provide an indication of suitability for organic or microbiological methods. Method blanks and calibration blanks provide the final demonstration that reagent water is of sufficient quality.

Test	High Quality Water	Medium Quality Water	
Conductivity	< 0.1 µmho/cm at 25°C or > 10 megohms	< 1 µmho/cm at 25°C or > 1 megohms	

Table 1 Water Quality Check

i) Routinely check and document temperature of sample refrigerators, incubator, and composite sampler's cooler.

5.0 Laboratory Data Handling and Reporting

- a) Bound lab books or bench data sheets must be available for all tests performed. Data sheets should document all essential checks made to verify that test results are valid.
 - i. Data sheets/logbooks must be dated, initialed by analyst, legible, and written in ink. Drawing a single line through the incorrect recording should make any corrections, insertion of corrected result, initialed and dated, and contain a comment.
 - ii. Data sheets/logbooks should include calibration measurements, incubator temperature records, and the time samples were placed in and removed from incubators, ovens, etc.
 - iii. Data sheets/logbooks should include equations used for deriving final test results. This helps ensure that all analysts are using the same calculations, and allows for cross checking of results prior to reporting on DMR.
 - iv. Control limits used for standards or duplicates can be included on data sheet/logbook for easy review and verification of data.

6.0 Essential Checks for BOD5

6.1 Sample Collection

Samples are to be collected at locations specified in permits. Collect the sample in the center of the channel at 40% - 60% of the channel depth where flow is turbulent and well mixed, and the settling of solids is minimal. For sampling of influent and primary effluent, automatic samplers should have an intake velocity greater than 0.76 m/sec (2.5 ft/sec).

6.2 Sample Preservation

Keep samples cold (ice or refrigeration) to between 0° and 6° Celsius (< 10°C for microbiology methods). This includes during the 24-hour compositing.

6.3 Holding Time

The holding time for BOD5 is 48 hours after the grab sample is taken or the compositing period ends. Also see 40 CFR 136.3 Table II.

6.4 Approved Methods (40 CFR Part 136.3)

Standard Methods 5210 B – 2011 (Current editions: 22^{nd} , On-line). Dissolved oxygen (DO) is measured using either the Winkler (Azide Modification) or Membrane Electrode Method. (Note: the 2001 version is equivalent to 2011, there were only editional changes in 2011).

Or alterntively

- a) Hach Method 10360, *Luminescence Measurement of Dissolved Oxygen in Water and Wastewater* can be used in conjunction with SM 5210 B -2011 (instead of the membrane electrode). This is Per footnote 63 in 40 CFR 136.3 Table II B
- b) USGS I-1578-78
- c) AOAC 973.44

d) SM 5210 B – 2016 (23rd Edition) - New version is not CFR but would be acceptable (includes optical probe option)

6.5 Analytical Checks

6.5.1. Sample Pre-Treatment Requirements

- a) Sample pH must be between 6.0 8.0 SU.
- b) Chlorinated effluent must be dechlorinated. Use sodium sulfite (Na₂SO₃).
- c) Dechlorinated effluent must be reseeded; use aged settled influent or sample from primary clarifier for this. DO depletion from seed should be between 0.6 1.0 mg/L.
- d) Sample temperature must be approximately 20°C for tests and the initial DO should not be supersaturated (DO > 9 mg/L). Samples that are supersaturated may lose oxygen during incubation, which will result in an overestimation of the BOD. Vigorously shaking, or using aeration device similar to that used for dilution water should aerate samples with DO's greater than 9 mg/L at 20°C.

6.5.2. Test Set-up

- a) A dilution water blank must be analyzed with each setup. The 5-day blank DO depletion should be less than 0.2 mg/L. Do not subtract blank DO depletions from the sample DO depletions. Should high dilution blank DO depletion be obtained, report the results with a note (qualifier) about the QA failure.
- b) A seeded blank should be run with each setup to verify that seed strength is between 0.6 to 1.0 mg/L DO depletion. If 2 mL seed are used in the samples 2 mL of seed in the seeded blank should fall within this range. This QC measure checks to insure that the dilution water matrix is not toxic to the seed at the concentrations used and to. This QC check is referenced in SM 18th edition 5210 B 4.d.2, but was dropped in the 20th edition. The 20th edition however says that a dilution-water check and a dilution-water blank are included in the method.
- c) The seed concentration must be determined using the same procedures as any other BOD5 sample; this is the seed control. Use the value computed from this test to subtract the BOD5 of the seed from the sample. Use multiple tests of the seed at various concentrations and average BOD5 results or plot results to determine the BOD5 of the seed. The DO depletion must be greater than 2 mg/L and at least 1 mg/L DO must remain in the sample bottle. Ideally the largest seed concentration should deplete 50% of the available DO.
- d) A glucose/glutamic acid standard should be run with each setup to verify proper test performance. The concentration of BOD5 in the standard is 198 ± 30.5 mg/L. The standard requires seeding.
 - a. Glucose/glutamic acid standard is prepared from the following reagents: Dextrose, anhydrous, reagent grade: 150mg/L and L-Glutamic Acid, 150mg/L. (Note: Potassium Hydrogen Phthalate (KHP) <u>may NOT</u> be used as a substitute for glucose/glutamic acid)
- e) At least three (3) dilutions that will give a reading that meets the criteria below should be run for each sample. Standard Methods recommends that five (5) dilutions are to be run if you do not know the expected concentration range and unsure if you will get 3 readings in range. Reportable dilutions must meet the following criteria:
 - i. A minimum DO depletion of 2 mg/L.

- ii. A final DO of at least 1 mg/L.
- a. Report the average of all dilutions that meeth the criteria.
- b. If no dilution meets these requirements average the calculated results and report it as an estimate.

6.5.3. Calculation Basics

a) Unseeded BOD5 calculation:

$$BOD5 = (DOi - DOf) \times \frac{300}{v}$$

Where:

DOf = Final DO DOi = Initial DO v = sample volume in milliliters (mL)

b) Seeded BOD5 calculation

$$BOD5 = [(DOi - DOf) - \frac{(Si - Sf) \times Y}{Z}] \times \frac{300}{v}$$

Where:

Y = volume of seed added to sample (mL)
Z = volume of seed added to seed blank (mL)
Si = Initial DO from seed BOD
Sf = Final DO from seed BOD

7.0 Total Suspended Solids (TSS)

7.1 Sample Collection and Preservation

As described for BOD5

7.2 Holding Time

The holding time for TSS is 7 days after the grab sample is taken or the compositing period ends. Also see 40 CFR 136.3 Table II.

7.3 Approved Methods

Standard Methods SM 2540D - 2011 (Current editions 22nd, On-Line)

Or alterntively

- a) USGS I-3765-85
- b) ASTM D5907-07
- c) SM 2540D 2015 (23rd Edition) New version is not CFR but would be acceptable

Gravimetric, residue post drying 103 - 105°C

7.4 Analytical Checks

 a) Method Detection Limit (MDL): From statistical analysis of method blanks (at least 7). See 40 CFR 136 Appendix B

- b) Balance: a certified repairman should service the analytical balance annually. The balance should be located in an area free of drafts and sources of humidity. The balance should be on a hard, stable surface and away from vibrations.
- c) Oven temperature logs should be maintained to document oven temps. of 103° 105°C.

Note: The temperatures may also be recorded on a bench sheet (temp in / temp out)

d) Filters should be pre-washed and dried at 103° - 105°C before initial weighing.

Note: Prewashed / preweighed filter are available commercially.

- e) Filters must be stored in a desiccator prior to weighings.
- f) Choose a sample volume to yield 2.5 to 200 mg of dried solids. Filtration should be complete within 2 minutes. If it takes longer than 10 minutes to filter sample, use a larger filter or reduce volume, but do not produce less than 2.5 mg residue. Clogging of filters reduces the effective filter pore size and introduces a positive bias to TSS result.

Note: SHAKE or STIR SAMPLE WELL prior to pouring into filter.

g) Drying time of 1 hour should be verified such that the change in weight between successive weighings does not exceed 0.5 mg.

Note: The USGS method has overnight drying without the requirement to reweigh.

- h) Filter DI water through a filter and put through the procedure for a method blank.
- i) Samples should be periodically run in duplicate, as a precision check.
- j) An ASTM Certified thermometer should be available to verify accuracy of working thermometers. Thermometer checks need to be documented, and correction factors posted on equipment.

Note: a laboratory control sample can be made measuring a known amount of Diatomaceous earth into 500 or 1000 mL of water in volumetric flask. Example weigh 0.0500 mg into 10 Liter volumetric flask and bring to line with DI water = 50 mg/L TSS standard.

8.0 pH

8.1 Sample Collection

Grab samples collected at locations as outlined for BOD5 samples, or in other areas of plant for process control. Samples must be analyzed immediately in the field (within ~ 15 minute).

8.2 Approved Methods

Standard Methods 4500-H+ B-2011 (Current editions 22nd, 23rd, On-Line)

Or alterntively

- a) ASTM D1293-99
- b) USGS I-1586-85
- c) AOAC 973.41

Electrometric

8.3 Meter Specifications

a) Desirable Performance Characteristics:

Accuracy: 0.05 SU Scale Divisions: 0.1 SU Temperature Compensation: either manual or automatic Slope adjustment ability required.

8.4 Electrodes

A wide variety of pH electrodes are available. Generally a rugged full-range glass or plastic-bodied combination electrode is a good choice.

8.5 Analytical Checks

- a) Electrodes should be stored in distilled water or buffer between uses, as recommended by the manufacturer.
- b) Uncover fill hole in electrode before use.
- c) Meter should be calibrated before each use by performing a 2-point standardization with pH 7 and either pH 4 or 10 buffer. Calibrations must be documented in the lab notebook. The buffers used should bracket the pH of the samples. Test the third buffer using it as a QC check. Record the QC but do not adjust the calibration with the third buffer.
- d) The sample temperature should be recorded when pH is measured. pH readings should be normalized to 25°C. Most pH meters have built in temperature compensation, if not you will have to manually adjust the meter during calibration.
- e) The time required to obtain a stable reading is dependent on the electrode and whether the sample is being stirred during measurement. A rule of thumb would be at least 1 minute to obtain a stable reading.

9.0 Total Residual Chlorine

9.1 Sample Collection, Preservation and Handling

Grab samples are to collected at discharge from chlorine contact chamber. Samples must be analyzed immediately.

Also see 40 CFR 136.3 Table II.

9.2 Approved Methods

Standard Methods 22nd or 23rd, editions or Standard Methods On-Line

Iodometric Method I (SM 4500-Cl B 2011) or II (SM 4500-CL C 2011) Amperometric Titration (SM 4500-CL D 2011) Amperometric Low level Titration (SM 4500-CL E 2011) DPD Ferrous Titration (SM4500-CL-F 2011) DPD Colorimetric (SM4500-CL-G 2011) Historically, EPA and DEQ have approved DPD kits in special cases if they are basically equivalent to the standard methods.

Colorimetric methods should not be used if the waste stream has significant color due to inherent interferences.

9.3 Analytical Checks

- a) Method Detection Limit (MDL): From statistical analysis of low level QC samples and method blanks (at least 7). See 40 CFR 136 Appendix B
- b) A reference standard should be analyzed routinely.
- b) Reagents should be dated when received.
- c) (kits) the color wheels must be protected from sunlight when not in use to prevent fading.

10.0 Bacteria (E. coli, Coliforms, Enterococci)

10.1 Sample Collection

Grab samples are collected in sterile polyethylene or borosilicate glass bottles at discharge after disinfection.

Note: If at all possible, use the sample bottle directly to take the sample. Do not use a scoop or other transfer device as they are susceptible to contamination.

10.2 Sample Preservation

Chlorinated samples must be dechlorinated with sodium thiosulfate (Na₂S₂O₃).

Note: The addition of 1mL of a 1% Na₂S₂O₃ solution to the sample bottle prior to sterilization will neutralize up to 15mg/L chlorine.

Note: <u>Sodium sulfite (Na₂SO₃) that is used for dechlorination for the BOD5 method is toxic to gram-</u><u>negative microbes such as coliform and therefore **MUST NOT** be used for dechlorination of coliform <u>samples.</u></u>

After collection, <u>immediately</u> put the samples on ice or refrigerate at < 6°C until the time of analysis.

10.3 Holding Time

Per 40 CFR part 136.3, for compliance testing, the sample analysis must be started within 8 hours of sampling. Results MUST be qualified if the holding times are not met. If the permitee is at a location where having samples analyzed in 8 hours is not possible or impracticle, contact your permit inspector for guidance.

If the testing is not for compliance purposes, the maximum holding time is 24 hours according to Standard Methods.

10.4 Approved Methods

- a) EPA Method: "Microbiological Methods for Monitoring the Environment, Water and Wastes", 1978, EPA 600/8-78-017
- b) Standard Methods (SM) 21st, 22nd, and On-line editions
- c) Colilert®, Quanti-Tray®, Quanti-Tray/2000®, Colilert-18® (IDEXX Laboratories): E. Coli, Fecal Coliforms (Colilert-18)
- d) Enterolert® (IDEXX Laboratories): Enterococci only

e) ASTM D6503-99: Enterococci only

f) *mColiBlue-24*® (Hach Company): **E. coli only**

Table 2 Bacteria Method Information (number per 100 mL)

	Reference:	Comments	Incubation	Numeration Limits
E.Coli				
MPN	SM 9223B- 2004 (Colilert®-Quanti- Tray® and Quanti- Tray/2000®) AOAC 991.15	Colilert and Colilert-18 are approved	35°C ± 0.5° 24 hrs	From IDEXX Table
MF (single step)	mColiBlue-24®		35°C ± 0.5° 2 hrs/ 44.5°C <u>+</u> 0.2° 23 ± 1hr	From Hach Table
Fecal Coliform				
MPN (5-tube, 3- dilution method)	SM 9221 C,E- 2006	EC Medium	44.5°C ± 0.2° 24hrs ± 2 hrs	From Method Table (9221.IV) ²
	SM 9221 C,E -2006	A-1 Medium	35°C ± 0.5°3 hrs/ 44.5°C <u>+</u> 0.2° 21 ± 2hr	From Method Table (9221.IV) ²
Multiple tuble/multiple well	Colilert-18®	New Non- chlorinated only	44.5°C <u>+</u> 0.2°	From IDEXX Table
MF (<u>single step</u>)	9222 D ¹ -2006	Non- chlorinated only	44.5°C ± 0.2° 24hrs ± 2 hrs	20-60 Colonies
Total Coliform				
MPN (5-tube, 3- dilution method)	9221 B- 2006		35°C ± 0.5° 24 hrs ± 2 hrs	From Method Table (9221.IV) ²
MF (<u>single step or two</u> <u>step</u>)	9222 B- 2006	Non- chlorinated only	35°C ± 0.5° 22-24 hrs.	20-80 Colonies
MF (<u>with enrichment</u>)	9222 B B+B.5c,d- 2006	Chlorinated only.	35°C ± 0.5° 20-22 hrs.	20-80 Colonies
Entorococci				
	Entorolort® (Quanti		$41^{\circ} \pm 0.5^{\circ} C.24$	Erom IDEVV
tiube/multiple well)	Tray® and Quanti- Tray/2000®)		hrs.	Table
MPN (multiple tiube/multiple well)	92 <mark>30 D - 2007</mark>		41° ± 0.5°C 24 hrs.	From method tables

	Reference:	Comments	Incubation	Numeration Limits
MPN (5-tube, 3- dilution method)	9230 B - 2007		41° ± 0.5°C 24 hrs.	From method tables
MF (<u>single step</u>)	9230 C- 2007		41° ± 0.5°C 24 hrs.	20-80 Colonies

MF = Membrane Filter method MPN = Most Probable Number

¹ Since Chlorination stresses fecal coliforms and significantly reduces recovery, this method should not be used with chlorinated wastewater. Any decision to use this test method for stressed micro-organisms requires MF/MPN evaluations. A Modified MF technique for fecal coliforms in chlorinated wastewater may be used if parallel testing over a 3 month period with multiple-tube fermentation technique shows comparability for each site-specific type of sample.

² See Section 10.1 below

10.5 Equipment Requirements

Follow guidelines in Standard Methods Method 9030B - Equipment Specifications

- a) Incubator: water bath or aluminum block that controls temperature at:
 - a. $44.5^{\circ} \pm 0.2^{\circ}$ C, and which is equipped with thermometer readable at least to the nearest 0.2°C.
 - b. $35.0^{\circ} \pm 0.5^{\circ}$ C, and which is equipped with thermometer readable at least to the nearest 0.5° C.
 - c. $41.0^{\circ} \pm 0.5^{\circ}$ C, and which is equipped with thermometer readable at least to the nearest 0.5°C.
- b) Sterilizing Equipment (Any may be used):
 - a. Autoclave: 121°C @ 20psi
 - b. Hot air oven: $170^{\circ} \pm 10^{\circ}$ C
 - c. Ultraviolet light.
- c) Stereoscope
- d) Balance providing a sensitivity of at least 0.1 g at a load of 150g. Single pan rapid-weigh balances are most convenient.
- e) Bunsen burner, alcohol burner, or electric incinerator.

10.6 Analytical Checks

Follow guideline in Standard Methods Method 9020 - Intra-laboratory Quality Control Guidelines as well as the actual referenced methods

- a) All Micro: Incubation time and temperature requirements:
 - a. See Table 1 for incubation temperature and time requirements.
 - b. An NBS or ASTM certified thermometer is necessary to document the accuracy of the working thermometer of the incubator or water bath. Thermometer accuracy should be checked against a Certified Standard and documented semi-annually. Incubator thermometer must be graduated in tenths of a degree.
 - i. For general use: Use thermometers with graduations of at least 0.5°C (0.1°C graduations is preferred).

- ii. For incubators used above 40°C: Use thermometers with graduations of at least 0.1°C)
- iii. For 44.5°C water bath use thermometers with graduations of at least 0.2°C.
- b) All Micro: Use only sterile, nonbuffered, oxidant-free water for dilutions.
- c) All Micro: A positive control should be run monthly, or whenever a new lot of media is purchased, to demonstrate the lab's ability to obtain a positive test from a known contaminated sample. Positive controls can be purchased (E. coli), or analyze 1 ml of influent diluted to 100 ml with dilution water.
- d) All Micro: A negative control must be run with each test on dilution/rinse buffer to verify the sterility of equipment and solutions.
- e) All Micro: If a laboratory prepares its own media, records must be maintained documenting the date prepared, type of medium, lot number, sterilization time, heat exposure time, final pH, and preparer initials Membrane Filtration (MF) methods: Filters should be incubated within 30 minutes of filtration and do not hold diluted samples more than 30 minutes before inoculation.
- f) E.coli (Colilert® Quanti-Tray®): <u>Only yellow colonies that fluoresce are counted</u>. Look for fluorescence with a 6-watt, 365-nm UV light within 5 inches of the sample in a dark environment. Yellow colonies that <u>do not</u> fluoresce are positive for Total coliform.
- g) E.coli, Fecal (Colilert® Quanti-Tray®): In samples with excessive chlorine, a blue flash may be seen when adding Colilert. If this is seen, consider sample invalid and discontinue testing.
- h) **E.coli, Fecal (Quanti-Tray**®): Use Quanti-Tray 2000® for enumeration since QT2000 can read up to 2419/ml.
- i) **Fecal and Total (MF Methods):** Coliform colonies should be counted within 30 minutes from removal from incubator.
- j) Fecal and Total (MF Methods): Test should be prepared at volumes such that the number of colonies per filter is between 20 and 60 for Fecal Coliforms and between 20 and 80 for total coliforms and enterococcus. More than one aliquot volume should be run on each sample to ensure this criterion can be satisfied.
- k) Fecal (MF Method): Only the blue colonies are counted as fecal coliforms.

11.0 Calculation & Reporting of Results

11.1 Individual Sample Calculations and Reporting - MPN Methods

- a) For Quanti-Tray® and Quanti-Tray/2000® *and mColiBlue-24*® use the table provided by the manufacturer to quantitate MPN/100ml values for samples.
- b) For the 5 Tube, 3 dilution methods (SM 9221) for Fecal and Total Coliforms follow guidance below:

Use **Table 9221.IV** from Standard methods for enumeration of MPN 5 tube / 3 dilution methods. Report results as written from table (do not round further).

Table 9221.IV is based on 3 dilutions: 10ml, 1ml, and 0.1ml.

From Standard Methods: When the series of decimal dilutions is different from the table, select MPN value from **Table 9221:IV** for the combination of positive tubes and calculate according to the following formula:

$$\frac{MPN}{100mL} = MPN \ Index \ value \ from \ Table \ 9221. IV \ \times \ \frac{10}{Largest \ volume \ tested}$$

See Examples A and D below

When more than 3 dilutions are used in a decimal series of dilutions, use the results from only 3 of these in computing the MPN. To select the 3 dilutions to be used, choose the highest dilution (most diluted) that gives positive results in all 5 portions tested for that volume (no lower dilution giving negative results) and the 2 next succeeding dilutions. See Examples A-D below.

Example	10 ml	1 ml	0.1 ml	0.01 ml	Combination of Positives	MPN /100ml
А	5/5	5/5	2/5	0/5	5-2-0	500
В	5/5	4/5	2/5	0/5	5-4-2	220
с	0/5	1/5	0/5	0/5	0-1-0	2
D	5/5	5/5	3/5	3/5	5-3-3	1700
E	5/5	3/5	1/5	1/5	5-3- 2	140
F	5/5	3/5	2/5	0/5	5-3-2	140

When a case such as that shown in **Example E** arises where a positive occurs in a dilution higher that than the 3 chosen according to the rule, incorporate it in the result for the highest chosen dilution, as in **F**.

11.2 Individual Sample Calculations and Reporting - Membrane Filtration Methods

40 CFR Part 136 references both Standard Methods and EPA 600/8-78-017. However, each reference uses different procedures for calculating results when multiple sample volumes are used. The following calculation procedures were taken from the EPA document (refer to 6.e.vii. (4)), and should be used over those given in Standard Methods. Standard Methods shows an example of averaging results when all aliquots produce colonies that are below the acceptable limit. However, the larger the sample volume filtered the more likely it is accurate.

The criteria for selecting the appropriate aliquot to make the calculation are as follows:

- Use the test result that produced a number within the acceptable range of 20 to 60 colonies (Fecal Coliforms) or 20-80 colonies (Total Coliforms) See Numeration Limits in Table 2).
- If more than one aliquot yields acceptable results or none produced enough colonies (i.e. all were <20 colonies), use the larger sample volume to calculate the report value. Do not average the results as given in Standard Methods.
- If all aliquots produced numbers that exceed 60 colonies (or 80 colonies) use the smallest sample volume.

Note: All examples below use the Fecal Coliform 20-60 colony criteria for the decision point, the same rules apply for the Total Coliform methods that use 20-80 colony criteria, just use 80 as the decision point and rather than 60.

a) Both references use the following formula.

• Colonies $\frac{cfu}{100ml} = \frac{N}{V} \times 100$

Where: *N* = Number of Colonies Counted *V* = Volume of Sample filtered (ml) *cfu* = Colony Forming Units

b) **Example Equation** where only one plate count is within acceptable limits (20 - 60)

5 ml sample produced 30 counts and 10 ml sample produced 15 counts. Use the 5 ml sample because 30 colonies are within the acceptable range and 15 colonies isn't.

- FC $\frac{cfu}{100ml} = \frac{30}{5} \times 100 = 600$ FC $\frac{cfu}{100ml}$ Where FC = Fecal Coliform
- c) Example Equation where more than one plate count is within acceptable limits (20 60)

5 ml sample obtained 60 counts and 1 ml sample obtained 20 counts. Use the value obtained from the largest filtered volume (1200 cfu/100ml). The larger volume has a higher probability of being accurate

- $\operatorname{FC} \frac{cfu}{100ml} = \frac{60}{5} \times 100 = 1200 \operatorname{FC} \frac{cfu}{100ml}$
- $\operatorname{FC} \frac{cfu}{100ml} = \frac{20}{1} \times 100 = 2000 \operatorname{FC} \frac{cfu}{100ml}$
- d) **Example Equation** where ALL MF counts are below the lower limit of 20, select the value obtained from the <u>largest</u> volume sampled and report as "est" (estimated).

Sample volumes of 1, 0.3, and 0.01 ml produced counts of 14, 3, and 0 respectively. In this example the 1 ml dilution is larger than the other dilutions and is used for the calculation.

• FC
$$\frac{cfu}{100ml} = \frac{14}{1} \times 100 = 1400 \text{ est FC } \frac{cfu}{100ml}$$

e) **Example Equation** where ALL MF counts are zero, calculate the final result using the count from <u>largest filtered</u> volume and report as "<" (less than).

Volumes of 25, 10 and 1 ml of sample filtered. In this case use the 25 ml volume.

• FC
$$\frac{cfu}{100ml} = \frac{1}{25} \times 100 = <4$$
 FC $\frac{cfu}{100ml}$

f) **Example Equation** where all membrane counts exceed 60, calculate the final result using <u>smallest</u> sample volume and report as "est (estimated).

Volumes of 25, 10 and 1 ml of sample filtered with counts of 90, 100, and 70 respectively. In this case use the 1 ml volume.

• FC
$$\frac{cfu}{100ml} = \frac{70}{1} \times 100 = 7000$$
 est FC $\frac{cfu}{100ml}$

g) **Example Equation** where all membrane counts are TNTC (too numerous to count), calculate the final result using the <u>smallest</u> volume and <u>upper</u> counts limit (60) and report as ">" (greater than).

Sample volumes of 10, 5 and 1 ml all obtained TNTC counts. Use the 1 ml volume

•
$$\operatorname{FC} \frac{cfu}{100ml} = \frac{60}{1} \times 100 = > 6000 \operatorname{FC} \frac{cfu}{100ml}$$

If TNTC results are obtained, analysis should be repeated with a new sample and dilutions adjusted to allow for an accurate determination of the effluent coliform concentration.

11.3 Distribution of Bacterial Populations

Because bacterial counts often have a skewed distribution because of many low results and a few high ones, it can lead to an arithmetic mean that is much larger than the median, when averaging results over time (e.g. calculation of daily, weekly or monthly averages), use the geometric mean calculation as opposed to a straight arithmetic averaging of results. There are two ways of determining the geometric mean and they are shown below.

Use the following table to help decipher what results to use in the calculations.

Table 3 Bacteria Results to Use in Calculations

Result Type	Result Reported	Use this number in calculation	
No Colony Growth	<4	4	
# of Colonies <20	15 est	15	
Colonies between 20-60	40	40	
Colonies > 60	150 est	150	
Colonies TNTC	> 6000	6000	

11.3.1. Option 1: Calculate nth root of the product of coliform results:

$$Xg = \sqrt[n]{(Xi)(Xi)(Xi) \dots Xn}$$

Where

Xg = geometric mean n = number of data points (Xi) = individual results

Example: Five measurements for FC over a one-month period were 5 samples taken: 4, 15, 40, 150, and 6000 counts/100 ml. Calculate the geometric mean.

$$Xg = \sqrt[5]{(4) \times (15) \times (40) \times (150) \times (6000)}$$

$Xg = \sqrt[5]{2,160,000,000}$
$Xg = 74 \frac{cfu}{100 ml}$

11.3.2. Option 2: Calculate antilog of arithmetic mean of log transformed FC results:

$$Log Xg = \frac{\sum (\log Xi)}{n}$$

Where

Xg = geometric mean n = number of data points Xi = individual results

Example: Five measurements for FC over a one-month period were 5 samples taken: 4, 15, 40, 150, and 6000 counts/100 ml. Calculate the geometric mean (Log mean).

 $Log Xg = \frac{(Log 4) + (Log 15) + (log 40) + (Log 150) + (Log 6000)}{5}$

$$Log Xg = \frac{(0.60) + (1.18) + (1.6) + (2.18) + (3.78)}{5}$$

$$Log Xg = \frac{9.34}{5}$$

Xg = antilog 1.868

$$Xg = 74 \frac{cfu}{100 \, ml}$$

Note that the same result was obtained using either calculation method.

12.0 Statistical Measures

Statistical methods provide the basis for determining the precision and accuracy of data. Once precision (Appendix A) and accuracy (Appendix B) performance has been established, it can be used to verify self-monitoring data by establishing quality control limits. Data that does not meet set control limits for precision or accuracy indicate that some sort of error has occurred due to equipment failure, analyst error, or a calculation error. Identification and correction of errors improves the overall quality of the data generated by a laboratory.

Appendix A Procedure for Calculating Method Precision

1 **Definition:**

Precision is a measure of the spread in the data, or the repeatability of obtaining a given result on a sample or standard. Precision is expressed either in standard deviation (multiple results), range (difference between duplicate analyses) or relative percent difference (RPD: range divided by the mean between duplicate analyses and multiplied by 100). Range is commonly used for samples that do not vary appreciably in concentration (such as effluent BOD) whereas RPD is used for samples, which may vary in concentration several orders of magnitude (i.e. 1 - 500 mg/L) and range is dependent on concentration.

2 Calculation:

$$RPD = \frac{|Xs - Xd|}{\frac{(Xs + Xd)}{2}} \times 100$$

Where

RPD = Relative Percent Difference *Xs* = Sample measurement *Xd* = Duplicate measurement

Example: Duplicate analysis for BOD5 on an effluent sample obtained the following results: 15 and 20 mg/L.

$$RPD = \frac{|15 - 20|}{(15 + 20)} \times 100$$
$$RPD = \frac{|-5|}{17.5} \times 100$$
$$RPD = 28.6\%$$

Note that calculation utilizes the absolute value in the numerator and therefore it doesn't matter which number is greater.

3 Establishing Control Limits:

Based range (difference) between duplicate analyses:

Establishing control limits in this fashion should only be used if the waste stream is consistent with little variability in analyte concentration. If concentrations of an analyte vary significantly precision control limits should be calculated based on variation in % RPD. After approximately 20 separate measurements have been made, the average range is calculated and control limits set. To determine the average, sum all of the ranges (difference between duplicate analyses) and divide by the number of measurements. Multiply the average range by the student "t" factor of 3.27 for a 95% confidence interval to determine the control limit:

Equation

$$Rc = \left(\sum_{i=1}^{n} R \div n\right) \times t$$

Where

Rc = Range control limit n = number of data points i = individual data point t = student-t factor for n

Example:

<u>Measurement</u>	Range (difference)	<u>Measurement</u>	Range (difference)
1	5	11	6
2	8	12	3
3	1	13	4
4	0	14	8
5	7	15	5
6	2	16	9
7	6	17	4
8	1	18	3
9	2	19	0
10	4	20	1

 $\sum_{i}^{20} R = 79$ $Rc = (79 \div 20) \times t$ $Rc = (3.95) \times 3.27$ Rc = 12.9 (or 13)

Duplicate analyses that have a difference of > 13 would be an exceedance of the control limit and an indication that an error may have occurred. The error may be due to sample matrix, a non-homogeneous sample, sampling error or a laboratory procedural error.

4 Establishing Control Limits:

Based on % RPD between duplicate analyses:

Establishing control limits in this fashion can be used anytime but must be used if the analyte concentration in the waste stream is variable. After approximately 20 separate measurements have been made, the average RPD is calculated and control limits set. To determine the average, sum all of the RPDs and divide by the number of measurements.

Calculate the standard deviation (SD) of the data set. The control limit is determined by adding the average %RPD and 3xSD.

Equation 1: RPD Control Limit

$$RPDc = \left(\sum_{i=1}^{n} RPD \div n\right) + (3 \times SD)$$

Where

RPDc = RPD control limit n = number of data points i = individual RPDs t = student-t factor for nSD = standard deviation of data set

Equation 2: Standard Deviation

$$SD = \sqrt{\frac{\sum_{i=1}^{n} (RPDi - RPDa)^2}{n-1}}$$

Where

SD = Standard Deviation n = number of data points RPDi = individual RPDs RPDa = Average RPD

Scientific calculators have statistical functions for calculating averages and standard deviations. These calculations can also be performed easily with Microsoft Excel® or other commercially available spreadsheet software.

Example:

<u>Measurement</u>	<u>% RPD</u>	<u>Measurement</u>	<u>% RPD</u>
1	15.2	11	25.2
2	16.8	12	19
3	10.3	13	17.3
4	15.6	14	25
5	25.6	15	30
6	28.5	16	14.2
7	18.8	17	14.3
8	28.6	18	23.8
9	14	19	12.2
10	27.8	20	16.9

Note: Standard Deviation (SD) = 6.215

$$\left(\sum_{i=1}^{20} RPD = 399.1\right)$$

$$RPDc = (399.1 \div 20) + (3 \times SD)$$

$$RPDc = (19.955) + (3 \times 6.215)$$

$$RPDc = (19.955) + (18.645)$$

$$= 38.6\% (39\%) RPD$$

Duplicate analyses that have an RPD of > 39% would be an exceedance of the control limit and an indication that an error may have occurred. The error may be due to sample matrix, a non-homogeneous sample, sampling error or a laboratory procedural error.

Appendix B Procedure for Calculating Method Accuracy

A. Determining Accuracy

1 **Definition:**

Accuracy is a measure of how well an analytical measurement compares to the "true value". Accuracy can be determined from the analysis of a known *reference standard* or with the analysis of a *sample spike* (addition of a known amount of analyte to a sample) and is measured as percent recovery.

2 Calculation of Percent Recovery (%R):

Reference Standard

$$\% R = (\frac{Sr}{Tv}) \times 100$$

Where

%*R* = Percent Recovery *Sr* = Standard result measured *Tv* = True value of Standard

Example: A glucose/glutamic acid standard for BOD5 analysis was analyzed and a result of 185 mg/L obtained. Theoretical value of standard is 200 mg/L.

$$\% R = (\frac{185}{200}) \times 100$$

 $\% R = 92.5\%$

Sample Spike

$$\%R = (\frac{(Sp - Sa)}{Tv}) \times 100$$

Where

Sp = Spike sample result Sa = Sample Result (unspiked)

Example: A sample for an Ammonia analysis was analyzed and a result of 2.5 mg/L obtained. A known amount of ammonia (10 mg/L) was added to a second aliquot of the sample and the analyzed and a result of 11.6 mg/L was obtained

$$\%R = (\frac{(11.6 - 2.5)}{10}) \times 100$$
$$\%R = 91.0\%$$

3 **Determining Control Limits:**

The control limits for percent recovery are calculated similar to the example of %RPD control limits (Appendix A.4 Equation 1). The standard deviation (dispersion statistic) is calculated using the equation in Appendix A.4 Equation 2 except RPD is substituted with percent recoveries. Often warning limits are also established as an indication that there may be some deterioration in the process that should be evaluated at before control limits are exceed. Warning limits use the mean \pm 2SD instead of \pm 3SD used for control limits.

Control limits for percent recovery are based on variations from the mean both positive and negative whereas the RPD control limit is based on only the positive deviation from the mean.

Using the equations in <u>Appendix A.4:</u>

Control Limits for % Recovery = Average Recovery \pm 3SD Warning Limits for % recovery = Average Recovery \pm 2SD

Example: A glucose/glutamic acid standard for BOD_5 analysis obtained an average percent recovery over time of 94% with a standard deviation of 5%. The control and warning limits would be:

Warning limits = $94 \pm 2(5) = 94 \pm 10\% = 84 - 104\%$ Control limits = $94 \pm 3(5) = 94 \pm 15\% = 79 - 109\%$

Appendix C Control Charting

Control charts are an effect tool to monitor Precision or Accuracy on individual data points over time. The concept is to plot each percent recovery, RPD, Range, etc. on a chart showing the control limits (and Warning Limits) to give a visual display of performance and trends.





The dark bar is the upper control limit.



Figure 2 Accuracy Control Chart as a function of percent recovery

Inner two bars are warning limits

Outer two bars are control limits

Appendix D Example Analysis Form I

DAILY PLANT COMPOSITE

DAY: DATE:

Suspended Solids:

Oven temperature (°C): _____ (in) 5g class S weight (g): _____

_____ (out)

Row		SAMPLE: QC*		
	Filter#:			
A =	Volume (ml):			
B =	TSS Filter + Dry Solids (g):			
C =	TDS Dish + Dry Solids (g):			
D =	Filter Tare (g):			
E =	Dish Tare (g):			
	Mass diff. TSS			
F =	B - D (g):			
	Mass diff. TDS			
G =	С-Е(g):			
	TSS (mg/L) = 1,000,000 × F/A:			
	TDS (mg/L) 1,000,000 × G/A:			

<u>рН:</u>

Sample temperature (°C): _____ pH 7 buffer: _____ pH 4 buffer: _____ pH 10 buffer: _____

* QC sample for TDS is a solution of KCl, and for TSS a suspension of diatomaceous earth.

Appendix E Example Analysis Form II

DAILY PLANT COMPOSITE: BOD

DAY: DATE: Final DO date:

Unseeded BOD₅ calculation:

BOD 5 = $(DO_I - DO_F) x \frac{300}{X}$

Where:

DO_F = Final DO

DO_I = Initial DO

X = Sample volume (ml).

300 ml BOD5 bottle.

Seeded BOD₅ calculation:

BOD 5 =
$$\left[(DO_I - DO_F) - \frac{(S_I - S_F)xY}{Z} \right] x \frac{300}{X}$$

Y = volume (ml) of seed added to sample.

Z = volume (ml) of seed used in seed BOD.

 S_I = initial DO from seed BOD.

 S_F = final DO from seed BOD.

Sample/Bottle #	:	Seed (ml)	Sample (ml)	Intial DO (mg/L)	Final DO (mg/L)	BOD (mg/L)
Dilution water <0.2?	:					
Glucose Glutamic Acid	:					
Seeded Dilution water = 0.6-1.0 ?	:					
Seed control /	:					
Seed control /	:					
	:					
	:					
	:					
	:					
	:					

Appendix F Revision History.

Revision	Date	Changes	Editor
1.1	April 2001	Original Document	CLR
2.0	October 2009	Reformatted Updated Method references Extensive clarifications to microbiology requirements including Holding Time requirements.	SCH
2.1	12/15/2009	Corrected pagenation error	SCH
2.2	05/06/2010	Clarified	SCH
2.3	4/25/2013	Added 2.2.3 (Acceptable methods section). Added Note to 2.2.4. Updated throughout to reflect 2012 changes in 40 CFR part 136 references to Standard Methods.	SCH
3.0	09/13/2017	Updated Formating. Added section 2 Useful Resources, added clarification regarding what parameters cannot be from composite samples, updated all method references to reflect 2017 MUR, Added information regarding QA/QC requirements in 40 CFR 136.7 and updated some of the method specific information to reflect CFR requirements. Added more references to the CFR in general.	SCH
3.1	1/ / 2019	Editorial fixes related to 23 rd edition of Standard Methods. Modification to language related to 8 hour HT for Micro methods.	SCH