# Quality Assurance Project Plan

## City of Columbia Water Quality Monitoring as Required for Supplemental Environmental Projects (SEP)

Prepared by City of Columbia Department of Utilities & Engineering

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Project Location: Station 1 – C-001 – Gills Creek @ Garners Ferry Road

Station 2 – B-280 – Smith Branch @ North Main Street

Station 3 – C-017 – Gills Creek @ Bluff Road

## A1. SIGNATORY PAGE:

Project Manager: _____ Date: _____
Tracy Mitchell, EIT, CFM

City QA Manager: _____ Date: _____
Tracy Mitchell, EIT, CFM

City of Columbia
U&E Director: _____ Date: _____
Joseph D. Jaco, PE

SCDHEC
Bureau of Water
WQ Manager: _____ Date: _____
David Graves

SCDHEC
QA Office: _____ Date: _____
Nydia Burdick, Manager

## A2. Table of Contents

# Appendices

# Project Management

## A3. Distribution List

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## A4. Project/Task Organization

The tasks of the City of Columbia's QAPP will be to monitoring 4 parameters at 3 different S.C. DHEC established water quality monitoring stations for a period of 6 years. Concurrently, there will be Supplemental Environmental Projects occurring at various stages of completion and activity. The goal is to compare the water quality monitoring data collected during these improvement projects to the historical DHEC data at these stations. This will help determine the overall success of the projects efforts as well as indicate the current level of water quality in these areas. The following is a breakdown in general responsibility:

Project Manager / City of Columbia Staff (City of Columbia) - Will manage the project including developing and maintaining the QAPP and submitting reports to hand off to CDM Smith and EPA, per the Consent Decree Schedule.

City of Columbia QA Manager – Will oversee any potential issues and adherence to the QAPP for the duration of the project.

Access Analytical – Will perform field analysis / sampling and confirm / compile data for City reports.

Nydia Burdick (SCDHEC QA/QC) – Will review and approve the QAPP.

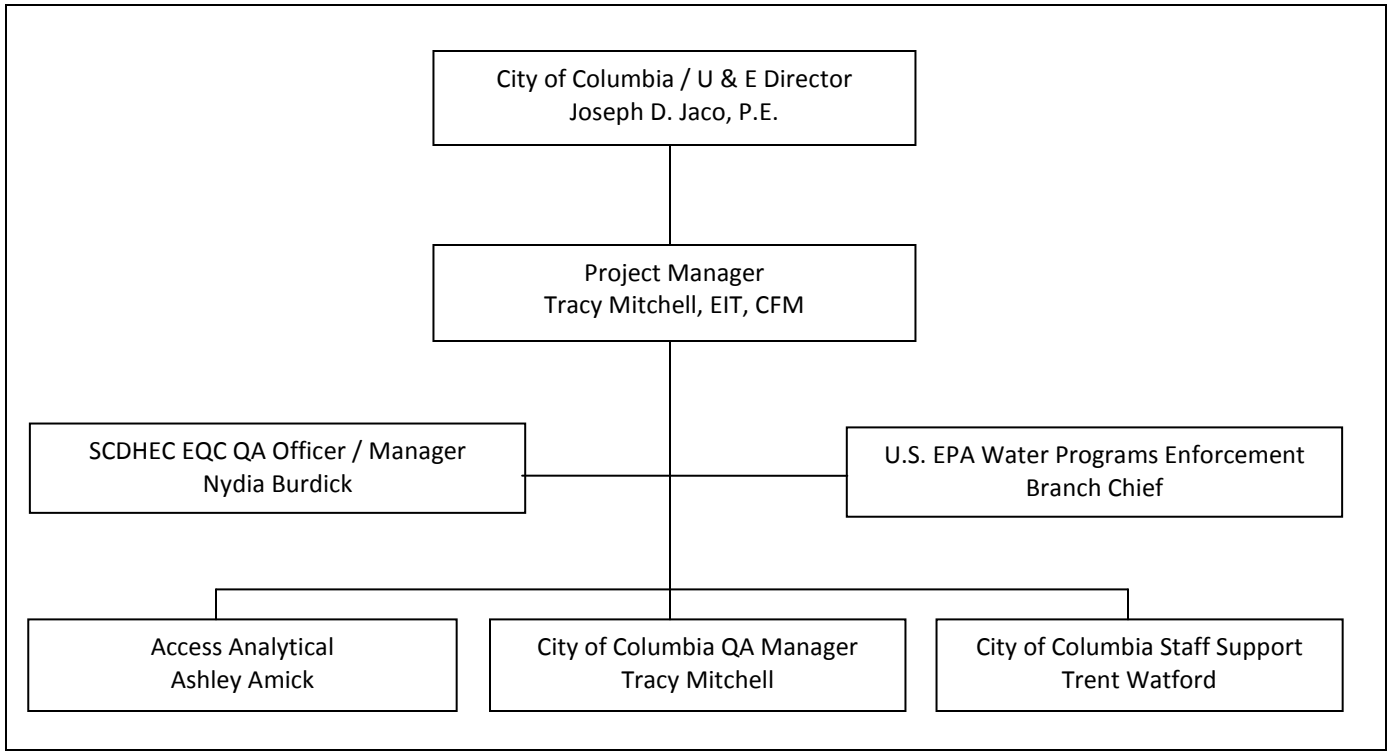


**Figure 1: Organizational Chart**

## A5. Problem Definition / Background

Effective May 21, 2014, the City of Columbia (Columbia) entered into a Consent Decree (CD) as a result of violations of the Clean Water Act through the City's Wastewater Program. Among the objectives of this CD, the City agreed to implement a program for ambient monitoring of four different parameters at the three existing monitoring stations, as requested by DHEC and EPA that correspond to Supplemental Environmental Projects (SEP). This information is being collected to comply with the Water Quality Monitoring Component of Revised Appendix I of the CD.

## A6. Project / Task Description / Schedule

**I.** Monitoring

The City of Columbia will implement a program for ambient monitoring of dissolved oxygen (DO), total suspended solids (TSS), temperature (temp) and *E. coli*[1] at the monitoring sites listed below. Columbia will conduct the monitoring in accordance with an approved South Carolina Department of Health and Environmental Control (DHEC) quality assurance project plan (QAPP). Columbia will have the TSS and *E. coli* data analyzed at a DHEC certified lab.[2] By using established monitoring sites, water quality data collected by Columbia will be available for comparison to historic water quality data taken by DHEC for assessment purposes.

Within sixty (60) days of entry of the Consent Decree (May 21, 2014), Columbia is required to submit this QAPP to DHEC for review and approval. Columbia will begin monitoring within thirty (30) days of DHEC's approval of the QAPP. As indicated below, Columbia will monitor quarterly for the first 3 years under the Consent Decree and monthly (or every other month at Site C-17) from years 4 through 6 under the Consent Decree.

**II.** Water Quality Stations (see attached map):

| Site | Description | Impairment | TMDL | Monitoring Parameters | Frequency |
|---|---|---|---|---|---|
| C-001 | Gills Creek @ Garners Ferry Road | Fecal Coliform | Yes | DO<br>*E. Coli*<br>Temp<br>TSS | Quarterly during years 1-3; Monthly during years 4-6 |
| B-280 | Smith Branch @ North Main Street | Fecal Coliform | Yes | DO<br>*E. Coli*<br>Temp<br>TSS | Quarterly during years 1-3; Monthly during years 4-6 |
| C-017 | Gills Creek @ Bluff Road | Fecal Coliform; Dissolved Oxygen | Yes | DO<br>*E. Coli*<br>Temp<br>TSS | Quarterly during years 1-3; Monthly during years 4-6 |

1 *E. coli* standard replaces the existing fecal coliform standard.
2 The temp and DO parameters measured in the field with a probe are not subject to the certified laboratory requirement but will still be collected and analyzed by a DHEC certified lab.

**Table 1: Water Quality Monitoring Stations/Sites**

NOTE: By extension of utilizing a DHEC-certified lab for the collection and analyzation of all parameters required of this QAPP, all parameters listed throughout this document will have been collected and analyzed by a DHEC-certified laboratory, whether or not that is a requirement.

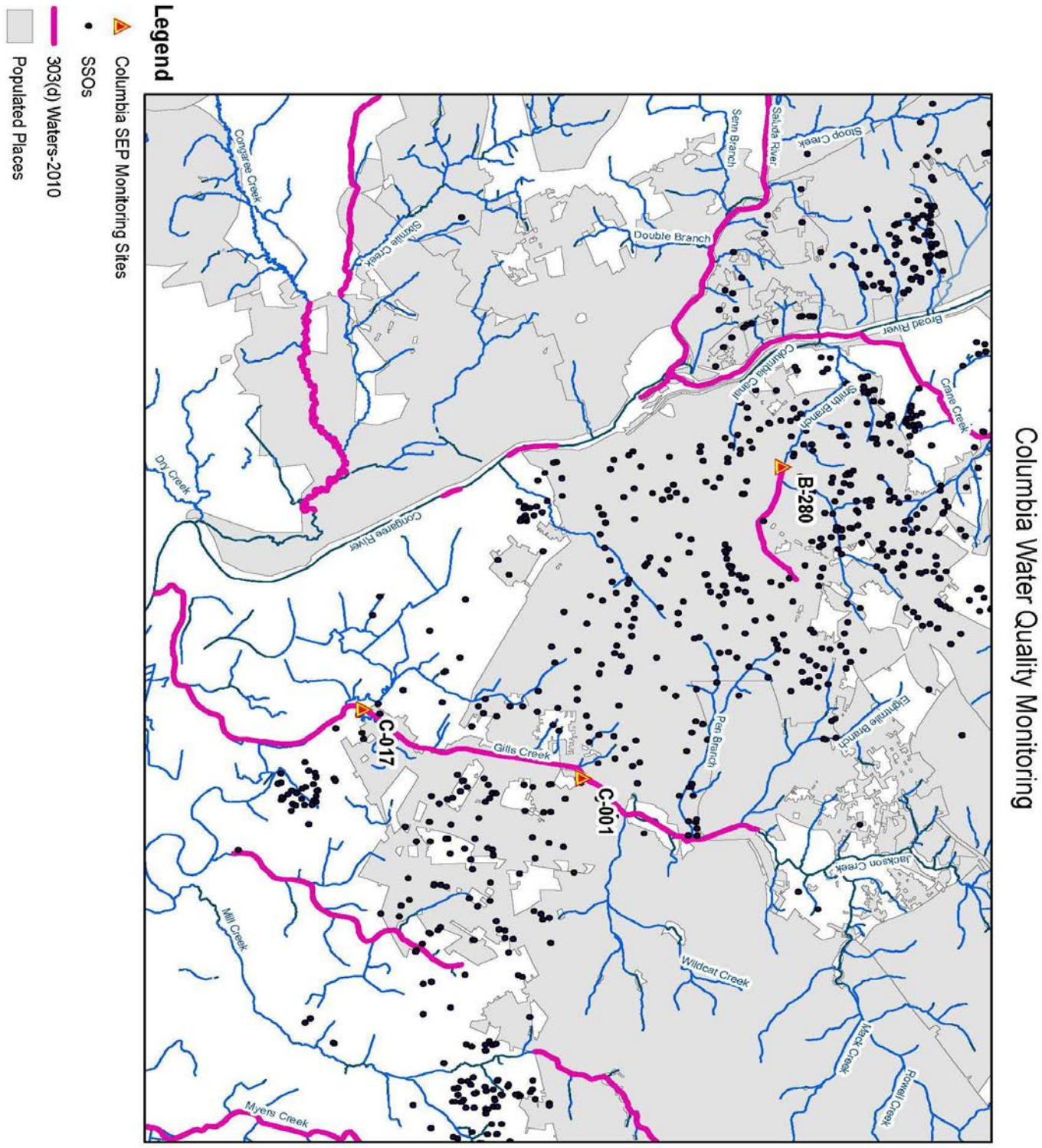


**Figure 2: Map of DHEC Monitoring Stations / Sampling Sites**

# A7. Data Quality Objective (DQOs) and Data Quality Indicators (DQIs)

## A7.1 The DQO Process

a. **State the Problem:** The problem of this project is to be compliant with the City's Consent Decree. As such, the City is to monitor four specific parameters at three established DHEC water quality monitoring stations within the Gills Creek (Gills Creek) and Broad River Watersheds (Smith Branch) for 6 years. This monitoring will be performed quarterly during years 1-3 and monthly from years 4-6.

b. **Identify the Decision-** All data collected under this plan is collected to ensure environmental compliance. By using established monitoring sites, water quality data collected by Columbia will be available to DHEC for comparison to historic water quality data taken by DHEC for assessment purposes. Ultimately, no additional sampling will occur, regardless of the results.

c. **Inputs to the Decision-** Lab and field data, in addition to historical data from DHEC monitoring

d. **Define the Study Boundaries-** The study boundaries are noted and discussed in Section A6 and Figure 2. At each sampling site within the study boundaries, water samples will be collected at a depth of 6-12 inches.

e. **Develop an analytical approach and a decision rule-** The analytical approach to this sampling effort was established by the EPA and DHEC. All data collected under this plan is done so to ensure environmental compliance with the SEP. No future efforts are planned based on the outline of this plan.

f. **Specify Limits on Decision Error**- See Section B5 for information on error-minimization strategies used in this study.

g. **Optimize the design for obtaining the data**- The quality of measurements made for the plan by the laboratory is determined by the following data quality indicators (DQIs), or characteristics: representativeness, accuracy, precision, detectability, completeness, and comparability. Specific criteria for each characteristic were established to assist in the selection of appropriate sampling and analytical protocols and to identify applicable documentation, sample handling procedures, and measurement system procedures. These DQI criteria were established based on site conditions, requirements of the project, and knowledge of available measurement systems, and were addressed whenever appropriate for the data generated.

## A7.2 Representativeness

Representativeness is a qualitative measure of the extent to which a sample acquired from a matrix describes the chemical or physical characteristics of that matrix. Sample collection, handling (e.g., splitting, preservation, storage), and measurements are all conducted according to protocols allowing for the highest degree of representativeness possible for the sample media (air, soil, water, etc.). Recording procedures are utilized which document adherence to proper protocols and maintain sample identification and integrity.

## A7.3 Accuracy

Accuracy describes the degree of agreement between an observed value and an accepted reference (true) value. It includes a combination of random error (precision) and systematic error (bias) components which are introduced in sampling and analytical operations. DQI criteria for accuracy are established through quality control limits for each parameter measured and for each analytical technique, per matrix where applicable. These objectives are assessed through the analysis of sterility checks, positive and negative culture checks, blanks, matrix spike (MS)/matrix spike duplicates (MSDs), and laboratory control samples (LCSs), as specified by the analytical method, required by the project, or generated and updated from data acquired through required quality control measurements. Nominal quality control limits for each parameter and analytical technique are specified in the analytical methods.

## A7.4 Precision

Precision is a measure of the reproducibility of an analysis under a given set of conditions, regardless of the true value of the target analyte in a sample. The overall precision of a sampling event has both a sampling and an analytical component. DQI criteria for

precision are established through quality control limits for each parameter measured and for each analytical technique, per matrix where applicable. These objectives are assessed through the analysis of MSDs (if practical), LCS duplicates (if available), field duplicates, laboratory replicates, and split laboratory samples, as specified by the analytical method, required by the project, or generated and updated from data acquired through required quality control measurements. Nominal quality control limits are specified for each parameter and analytical technique in the analytical methods.

### A7.5 Detectability

Method detectability objectives define the lowest concentration or quantities required of the measurement system for each analyte or parameter. The laboratory has established reporting limits (RLs) which are the minimum concentrations to be reported without qualification for routine laboratory conditions. Data quality indicator criteria for detectability (i.e., RLs) are established for each parameter measured and for each analytical technique. These criteria are specified by the analytical method, required by the project, or determined and updated from data acquired through required quality control measurements (e.g., the replicate analyses of samples or standards containing low concentrations of the analyte of concern).

The RL for an analyte is a function of the specific analytical procedures and can vary substantially as a result of dilutions and similar procedure modifications. In all cases, the RL necessary to fulfill data quality objectives is confirmed by laboratory measurements. Nominal RLs for each parameter and analytical technique are listed in the analytical methods and on the report of analysis.

### A7.6 Completeness

The characteristic of completeness is a measure of the amount of valid data obtained compared to the amount that was expected to be obtained under normal conditions. The amount of valid data expected is based on the measurements required to accomplish project objectives.

### A7.7 Comparability

The characteristic of comparability reflects both internal consistency of measurements and expression of results in units consistent with other organizations reporting similar data. The generation of comparable data requires operating within the calibrated range of an instrument and utilizing analytical methodologies which produce comparable results. Appropriate standard units for measurement values are utilized for each measurement system, which yields internally and externally comparable results assuming other comparability criteria are met.

### A7.8 Project DQIs

Because of the intended data uses, the general philosophy for determining the project's DQI criteria was that data quality should meet current industry standards for such measurement data. In general, measurement DQI criteria are based on the published analytical method for each parameter. Specific criteria for measurement DQIs for the analyses to be performed are summarized below.

| Parameter | Units | Accuracy[a] (LCS) | Accuracy[a] (Matrix Spike) | Precision[a] (RSD or RPD) | MDL[b] | RL[c] | Complete-ness (%) |
|---|---|---|---|---|---|---|---|
| E. coli | CFU/100ml | NA | NA | RPD≤ 200% for <150 CFU/100 ml<br>RPD≤ 100% for ≥ 150 CFU/100 ml | 1 C1 CFU/100 mL FU | *1 CFU/100 mL*<br>*if sample is not diluted* | 100 |
| Total Suspended Solids (TSS) | mg/L | 90-110% | NA | ≤5% | ≥2.5 mg to ≤200 mg | *≥2.5 mg to ≤200 mg* | 100 |
| Dissolved Oxygen | mg/L | 90-110% | NA | ≤25% | <0.3 | *<0.3* | 95 |
| Water Temperature | °C | ± 0.5°C | NA | ± 0.5°C | NA | *NA* | 95 |

LCS = laboratory control sample
MDL = method detection limit
MS = matrix spike
NA = not applicable
% R = percent recovery
RL = reporting limit
RPD = relative percent difference
% RSD = percent relative standard deviation

[a] Criteria apply to concentrations ≥ RL.
[b] For undiluted samples.
[c] For undiluted samples. If sample is diluted, RL is proportionally higher.

**Table 2: Criteria for Measurement DQIs**

## A8. Special Training Requirements and Certifications

The Certificate issued by the SC DHEC Office of Environmental Laboratory Certification for Access Analytical, Inc. is 32575001. On August 1, 2014, Access Analytical finalized acquisition of On Line Environmental and is now a wholly owned subsidiary of Access Analytical, Inc. For the time being, (it is being worked out to be one certification number for both) there are two separate SCDHEC certification numbers since the offices are currently separate. At this time,

the Access Analytical, Inc. certification number remains as state above and the On Line Environmental certification number is 32571001. The samples will be run under the On Line Environmental certification. Access Analytical, Inc. currently resides at a separate address: 7478 Carlisle Street, Irmo, SC 29063.

The generation of reliable data by a laboratory requires that all operations are conducted by knowledgeable and trained personnel. The laboratory requires the accomplishment of a prescribed sequence of training objectives by a staff member before that individual is designated as qualified and permitted to independently conduct any assignment or analyses. The indoctrination and qualification process includes as a minimum:

- Reading and understanding applicable laboratory SOP,
- Reading and understanding applicable reference documents,
- Hands-on training under the supervision of an experienced and qualified individual, and
- For analytical methods used for measurements, a successful initial demonstration of analytical capability (i.e., IDC) by performing four replicate measurements which satisfy precision and accuracy criteria for the method as well as an MDL study.

Training records for staff are maintained by the Laboratory Director or Supervisor of the lab contracted to perform the work, and training files are kept for each staff member in the training and qualification files. Lab analysts shall also collect samples and perform field measurements. A summary of training accomplishments is recorded on file on the contracted lab's premises. Otherwise, no additional, specialized training will be needed for this project. For additional information, contact the laboratory for specifics.

## A9. Documentation and Records

The QAPP will be maintained, revised, managed and facilitated by City of Columbia Staff, as listed in the Organizational Chart with the Project Manager as primary lead. S.C. DHEC's Quality Assurance Manager will review modifications pertaining to the QAPP and grant approval. Updates or changes regarding the QAPP will be e-mailed to individuals on the distribution list, unless otherwise specified. Sample collection times, field observations, and etc. will be recorded within a separate logbook by laboratory staff, as appropriate. Maps, GPS coordinates, photos, and etc. may be utilized to track progress, if necessary.

Data will be provided to the Project Manager by the lab on a quarterly basis for the first 3 years and on a monthly basis for the last three years of the project's duration. Any summaries or

comments associated with the data will be drafted and finalized by the Project Manager and provided to appropriate personnel as defined in the organizational chart for distribution to all those required to receive notification pursuant to the SEP. All those required to receive notice are listed in the distribution list at the front of this document.

All raw data and/or data reports received form the lab along with summaries and commentary will be backed up, when received, to a shared folder for staff and management to access, when appropriate. Annually, electronic records will be backed up onto an external hard drive and kept for a minimum of 10 years or as defined in the Consent Decree. Hardcopies will be bound and stored for a minimum of 10 years or as defined in the Consent Decree. All records are kept onsite.

### A9.1 Data Reporting

After completion of analyses, analysts enter results for both samples and QC measurements into the laboratory's computer-based report templates. After peer review of the data is completed and the results are acceptable, the Laboratory Director reviews the preliminary report and works with necessary laboratory personnel to make any needed corrections. A final report is then produced and submitted to the City, either electronically or by mail depending on the contract. For this project, the laboratory will forward final reports containing completed, reviewed, and approved project results to the Program Manager pursuant to the project schedule. DHEC will receive the data on a quarterly basis for years 1-3 and monthly years 4-6.

The copy of the data package provided to the City and all associated raw data are typically kept for a period of at least 10 years or as defined in the Consent Decree. These records are stored in the laboratory for approximately two years, and then transferred to a storage room for secure, long term storage. For electronic data deliverables in Microsoft Excel or similar formats, files are maintained on the laboratory's desk top computers. Backup copies of the electronic files are prepared at least annually and stored in a secure area.

Laboratory and field data for the four required parameters are the only items being collected and evaluated through this QAPP. All reports, records and electronic files from the laboratory will be supplied by the City and DHEC on the quarterly or monthly basis, as described previously.

# B. Measurement/Data Acquisition

## B1. Sampling Process Design (Experimental Design)

The DHEC water quality monitoring stations listed in the Project Schedule table will be the focus of where sampling takes place. These locations were outlined in the SEP language of the City's Consent Decree and, therefore, mandated to be the sites of collection. No explanation was

given as to why these sites were chosen, although it is assumed that since DHEC already had sites set up at these locations, it was more likely that they would be able to compare the data collected through this QAPP to the historical data on file. All samples that require analysis will be taken at the outfall of the station, with the exception of those that can be taken in the field by handheld devices and are not subject to the standards of a DHEC certified lab method.

It is not predicted that the sampling sites will ever be inaccessible for data collection. This is primarily due to the fact that these sites were originally set up to be a long-term monitoring site for DHEC and should not only have proper flow through and position in the watershed, but is easy to access for maintenance and collection.

While the set schedule has yet to be determined, the samples will be collected at the same time and day each month or quarter, depending on which year the project is in and is independent of weather. Every sample will follow the EPA method and laboratory protocol for handling and hold times in which it should be analyzed. Each sample has been guaranteed to be analyzed within their appropriate hold time(s) and will then be finalized for release of result. Once the Project Manager receives this laboratory report, the information will be provided to DHEC according to the distribution list. If a sample is destroyed anywhere in the process of collection, transport or analysis, the sample will need to be recollected in total and the occurrence should be noted with reason given. For more information on this procedure, please see Section D.

## B2. Sampling Methods

As mentioned before, four parameters will be measured on a quarterly basis for Years 1-3 and on a monthly basis for Years 4-6.

Sampling efforts will involve the collection of water samples for the following analytes: total suspended solids (TSS), *E. coli*. At the time of sample collection, in situ measurements will also be made for temperature and dissolved oxygen (DO) at each sampling location through the use of calibrated field probes (YSI).

Field measurement procedures and sample collection, handling, receiving, storage, and associated record keeping procedures are integral parts of the laboratory's QA program. The policies are designed to ensure that each measurement result and each sample are accounted for at all times. The primary objectives of measurement and sample control procedures are as follows:

- Each field measurement is recorded and uniquely identified at the time of measurement,
- Each sample received for analysis is uniquely identified,
- The correct samples are analyzed and are traceable to the applicable data records,

- Important and necessary sample characteristics are preserved,
- Samples are protected from loss, damage, or tampering,
- Any alteration of samples during collection or transport (e.g., filtration, preservation, breakage) is documented,
- Records of field measurements and sample custody (i.e., chain of custody) and integrity are established which will satisfy legal scrutiny, and
- A record of ultimate sample disposition (i.e., disposal or release from laboratory) is established.

### B2.1 Sample Collection

A summary of sample collection, handling, and preservation activities is provided in **Table 3.**

| Sample Type | Parameter Measured | Sample Container | Minimum Sample Size | Preservation Method/ Storage |
|---|---|---|---|---|
| Urban stream/ditch water, collected via grab samples | E. coli | Sterile plastic with sodium thiosulfate | 100 mL | Field: store in cooler at 1-6 °C<br>Lab: store in refrigerator at 1-6 °C and start analysis within 8 hours |
| Urban stream/ditch water, collected via grab samples | Total Suspended Solids (TSS) | plastic | 500 mL | Field: store in cooler at 1-6 °C<br>Lab: store in refrigerator at 1-6 °C and start analysis within 7 days |

**Table 3: Sample Collection Criteria**

Samples collected by laboratory personnel are placed in appropriate containers, having the required preservatives or additives, and labeled with site-specific information to uniquely identify each container at the time of collection. Conditions of sampling sites, sample IDs, number of samples, dates/times of collection, equipment calibrations, etc., are recorded on site in field logbooks or on laboratory chain of custody forms as appropriate. Unless otherwise specified, samples are stored on ice in coolers at 1-6 °C until their receipt at the laboratory. Samplers may be the Laboratory Director, Laboratory Master Technician and/or Laboratory Technicians trained in sampling. In general, samples collected are grab samples (i.e., sample collected at a specific time and place) and collected manually. For bacteria analysis, samples are collected using sterile glass or sterile plastic

sample bottles and collected carefully at just below the outfall/station so as to not contaminate by touching the inside of either the bottle or its lid. The bottle is filled with sample to approximately one-inch from the top, and then the lid is replaced. The bottle is then placed in a snap and seal plastic bag and a cooler with ice for storage and transport to laboratory. For analyses other than bacteria, samples are collected in plastic bottles. Bottles collecting samples for TSS only, is rinsed with river water at the site three times (due to lack of preservative), carefully filled with river, capped, and then placed in a cooler for storage and transport to the laboratory.

Based on the approach of grab sampling, very little decontamination will be required, if any. Most of the telescopic grab samplers that are used for this procedure simply have a small basket at the end which you can place the sample container. The equipment/technician is required to simply dip the container in the water and fill it up. In this case, it will be added to the procedure that the technician will add a rinse at the end of the sample collection with some distilled water, between each sample, for decontamination.

The new division of Access Analytical, On Line Environmental Labs, located at 200-B Rich-Lex Drive, Lexington, SC 29072, will be utilized as a support facility. Equipment used for sampling includes a chlorine meter, temperature/DO meter, etc. Transportation will be provided by an Access Analytical company truck and the sample collector(s) will be one of the assigned Access field technicians. If issues occur in the field, the sample collector will handle these and record the issue and the corrective action in field books and/or logs. If the sample collector cannot fix the situation, then the Project Manager and Laboratory Director will be contacted.

All SOPs are provided in Appendix B, which provides more specifics on both in-situ and laboratory analyzed equipment, operation, deployment, procedure, maintenance, disposal and troubleshooting.

## B3. Sampling Handling and Custody Requirements

For laboratory samplers at the time of sampling, a chain of custody (COC) form must be filled out. The following information must be recorded by samplers:

- Date sample was collected
- Time sample was collected
- Location of sample: city, general location, and specific location.
- Example for a river sample: _____
- Name of sampler
- ID of sampling bottle is the site name and the date collected.
- Analysis (e.g., bacteria) to be conducted, which must also be written in indelible ink on the sample bottle
- Environmental conditions (e.g., waves, currents, tide, wind, sky, rain, runoff)
- Describe in comments section any problems encountered during sampling and corrective actions taken

The sample collector is considered to have custody of the sample until relinquishing the sample. This sample is properly in the custody of the sampler as long as the sample is in possession of the sampler, within sight of the sampler, or locked in a secure place. When the sampler relinquishes custody he/she should sign, date, and write the time the sample was relinquished on the COC form. The person receiving the sample should then sign, date, and write the time the sample was received on the same line. The sample can be relinquished to other qualified individuals in the same manner. Sample receipt in the laboratory is indicated by the Laboratory Director, Laboratory Master Technician or a Laboratory Technician accepting the sample and documenting it on the COC form. If the same individual transports the sample to the lab and processes that sample in the laboratory, then that person will record both accepting and relinquishing the sample on the COC form. A copy of the COC form is provided in Appendix A. For temperature and DO analytes, the readings will be recorded both on the COC and in the field notebook.

### B3.1 Sample Receiving and Storage

Samples must be delivered to the laboratory in coolers packed in ice less than six hours after sample collection. Analysis of the samples must begin within the stated hold times for each parameter from the time of sample collection with the exception of DO and temperature which are in-situ and read immediately after stabilization. At the beginning of sampling, a sample bottle containing water should be placed in the cooler with ice, and then upon delivery of the cooler to the laboratory, the water in this bottle is measured to determine the sample receipt temperature.

Prior to accepting custody and signing for the samples, the laboratory representative verifies that all samples submitted are listed on the COC and that the COC documentation is complete. Received samples and corresponding documentation are carefully reviewed for compliance with regard to condition of containers, sample preservation and temperature (i.e., reading temperature of water blank in cooler), holding times (collection date/time), and accurate identification on the COC.

Once the COC has been verified against the delivered samples, sample information is entered into the laboratory receipt log. The receipt log for samples is kept as a Microsoft Excel spreadsheet. The file is password protected.

Samples received by the laboratory are identified by unique laboratory identification numbers. The sample's laboratory number is transcribed to each container associated with that sample using an indelible marker. Numbered samples are stored in secured areas according to aliquot preservation requirements.

At the end of the day or as soon as practical, the receipt log for all samples received on a day is printed and placed in a logbook in chronological order. The printed sheet(s) must be reviewed for correctness and then initialed at the bottom of the sheet. In the event an error is later found in the receipt log, the change must be made on all recording

documents, electronic and hard copy, as applicable. Hard copy corrections must be made by drawing a single line through the error, writing the correct data above or to the side, and initialing and dating the entry.

### B3.2 Sample Distribution and Handling

Samples retrieved from their designated storage areas must be documented internally. Personnel removing samples from the storage areas are required to record the sample numbers removed, date, time, and their initials on the form. Staff must also document on that form the date and time samples are returned to storage. Several coolers and a refrigerator in the laboratory are for temporary storage of samples requiring refrigeration and awaiting preparation or analysis.

Notification of samples with parameters with critically short hold times (i.e., less than 48 hours) is provided verbally or in writing to the laboratory analytical staff on the day of receipt of such samples. Once notified, it is the responsibility of the analyst to perform the requested analysis within the appropriate hold time.

### B3.3 Sample Disposal

In general, samples are disposed of approx. 14 days after results have been reported to the client. Arrangements for shorter or longer storage times are made with client approval based on specific project requirements. All sample container labels are removed or obliterated prior to disposal. Destruction of samples are noted on internal COC forms.

All samples suspected to be bacterially hazardous, incubated samples, used media, and bacteria control samples are sterilized by autoclaving for 30 minutes at 121 °C. In general, other samples found to be hazardous, or RCRA "D" listed, is returned to the client for disposal. Other hazardous wastes are disposed of by the science building staff by sending directly to an in-state permitted landfill.

Sterilized and non-hazardous aqueous samples are disposed of by pouring the sterilized, neutralized, or non-hazardous sample into a conventional drain to the municipal sewage treatment system. Non-hazardous solid wastes (including emptied disposable containers from aqueous samples) are disposed of by placing in a dumpster for municipal landfill disposal. The date of sample disposal is recorded internally.

## B4. Analytical Methods

### B4.1 Control of Analytical Processes

All aspects of laboratory operations are controlled by key documents: quality assurance manual(s) and standard operating procedures (SOPs). The SOPs detail and document the

procedures which implement the activities and requirements specified in the quality assurance manual.

To perform the tasks described in this QAPP, the laboratory uses 2 field and 2 laboratory analysis procedures:

- E. coli by IDEXX Colilert-24™ QuantiTray™ method , based on IDEXX 06-02027-24
- Total Suspended Solids (TSS) by gravimetric measurement, based on Method 2540 D of *Standard Methods*
- Dissolved oxygen by membrane electrode method, based on Method 4500-O G of *Standard Methods*
- Water temperature by thermometer or thermistor, based on Method 2550 B of *Standard Methods*

The step-by-step procedures of these techniques are provided in laboratory SOPs:

- SM 9223B (E. coli)
- SM 2540-D-2011 (Total Suspended Solids)
- SM 4500-0 G-2011 (field measurement of DO)

All laboratory SOPs referenced in this QAPP can be found on-site of the contracted laboratory at all times. Protocols are also in place, should issues occur in the laboratory. Appropriate corrective actions are outlined within each individual SOP, where applicable.

When samples are completely used or destroyed, a notation is made on the internal chain of custody.

Laboratory turnaround time is generally associated with meeting holding times for samples for analysis but will always be within 10 days after receipt of samples.

Data reports will go through the QA/QC process and then be sent to the City's project manager immediately after validation. The City's project manager will process the report information and submit to DHEC on a quarterly (years 1-3) or monthly (years 4-6) basis.

## B5. Quality Control (QC)

### B5.1 Dissemination of Quality Requirements

The laboratory uses several means of communication to ensure staff is informed of all quality requirements. Routine operational requirements are communicated to applicable staff through

distribution of the QAPP and laboratory SOPs. All these documents are controlled internally and are issued to selected laboratory staff on an individual basis, depending on staff assignment, task responsibilities, and work location. The QAPP and all SOPs are available to all laboratory staff on the laboratory's computer network. Changes in requirements are communicated to laboratory staff by distribution of revisions to this QAPP and applicable SOPs.

Any laboratory staff member observing any occurrence (e.g., equipment failure) that impacts laboratory capabilities or schedule of deliverables (i.e., analysis results are to be reported to SC DHEC and clients within 24 hours of completion of analysis) must immediately bring that observation to the attention of the Laboratory Director. The Laboratory Director shall immediately communicate the situation to the affected customer. A copy of this communication should be placed in the project file and the laboratory director can determine if any corrective actions are necessary.

Quality control (QC) procedures for laboratory measurements in this project are summarized in Tables 4-6. When recording results of QC measurements on samples (e.g., duplicate analysis), an acronym suffix is added to the sample number; the suffixes are as follows:

duplicate = D or DUP

replicates = R# or REP#

matrix spike = MS

matrix spike duplicate = MSD

Acronyms for recording other QC measurements are as follows:

blank = B or BLK

method blank = MB

calibration standard = CAL or CALIB

calibration verification standard = CV

initial calibration verification standard = ICV

primary standard = PS

working standard = WS

laboratory control sample = LCS

Duplicates are typically not helpful for bacterial analyses and are not customarily run for bacteria unless specifically requested. Temperature is measured with a thermometer in-situ conditions. For each cooler of samples that is transported to the analytical laboratory, a 100ml plastic container (prepared by the laboratory) will be included that is marked “temperature blank.” This blank will be used by the laboratory’s sample custodian to check the temperature of samples upon receipt to ensure that samples were maintained at the temperature appropriate for the particular analysis. Typically, a sample is collected in a 250 mL bottle with

no preservative and the hold time is considered immediate. Temperature should be taken by a calibrated NIST thermometer.

### Accuracy

Accuracy (bias) is a measurement of the extent to which a measured value of a quantity (parameter or analyte) agrees with the accepted value of that quantity. It is assessed by the analysis of samples of known concentration for the analytes of concern.

For LCSs, calibration standards, field reference standards, or additional QC samples of known concentration, accuracy is quantified by calculating the *percent recovery* (%R) of analyte from a known quantity of analyte as follows:

$$\%R = \frac{V_m}{V_t} x100$$

where:

$V_m$ = measured value (concentration determined by analysis)

$V_t$ = true value (concentration or quantity as calculated or certified by the manufacturer)

A matrix spike (MS) sample or a matrix spike duplicate (MSD) sample is designed to provide information about the effect of the sample matrix on the digestion and measurement methodology. A known amount of the analyte of interest is added to a sample prior to sample preparation and instrumental analysis. To assess the effect of sample matrix on accuracy, the %R for the analyte of interest in the spiked sample is calculated as follows:

$$\%R = \frac{(SSR - SR)}{SA} x100$$

where:

SSR = spiked sample result

SR = sample result

SA = spike added

## Precision

Precision is a measurement of the random error in an analytical measurement process. It reflects the degree of agreement between independent measurements determined by the analysis of replicate samples. When calculated for duplicate sample analyses, precision is expressed as the *relative percent difference* (RPD), which is calculated as:

$$\mathbf{RPD(\%) = \frac{(S - D)}{\frac{S + D}{2}} x100}$$

where:

S = first sample value (original result)

D = second sample value (duplicate result)

When precision is calculated for three or more replicate determinations, the *relative standard deviation* (RSD), also known as the coefficient of variation, expressed in units of percentage, is used. This is an expression of the spread of the data relative to the mean value of the determinations. The specific formulas used for calculating the RSD are:

$$\mathbf{\bar{x} = \frac{\sum_{i=0}^{n} \left(x_i\right)}{n}}$$

$$\mathbf{s = \sqrt{\frac{\sum_{i=0}^{n} (x_i - \bar{x})}{n - 1}}}$$

$$\mathbf{RSD(\%) = \frac{s}{\bar{x}} x100}$$

where:

$\bar{x}$ = mean of n measurements

$x_i$ = result value for the $i^{th}$ measurement

n = total number of measurements

s = standard deviation

## Method Detection Limits

Method detection limits (MDLs) are determined for each analyte for each method used. These MDLs are determined by (a) conducting replicate analyses of standards at quantities approximately one to five times the estimated MDL, (b) determining the standard deviation, s, of the replicate measurements, and then (c) calculating the MDL from:

$$MDL = t_{(n-1,\ 1-\alpha=0.99)} \times s$$

where:

n = number of replicate analyses

$t_{(n-1,1-\alpha=0.99)}$ = t distribution value appropriate to a 99% confidence level (one-tailed) and standard deviation estimate with n - 1 degrees of freedom

s = standard deviation of the data set

The MDL calculated in this manner represents the minimum amount of a substance that can be measured and reported, with 99% confidence that the analyte quantity is greater than zero.

The MDL does *not* represent the analyte quantity for which there is a 99% probability that the analyte will be detected; there is a 50% probability of detection and reporting of the analyte whose actual amount is at the MDL. The analyte quantity at which there is a 99% probability that the analyte will be detected and reported is twice the MDL.

Because MDLs are usually determined using standards in a clean matrix, they represent optimum obtainable performance. MDLs for actual sample matrices are likely to be higher than those determined using clean matrices.

## Quantitation/Reporting Limits

Because of significant uncertainty (about 33% RSD) associated with MDLs determined in a "clean" matrix, plus possible additional variability due to actual sample matrix, EQL uses higher levels, referred to as "limits of quantitation" or "reporting limits", down to which it routinely reports measured values.

The *limit of quantitation* (LOQ) is defined as 10 times the standard deviation (s) from the MDL determination. Therefore, the LOQ is roughly 3.33 times the MDL, since the MDL is usually about three times s.

The *reporting limit* (RL) is not as rigidly, and usually not as conservatively, defined as the LOQ. It is usually chosen at a level two to 10 times higher than the MDL. As much as possible, it is also chosen at a level which is below applicable regulatory action levels

and which simplifies data review and reporting (e.g., RL of 1.0 µg/L for numerous parameters of similar chemical behavior, MDLs, and regulatory action levels).

## Completeness

The characteristic of completeness is a measure of the amount of valid analytical data obtained compared to the total number of analyses performed. Valid analytical data are those for which all QC specifications are met. Completeness of the reported data (expressed as a percentage) is calculated as:

$$\%C = \frac{M_v}{M_t} x 100$$

where:

$M_v$ = number of measurements judged to be valid (meets all QC specifications)

$M_t$ = total number of measurements performed (based upon number of samples submitted)

## Comparability

Comparability of analysis results is evaluated by at a minimum checking the following against project requirements:

- Analysis method utilized
- Analysis QC measurement results
- Units utilized for reporting measurement values

## Rejection of Data

Rejection of an analytical result for a sample may be required if established quality control acceptance criteria are not satisfied at any point during the course of analysis. Nominal quality control decision criteria are provided in analytical method SOPs and the corresponding data review checklists.

Additionally, outliers are determined using a statistical outlier test (*Standard Methods*, 1010 B. Statistics, 17th through 21st Editions) for evaluation of a questionable value from a group of replicate readings, measurements, results, etc., for an individual sample or standard. Briefly, the test involves dividing the difference between the questionable value and the replicates' mean value by the standard deviation for all replicate values, to calculate a quotient, T. The questionable value is rejected if the calculated T is greater than an established rejection T. The outlier test is conducted at the 99% confidence level, which means if the calculated T exceeds

the rejection $T_{0.99}$, then the questionable value may be rejected with 99% probability that it is significantly different from the other values (Table 4).

**Table 4. Outlier test for evaluation of a questionable group from a group of replicate values**

| Questionable Value[a] | Formula for Calculating T[b] | Rejection Number of Values | Rejection Quotient $T_{0.99}$ |
|---|---|---|---|
| Smallest value ($X_1$) | $T = \frac{X_{ave} - X_1}{s}$ | 3 | 1.15 |
| | | 4 | 1.49 |
| | | 5 | 1.75 |
| | | 6 | 1.94 |
| Largest value ($X_n$) | $T = \frac{X_n - X_{ave}}{s}$ | 7 | 2.10 |
| | | 8 | 2.22 |
| | | 9 | 2.32 |
| | | 10 | 2.41 |
| | | 12 | 2.55 |
| | | 14 | 2.66 |
| | | 16 | 2.75 |

[a] Arrange values in order of increasing magnitude.

[b] If $T > T_{0.99}$ reject questionable value.

$X_{ave}$= average value for all replicates.

s = standard deviation for all replicates, where $s = [\sum(X_n - X_{ave})^2/(n - 1)]^{1/2}$

Table 5. Summary of QC requirements for E. coli analysis by Colilert-24

| QC Sample or Activity | Minimum Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| Capability demonstration | Four (4) prepared samples analyzed prior to any customer sample analyses | Criteria for LCS recovery and duplicate precision | Repeat until acceptable |
| Media sterility check | Prior to use of new lot of Colilert-24 and weekly | No fluorescence | Investigate problem. Eliminate contaminations. Obtain new lot of Colilert-24,if necessary. Repeat until successful before using Colilert-18 lot. |
| Media positive check with control culture | Prior to use of new lot of Colilert-24 and weekly | Fluorescence | Investigate problem. Obtain new lot of Colilert-24 if necessary. Repeat until successful before using Colilert-18 lot. |
| Media negative checks with control cultures (gram+ and gram-) | Prior to use of new lot of Colilert-24 | No fluorescence | Investigate problem. Eliminate contaminations. Obtain new lot of Colilert-24 if necessary. Repeat until successful before using Colilert-18 lot. |
| Method blank | At least weekly, prior to sample analysis | ≤ 20 CFU/100 mL | Clean analytical system and repeat MB analysis. Identify and eliminate source of contamination. |
| Sample duplicate or matrix spike duplicate | At least one (1) weekly, and one with all large sample batches (~20 samples) | RPD ≤ 200% for <150 CFU/100 mL<br>RPD ≤ 100% for ≥ 150 CFU/100 mL | Investigate problem. If system precision is in control, qualify results. If system precision is out of control, reanalyze entire batch. |
| Internal PE sample | Samples and frequency determined by Lab QA Officer | Criteria for LCS recovery and duplicate precision | Investigate all unacceptable results. |
| Blind PE sample | Samples and frequency determined by accrediting agencies and projects | Determined by PE provider | Investigate all unacceptable results. |

LCS = laboratory control sample
QC = quality control
MB = method blank
%R = percent recovery
MDL = method detection limit
RL = reporting limit
PE = performance evaluation
RPD = relative percent difference

Table 6. Summary of QC requirements for TSS

| QC Sample or Activity | Minimum Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| Capability demonstration | Four (4) prepared samples analyzed prior to any customer sample analyses | 90 – 110% R<br>< 10% RSD | Repeat until acceptable |
| Balance Calibration Check | Prior to weighing any sample filters | Weight of certified 200 mg weight: 0.1998 – 0.2002 g | Investigate problem including cleaning weight and balance. If balance is out of calibration attempt recalibration or use another balance until obtain acceptable calibration check. |
| Method Blank | At least one (1) per analysis batch of up to 10 samples | For 1.0 L blank filtered: < 1.0 mg/L | Investigate, identify, and correct the problem. If system accuracy is in control, qualify results. If system accuracy is out of control, correct problem before analyzing samples |
| Sample analysis | For all sample analyses | Total residue on filter:<br>≥2.5 mg to ≤ 200 mg | If total residue on filter < 2.5 mg report result as < RL<br>If total residue on filter > 200 mg filter a smaller volume of sample. |
| Laboratory Control Sample | At least one (1) per year | 90 – 110% R | Investigate, identify, and correct problem. If system accuracy is in control, qualify results. If system accuracy is out of control, correct problem before analyzing samples. |
| Sample duplicate | One (1) per preparation batch of up to 10 samples | RPD ≤ 5% | Investigate problem. If system precision is in control, qualify results. If system precision is out of control, reanalyze entire batch. |
| Internal PE sample | Samples and frequency determined by Lab QA Officer | Criteria for LCS recovery and duplicate precision | Investigate all unacceptable results. |
| Blind PE sample | Samples and frequency determined by accrediting agencies and projects | Determined by PE provider | Investigate all unacceptable results. |

LCS = laboratory control sample
MB = method blank
MDL = method detection limit
MS = matrix spike
PE = performance evaluation
QC = quality control
%R = percent recovery
RL = reporting limit where RL = (2.5 mg /mL filtered) x 1000 mL
RPD = relative percent difference
RSD = relative standard deviation

Table 7. Summary of QC requirements for YSI Pro Plus probes

| QC Sample or Activity | Minimum Frequency | Acceptance Criteria | Corrective Action |
|---|---|---|---|
| Capability demonstration | Four (4) prepared samples analyzed prior to any customer sample analyses | DO 97-104% of theoretical DO<br>Others 75-125% R<br>Others RPD ≤ 25% | Repeat until acceptable. |
| Calibration stability monitoring | Immediately before calibration measure standards | Not applicable. | Not applicable. Results are used to monitor stability of probes and evaluate need for maintenance. |
| Calibration | Daily prior to sample analysis and after every 8 hours | After calibration, measure calibration standards (conductivity, pH, DO % saturation of water saturated air) as sample<br>pH ± 0.1 of expected, others 99-101% R | Investigate and fix any obvious problems. Repeat until acceptable. |
| Calibration check | Immediately following calibration | Measurement of calibration standards or LCS (conductivity, pH, DO % saturation of LCS or of water saturated air)<br>Cond. 90-110% R, pH ± 0.1 of expected, DO 97-104% sat<br>****DO method requires LCS to be read in duplicate with each calib. event**** | Investigate and fix any obvious problems. Recalibrate and repeat until acceptable. |
| Field duplicate (duplicate sample collected at one of sampling sites | One (1) per sampling event | RPD ≤ 25% | Investigate problem. If system precision is in control, qualify results. If system precision is out of control, reanalyze all sampling sites if possible. |
| Internal PE sample | Samples and frequency determined by Lab QA Officer | 75-125% R<br>RPD ≤ 25% | Investigate all unacceptable results. |
| Blind PE sample | Samples and frequency determined by accrediting agencies and projects | Determined by PE provider | Investigate all unacceptable results. |

## B6. Instrument/Equipment Testing, Inspection and Maintenance

Equipment, as used in this QAPP, refers to and includes equipment or instrumentation used in the areas of sample collection, preparation, or analysis. The laboratory utilizes all equipment (Table 8) as appropriate and necessary for a given technique, as specified in a referenced method, or as required by regulatory programs. The equipment investment and subsequent capabilities are sufficient for the laboratory's field and laboratory tasks for this project.

**Table 8. Equipment list**

| Instrument | Number of Units |
|---|---|
| Analytical Balance | **3** |
| Autoclave | **2** |
| Conductivity/Dissolved Oxygen/pH Field Meter | **3** |
| Incubator | **3** |
| Oven | **1** |
| Refrigerator/Freezer | **2** |
| Water deionizing system | **1** |
| Quanti-Tray sealer | **2** |
| Water Bath | **2** |

### B6.1 Preventative Maintenance

Manufacturer recommended preventative maintenance schedules are performed internally for all equipment, in all lab areas. Additionally, some equipment, such as autoclave and analytical balances, require service checks by the commercial vendor. Service calls of this nature are scheduled by the Laboratory Manager according to the maintenance schedule.

Maintenance logs are used to document any procedures performed either internally, or by vendor service technicians. These logs also document maintenance or repair which may be necessary as a part of corrective action resulting from QC failures. Documentation in the logs is the responsibility of the analyst or technician operating the instrument or equipment.

## B7. Instrument Calibration and Frequency

Equipment requiring calibration must be calibrated according to manufacturer's instructions or the analytical method. General guidelines for analytical instrument calibrations are covered in the corresponding analytical SOPs. A summary of instrument calibration procedures for this task's measurements is provided in Table 9.

For equipment where documentation of the calibration can be obtained in the form of hardcopy printouts, the calibration data must be filed with the analytical run data. Where printouts are not possible, the following minimum information must be recorded in a calibration log or on the raw data sheet: equipment identification, calibration date, analyst initials, standard(s) used, certified concentration(s), equipment reading(s) per standard, calibration verification standard(s) results, due date for next calibration. It is the responsibility of the analyst performing calibration to record this information in the calibration log. If repair work or service has been done to any equipment, the analyst shall record the details of this work performed, and obtain any applicable certificates from the vendor.

**Table 9. Instrument calibration procedures**

| Instrument | Calibration Procedure | Frequency | Acceptance Criteria | Corrective Action if Unaccepatable |
|---|---|---|---|---|
| Incubators and Water Bath | One-point or two-point calibration of thermometer with NIST traceable thermometer | Annual | ± 1.0 ˚C | Replace thermometer |
| Refrigerators and pH Meters | One-point or two-point calibration of thermometer with NIST traceable thermometer | Annual | ± 1.0 ˚C | Replace thermometer |
| Ovens | One-point or two-point calibration of thermometer with NIST traceable thermometer | Annual | ± 1.0 ˚C | Replace thermometer |
| Analytical Balance | Calibration verification using NIST traceable weights | Daily | ± 0.1% | Clean and autocal or repair |
| Quanti-Tray sealer | NA | NA | NA | NA |
| pH meter | Two-point calibration with standard buffers | Daily | Slope 90-102% pH ± 0.1 | Clean probe, replace electrolyte, or replace probe as needed. Repeat calibration until acceptable. |
| Conductivity / Dissolved Oxygen / pH field meter | One-point conductivity calib., one-point dissolved oxygen calib. with water saturated air, two-point or three-point pH calib. | Daily | Conductivity or salinity ± 10%, dissolved oxygen ± 5%, pH ± 0.1 | Investigate and correct problem. Repeat calibration until acceptable, if cannot recalibrate repair meter. |

## B8. Inspection/Acceptance Requirements for Supplies and Consumables

Upon receipt, buffer solutions, standards and reagents used in the field kit will be inspected by the laboratory receiving team for leaks or broken seals and to compare the age of each reagent

to the manufacturer's recommended shelf-life. Field personnel will also assure that all supplies and consumables have not expired, have not been tampered with and are appropriate for the work being performed prior to use in the field.

Reagents are replaced before they exceed manufacturer's recommended shelf life. These shelf lives are typically one to two years. However, specific replacement dates can be determined by providing the reagent lot number to the manufacturer. Reagent replacement dates are noted in the maintenance log.

Sample containers will be received by the contracted laboratory. The containers will be examined upon receipt to ensure that the appropriate number and type of containers have been provided to meet sampling needs. The containers will be checked to ensure that preservative has been added, if required. Any discrepancies will require additional containers to be obtained and re-checked. If any sampling problems or abnormalities occur during sampling in the field, the laboratory and the QA Manager for the City shall be notified.

## B9. Data Acquisition Requirements (Non-direct Measurement)

This is not applicable.

## B10. Data Management

A lab staff member collects the sample and preserves it according to the SOPs. The samples are brought to the laboratory. If they are performing the analysis they relinquish them to themselves. If not, they relinquish them to sample custodian who logs and disseminates the samples. The samples are analyzed. The analyst verifies the sample calculations and then they make a hard copy of the data and submit it to the laboratory supervisor. The laboratory supervisor performs a second verification. After which, the data is given to the Laboratory Manager who also reviews the QC, provides verification and then enters the data into a data archive spreadsheet. Once validation is complete, the data are released to the City's QA/Project Manager for review and reporting use.

# C. Assessment and Oversight

## C1. Assessments and Response Actions

**Table 10. Assessments and response actions**

| Assessment | Frequency | Description | Information reported to |
|---|---|---|---|
| Initial demonstration of capability (IDC) | Initially, prior to reporting client data independently | The analyst must prepare four aliquots of a known level of the analyte of interest, analyze them according to the appropriate method, and demonstrate the ability to recover the analyte within established acceptance criteria. | Laboratory Director |
| Data generator review | Every time data is generated | Conduct real-time review and verification of 100% of the data resulting from their activities. | Laboratory Director |
| Analysis of internal and/or external performance evaluation (PE) samples | Once per year or as required by specific client contract requirements. | Analysis of a blind sample for the analyte(s) of interest. Results are evaluated for accuracy by a third party. | Laboratory Director, PE provider, clients, SCDHEC |
| External audits | Per request | Review of entire scope of accreditation and project tasks by state, agency, or affiliations through whom EQL holds some form of certification or contract. | Lab Director, Program Director |
| Lab Certification Evaluations | Minimum of three years | Review of entire scope of accreditation and project tasks by SCDHEC's Office of Laboratory Certification | Laboratory Director, Program Director, SCDHEC, EPA Region 4 |

## C2. Reports to Management

Throughout the year, routine lab reports are prepared and archived for audit as well as the following information, as applicable:

- Goals
- Financial summary and projections
- Measures and comparisons
- Major activities and accomplishments for year
- Needs

# D. Data Validation and Usability

## D1. Data Review, Verification and Validation

| Item | Criteria | If not met sample is accepted, flagged or rejected? | Flag | Comments |
|---|---|---|---|---|
| Sample not analyzed within hold time | Sample received in the lab within 6 hours of collection and analyzed within 2 hours of receipt appropriate hold time | Rejected | HT | Out of holding time |
| Lost sample | Proper COC documentation not followed and sample is misplaced | (Unable to analyze) | LS | N/A |
| Unable to Collect Sample | Various circumstances (i.e., weather, lost sampling container) cause sample to not be collected | (Unable to analyze) | NS | N/A |
| Sample not held within required temperature range | Temperature blank within cooler indicates temperature above 6° C or proper storage equipment failed to read within range (refrigerator/freezer) | Rejected | T | Out of required temperature range |
| Temperature blank not placed within cooler during sample transport | Unknown receipt temperature | Flagged | UT | Noted |
| Incorrect sampling container used for sample collection | Incorrect sampling container used for sample collection | Flagged | SC | Noted |
| Improper preservation | Improper preservation (i.e., acidification, filtering) | Flagged | IP | Noted |

**Table 11: Criteria for accepting, rejecting, or flagging data**

## D2. Validation and Verification Methods

All data receive analyst review and independent analyst. The Laboratory Director and/or quality assurance personnel will review the data to varying degrees at different points in the review process. These review processes are appropriately documented before data are released from the laboratory.

*Data review* ensures that raw data are properly collected, reduced, and reported. *Data verification* confirms by examination of the measurement process and provision of evidence, that specified method, procedural, or contractual requirements have been met. For example, QC measurements must indicate that deviations between measured values and known values are smaller than the maximum allowable error (i.e., DQIs).

*Data validation* is the process of substantiating that specified performance criteria were achieved for an entire data set or data reporting group, including comparisons between analytes and samples to see if relationships are scientifically reasonable.

## D3. Reconciliation with User Requirements

Reconciliation of data with DQI criteria to determine data usability is performed primarily by the Laboratory Program Director working in direct communication with the clients.

# Appendices

## A. Chain of Custody (COC) Form

# Access Analytical - Chain of Custody Record

LAB USE ONLY

**Project Work Order #**_____ **PO #**_____ **Access Quote #** _____ LAB USE ONLY **Laboratory ID:**_____

Company Name: | Preservative: (*see codes below)

Report To:

Address:

City: State: Zip:

Phone: Fax:

Email:

Project Name:

Sampled By:

↓ REQUESTED LAB ANALYSIS: ↓

| Sample Location/Description | Date Collected: | Time Collected: | Type: (grab or composite) | Matrix: (see codes) | # of cont. |
|---|---|---|---|---|---|
| | | | | | |
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ACCESS ANALYTICAL, INC.

7478 Carlisle Street
Irmo, SC 29063

Phone: (803) 781-4243
Fax: 781-4303
Toll Free (888) 315-4243
www.axs-inc.com

*Preservative Codes (*place corresponding # in block above analysis field*): 0=None, 1=HCL, 2=$HNO_3$, 3=$H_2SO_4$, 4=NaOH, 5=$Na_2S_2O_3$, 6=$NaHSO_4$, Other=Specify

*Matrix Codes (*place corresponding code in sample matrix column*): GW=ground water, WW=waste water, DW=drinking water, S=soil, SL=sludge, A=air, IW=industrial waste, WO=waste oil, OT=other (*Specify in comments section*)

NOTES / COMMENTS
(*if sample is a composite please use space below to note start/finish times & days*)

**Turnaround Time:**

___ Standard
___ RUSH*
*Date Required:_____
(For rush work, results emailed/faxed by end of business day on date required)

LAB USE ONLY

Samples Recd. on Ice?
___Yes
___No
Receipt Temp ____

Project Location:
___SC
___NC
___Other_____(specify)

| Relinquished By: | Date: | Time: | Received By: |
|---|---|---|---|
| | | | |
| | | | |
| | | | |
| | | | |

**See Reverse for Terms and Conditions**

Original Copy - Returned w/Report
Yellow Copy - Access File Copy
Pink Copy - Client Copy

Pg_____of_____

## B. Standard Operating Procedures (SOPs)

- **E. Coli (Bacteria)**
- **Dissolved Oxygen (DO)**
- **Temperature**
- **Total Suspended Solids (TSS)**

Parameter: **E. Coli MPN Quantification Procedure**
(Revision Date: July 29, 2014)

Method #: SM 9223 B- 2004

**A.** Scope and Application

The purpose of the MPN (Most Probable Number) Quantification Procedure is to determine the number of E.Coli in waste water. It utilizes an index for reporting purposes. MPN tests the density of Coliform per 100 mL sample. This procedure utilizes a multi-well format and reagents to help determine the results. Chromogenic substrates are utilized to detect Total Coliform and E. Coli positive wells which are enumerated and used to determine test results.

**B.** Apparatus

1. 35 ± 0.5ºC Incubator
2. Long wavelength (365 nm), 6 watt ultraviolet lamp
3. Sterile (100 mL minimum) non-fluorescent borosilicate glass or polystyrene plastic water collection/ culture vessel or equivalent.
4. Quanti-tray Sealer and Quanti-trays (97 well Quanti-tray 2000)
5. Sterile loop
6. Bunsen burner or propane torch
7. IDEXX/Microtech plastic bacteriological bottles containing sodium thiosulfate
8. Color Comparator
9. IDEXX MPN table

**C.** Reagents

1. Colilert
2. Sterilized 100 mL portions of DI Water
3. Color and fluorescence comparators for MPN method
4. Bromocresol purple or other coloring agent (such as food coloring)

**D.** Calibration

Not available

**E.** Procedure

1. Samples are collected in either 120 mL or 250 mL sterilized containers containing sodium thiosulfate. If a sample is collected in a 120 mL container, shake sample thoroughly and aseptically pour sample down to the 100 mL line. This will serve as the reaction vessel. For samples collected in a 250 mL container, shake sample thoroughly and then transfer a measured 100 mL portion into another sample

1. vessel for analysis. This bottle with the 100 mL portion will now serve as the reaction vessel.
2. If necessary, perform a dilution on the waste water using sterile techniques and sterilized 100 mL portions of DI water. This is done by adding 1 mL of sample to a 99 mL portion of DI water and proceeding with the procedure. Typically dilutions are not required.
3. Next, aseptically open a packet of Colilert reagent and add the contents to a 100 mL water sample in an IDEXX bottle. Cap and seal the bottle.
4. Shake vigorously by repeated inversion to aid in dissolution of the reagent. Some particles may remain undissolved. Dissolution will continue during incubation.
5. After sample is added to sample and dissolved, allow any bubbles to dissipate prior to pouring sample into Quanti-tray.
6. Seal Quanti-tray using Quanti-tray sealer and incubate at 35 ± 0.5ºC for 24 hours.
7. Read the reaction after the 24 hour incubation period. This is done by counting the number of large wells and small wells that turn yellow and recording them on the bench sheet. Next, count the number of yellow wells that fluoresced, both large wells and small wells, and record on bench sheet. Look on chart to determine the MPN.

Colorless = Negative
Yellow = Total Coliform
Yellow/ Fluorescent = E. Coli

**A.** Test Results and Interpretation

At 24 hours, compare each reaction vessel against the color comparator provided. If no yellow is observed, the test is negative for Total Coliform and E. Coli. If the sample contains yellow wells greater or equal to the comparator, then the presence of Total Coliform is confirmed.

If yellow wells are observed at 24 hours, check each vessel for fluorescence by placing it 3-5 inches from the UV lamp. Observe for fluorescence in a dark environment. If fluorescence of wells are greater or equal to fluorescence of the comparator, the presence of E. Coli is confirmed. (The comparator is the lowest level of yellow and fluorescence which can be considered positive. A typical positive test tends to have a much more intense coloration than the comparator). **Note: Be sure to count only the yellow wells that fluoresce.**

If a sample is yellow after 24 hours of incubation, but slightly less than the positive comparator, it may be incubated up to an additional 4 hours. If the sample is Coliform positive, the color will intensify. If it does not intensify, consider the sample negative. If the color of the sample cannot be determined to indicate positive or negative, invalidate the sample and resample from the same site. Some water samples containing humic material may have an innate color. If a water sample has background color, compare inoculated Colilert vessel to a control blank of the same water sample.

For MPN quantification, use the table for the 97 well trays provided by IDEXX to obtain the MPN value based on number of yellow and yellow/ fluorescent wells.

**B.** Sampling and Preservation

Samples are to be placed in sterile bacteriological (either 100 mL or 250 mL) bottles that contain sodium thiosulfate.

Samples are to remain in refrigerator or on ice at <10°C until tested.

Samples are to be received in lab and analyzed within 8 hours of being sampled. Once received, they are to be run in accordance.

**C.** QA/ QC

1. The Quanti-tray sealer must be checked monthly for leaks by adding bromocresol purple or other coloring agent to 100 mL of water. If dye is observed outside the wells, maintenance must be performed.
2. In addition, a sterility control and positive control are run once each week per batch.
3. Routine quality control should be conducted on each lot of Colilert reagent received and each lot of sample bottles, whether factory sterilized or sterilized in the lab. For each new lot of Colilert purchased, tests are to be performed in the same manner as sampling utilizing E. Coli, Pseudomonas, Klebsiella, and a sterility control. E. Coli is used as the E. Coli positive control. Pseudomonas is used as the E. Coli negative control. Klebsiella is used as the Total Coliform positive control / E. Coli negative control. All QA/QC data performed on Colilert reagent and/ or sample bottles is to be recorded in the respective section of the QA/QC book.
4. Unknown Proficiency Test samples must be analyzed at least once each year.
5. Sample disposal of microbiological waste associated with testing are to be performed by autoclaving samples in a biobag for 30 minutes at 121° C.

**Parameter: Dissolved Oxygen (D.O.)**
(revision date: March 18, 2013 )

Method # SM 4500-O G- 2011

### A. Discussion

Membrane electrodes are used for DO measurements for stream surveys, control of industrial effluents, measurements in lakes and reservoirs, as well as continuous monitoring of DO in activated sludge units. Since they are completely submersible, membrane electrodes are suited for analysis in situ. They are particularly convenient for field applications due to their portability and ease of operation. In addition, the use of membrane electrodes for D.O. analysis is recommended for use under conditions that are unfavorable for use with the iodometric method or when the tests are subject to interferences.

### B. Reference

Standard Methods, 22nd Edition

### C. Apparatus

Dissolved Oxygen meter, accurate and reproducible to 0.1 mg DO/L with a range of 0 to 20mg/L, that has temperature-compensation adjustment. Currently in use are the Accumet AR40 D.O., the YSI Model 51B Meters, YSI DO200, as well as the YSI Model 55 Meter.

### D. Reagents

Refer to the Fisher or YSI Dissolved Oxygen Probe Operating Instruction Sheets in regards to reagents and supplies. D.O. electrolyte filling solutions as well as membranes are necessary for these meters and probes.

### E. Calibration of Meter

## ACCUMET AR40 D.O. Meter

**Standardization/(Calibration) and Usage Procedure**

Reference Fisher Scientific Probe Instruction Sheet for instructions on setting up and maintaining D.O. Probe.

Once the D.O. probe is set up properly, it should be connected to the proper DIN connector on the back of the Accumet meter. The probe must be allowed to warm up for 30 minutes prior to calibration. We keep the meter plugged in so that the probe should be ready for calibration immediately when needed, but always ensure that the probe is

properly warmed up. Always store the probe in a BOD bottle filled with at least one inch of DI water to keep the membrane in moist air.

The Accumet meter is equipped with a touch screen. A light touch on this screen is all that is necessary to access various meter functions. The meter is normally kept on STANDBY. To access the MODE screen simply touch anywhere on the screen.

For D.O. Standardization, we use the automatic standardization mode as follows:

1. **Gently blot the membrane with a Kim-Wipe to remove moisture droplets that will cause inaccurate standardization if left on the membrane.**
2. Once the membrane is dried make sure the meter is in D.O. mode. (Press "MODE" and "DO")
3. Press the STD button to enter standardization mode.
4. Erase the previous standardization by pressing "CLEAR"
5. Touch "STD" again to standardize the meter. MEASURING will flash until the signal is stable, at which time the meter will accept the reading and return to the measure screen. The screen will indicate the D.O. reading at the time of standardization. This, along with all other appropriate information, should be entered into the D.O. meter calibration log.
6. Touch "MEASURE" to begin measuring the D.O. of your BOD samples. Auto Read should be OFF so that the meter will continuously monitor the D.O.
7. Record the D.O. Reading for each bottle when the STABLE message appears on the screen.

## YSI Model 51 B D.O. Meter

### Standardization/(Calibration) and Usage Procedure

Reference YSI Probe Instruction Sheet for instructions on setting up and maintaining D.O. Probe.

For D.O. calibration in water saturated air:

1. Turn on the D.O. Meter and allow to warm up for 15 minutes.
2. **Gently blot the membrane with a Kim-Wipe to remove moisture droplets that will cause inaccurate standardization if left on the membrane.**
3. Place probe in partially filled BOD bottle.
4. Set 'zero' and 'full scale'
5. Turn dial to "Read Temp and Set Dial"
6. Set temperature dial on meter to correspond to current temperature.
7. Turn dial to "Read O2"
8. Read barometric pressure from barometer and record in mmHg. This value will be 'A'
9. Refer to the wet air calibration table to determine the wet air D.O. value at actual temperature (see step #6) and standard pressure (760mm). This value will be "B'.
10. Calculate the actual wet air (wa) D.O. using the following formula:

$$\text{Actual wa D.O., mg/L} = \frac{A - 15mm}{760\ mm} \times B$$

11. With meter switch set at "Read O2", adjust meter to read actual wa D.O. Meter is now calibrated for use. Remember to adjust temperature dial as required when reading samples. Be certain to make sure that meter is holding calibration as well, with checks for drift after reading out each batch of samples. Make certain that no air bubbles are trapped inside bottle during D.O. reading.

## YSI Model DO200 D.O. Meter
### Standardization/Calibration and Usage Procedure

Reference YSI Probe Instruction Sheet for instructions on setting up and maintaining D.O. Probe.

For D.O. calibration in water saturated air:

1. Ensure that the sponge inside the calibration chamber is damp. Insert the probe into the chamber, being careful to make sure the membrane does not touch the sponge.
2. Turn the instrument on by pressing the ON/OFF button. Wait for the DO and TEMP readings to stabilize. This may take 10-15 minutes.
3. Press the CAL button.
4. The meter will prompt you to enter the local pressure in mBar. Use the up and down arrows to increase or decrease the pressure to the desired number.
5. The local pressure in mBar is found by multiplying the mmHg by 1.333. The mmHg reading is taken from the barometer.
6. When the proper pressure in mBar is displayed, press the enter key to view the calibration value in the lower right of the display. Once the value in the main display stabilizes, press the enter key again to move to Salinity calibration.
7. Enter in the approximate Salinity of the water to be analyzed (0 for fresh water) and press the enter key. The meter will now return to the DO measurement mode. You are now ready to analyze samples.
8. Place calibrated probe in sample and stir gently.
9. Wait until probe equilibrates by observing temperature and dissolved oxygen readings that are stable for a full minute.
10. The meter will hold calibration even if it is powered off. It is recommended to check calibration with each use and recalibrate as necessary to prevent drift.

## YSI Model 55 D.O. Meter
### Standardization/Calibration and Usage Procedure

Reference YSI Probe Instruction Sheet for instructions on setting up and maintaining D.O. Probe.

For D.O. calibration in water saturated air:

1. Ensure that the probe in inserted into the calibration chamber on the meter and that the sponge inside the chamber is wet.
2. Turn the meter on and wait for the dissolved oxygen and temperature readings to stabilize, which usually takes 15minutes.
3. Enter the calibration menu by pressing the UP ARROW and the DOWN ARROW keys at the same time.
4. Enter the local altitude in hundreds of feet. For our laboratory, the altitude is entered as 0. Use the arrow keys to increase or decrease the altitude. Press ENTER once the proper altitude is entered.
5. CAL will now be displayed in the lower left side of the display. The current DO reading before calibration should be displayed on the main screen.
6. Make sure the DO reading is stable and then press the ENTER button. The meter will prompt the user to enter the salinity of the water to be analyzed. Enter this approximate value and press the ENTER key. (zero for fresh water)
7. The meter will then return to normal operation and display the calibrated DO reading on the main screen. The user can now move back and forth from DO in mg/L to % saturation by using the MODE key.

## YSI Model Pro20

### Standardization/Calibration and Usage Procedure

Reference YSI Probe Instruction Sheet for instructions on setting up and maintaining D.O. Probe.

For D.O. calibration in water saturated air:

1. Ensure that the probe in inserted into the calibration chamber on the meter and that the sponge inside the chamber is wet.
2. Turn the meter on and wait for the dissolved oxygen and temperature readings to stabilize, which usually takes 15minutes.
3. Once One Touch Calibration has been enabled, press and hold the **Cal** key for 3 seconds. The meter will indicate '*Calibrating %DO'* on the display and automatically calibrate the sensor to the barometer and salinity correction values.
4. 'Calibration Successful' will display for a few seconds to indicate a successful calibration and the instrument will then return to the run screen.
5. You are now ready to analyze samples.
6. Place calibrated probe in sample and stir gently.
7. Wait until probe equilibrates by observing temperature and dissolved oxygen readings that are stable for a full minute.
8. The meter will hold calibration even if it is powered off. It is recommended to check calibration with each use and recalibrate as necessary to prevent drift.

## F. Sample Measurement (Analysis)

1. Insert the probe into the sample.

2. Turn on stirrer or agitate the probe in the sample to dislodge air bubbles from the sensing area of the probe tip.
3. Stir the sample vigorously with the probe.
4. Turn the meter to "Read Temp and Set Dial" and wait for temperature reading to stabilize. Adjust dial if necessary to current temperature reading. (This is done for the YSI 51B meter only.)
5. Turn meter to "Read O2". Once meter stabilizes, record D.O. reading. (Model 51B)
6. For meters with automatic temperature compensation, wait for temperature and D.O. readings to stabilize and record value.
7. Rinse D.O. probe with DI water and store probe between measurements in the calibration and storage chamber (BOD bottle half full with DI water).

## G. Sampling and Preservation

Usually the D.O. test is performed immediately in the field at the time of sampling. If the sample cannot be read out in the field it must be analyzed within 15 minutes of sampling.

## H. QA/QC

1. The D.O. meter must be calibrated each day before use.
2. Monthly cleaning/maintenance should be performed on the D.O. probe according to manufacturer's instructions. Refer to the Accumet or YSI Dissolved Oxygen probe operating instruction sheets for cleaning and maintenance instructions. Currently, we are changing the membrane and filling solution as well as cleaning the probe weekly, or every two weeks depending on frequency of probe use.
3. Calibrate temperature annually against a NIST certified thermometer.
4. Analyze duplicates with each batch. For field analysis, duplicates are not analyzed on samples analyzed in-situ. A notation is made on the bench sheet that the analysis was performed 'in-situ' if no duplicate was run. For samples that are analyzed with duplicates, the duplicate is specifically a QC value only.
5. Zero check with a zero oxygen sample. (Sample made from excess Sodium Sulfite and a trace of Cobalt Chloride, or purchased from a supplier.) This only needs to be run if the manufacturer recommends this check after calibration. Currently our meters do not require this.

Parameter: **Temperature**
(revision date: December 11, 2012)

Method: SM 2550 B- 2010

*A. Discussion*

Temperature readings are used in many general laboratory operations. For instance, temperature is used in calculations of alkalinity, in studies regarding saturation and stability with respect to calcium carbonate, and in calculations for salinity. Elevated temperatures that are caused by discharging heated water may have important effects on the surrounding environment. Temperature readings are also helpful to identify sources of water supply, such as deep wells. Industries very often may require information about water temperature for use in their plant processes or for calculating factors such as heat-transmission.

*B. Reference*

Standard Methods, 22nd Edition

**C. Apparatus**

1. NIST-certified thermometer
2. Thermometer having a minimum scale marked for every 0.1°C (with markings etched on the capillary glass). The thermometer should have a minimal thermal capacity to allow for rapid equilibration.
3. For field use, use a thermometer that has a metal case to prevent breakage.

**D. Calibration**

Thermometers should be calibrated with a NIST-certified thermometer before use and at least annually thereafter.

**E. Sample Measurement (Analysis)**

1. Once the thermometer has been calibrated against a NIST-certified thermometer and a correction factor has been established, it is ready for sample measurement.
2. Make sure the thermometer is immersed in water long enough to allow for complete temperature equilibration before taking reading.
3. Record the reading taking into account the correction factor.

**F. Sampling and Preservation**

Temperature is generally a parameter that is tested on site at the time of sampling.

## G. QA\QC

NIST-certified thermometer must be sent in for calibration every 5 years. This calibration certificate is kept in the QA/QC manual. Thermometers used in the lab/field are calibrated using the NIST-certified thermometer annually and records pertaining to this calibration are kept in the QA/QC manual. Calibrations against the NIST-certified thermometer are performed at, or close to, the working temperature for that specific thermometer. An allowance of +/- 1 degree compared to the NIST-certified thermometer is acceptable; thermometers that are not within the 1 degree allowance are not to be used.

Parameter: **Total Suspended Solids (TSS)/ Volatile Suspended Solids (VSS)**
(revision date: April 17, 2014)

Method # SM 2540 D- 2011 / SM 2540 E (VSS)

## A. Discussion

The total suspended solids (TSS) test determines the non-filterable residue portion of a wastewater sample. A sample is filtered through a standard glass fiber filter, and the trapped residue is dried at 103-105°C. The weight of the dried residue is used to determine the TSS value of the sample.
Since most NPDES and pretreatment permits limit TSS in permitted discharges it is common to analyze for it in many effluents. Some influents are also analyzed for TSS in order to determine plant loading and removal efficiency.
Volatile suspended solids (VSS) is an indirect measurement of the organic solids of a sample. It is commonly used to roughly estimate the bacterial content of mixed liquor in an activated sludge plant. VSS is defined as the portion of solids volatized at 550± 50°C for 15 minutes.

## B. Reference

Standard Methods, 22nd Edition
Methods for Chemical Analysis of Water and Wastes

## C. Apparatus

1. Filtration Funnel for 55 mm and 110 mm size filters
2. Glass Fiber Filters (47 mm): Whatman 934-AH or equivalent in 55 mm and 110 mm sizes
3. tweezers
4. vacuum flask with tubing
5. analytical balance able to weigh to 0.0001 g
6. aluminum weighing pans for 55 mm filters and foil squares for 110 mm filters
7. drying oven maintaining 103-105°C
8. wash bottle
9. graduated cylinders (various sizes)
10. dessicator
11. vacuum pump
12. **muffle furnace (for VSS)**

## D. Reagents

Deionized Water

## E. Calibration

Zero analytical balance before weighing filters and after every 4 filters weighed to ensure accuracy.

## F. Sample Measurement (Analysis)

**PRIOR TO ANALYSIS:**

1. Insert glass fiber filter into filtration assembly with wrinkled side up. Apply vacuum and wash 3 times with 20 mL DI water. This "cleans" and prepares the filters for use in the test.

**NOTE**: If volatile solids are to be determined ignite the filter in muffle furnace at 600 ± 50°C for 15 minutes and proceed to step 3.

2. Dry the cleaned filter(s) at 103-105°C for 1 hour.

3. Cool in dessicator, and weigh filter(s) before use. Always use tweezers to transfer filters to and from balance. (Touching cleaned filters with your fingers may transfer oil from your skin onto the filter causing inaccurate weight measurements). This initial weight is the "tare" weight of the filter.

**SAMPLE ANALYSIS:**

**TSS**

1. Place a pre-weighed filter into the filtration funnel (using tweezers) wrinkled side up, and pull a small amount of deionized water through it in order to seat the filter.
2. Pour a portion (up to 1000 mL) of well-mixed sample into a graduated cylinder. The amount used is determined by the solids content of the sample. For example, a high quality effluent may filter easily. In such cases, 1000 mL of sample will pass through the filter before it plugs. Use historical data, where possible, to determine the optimum volume.

   **NOTE**: DHEC requires that a filter contain between 10-200 mg (0.01-0.20 g) of residue, whenever possible, unless 1000 mL of sample was pulled through the filter.

3. Filter the sample, and record the volume filtered. If it is not possible to pull 1000 mL of sample through the filter the analyst should note on the bench sheet that the filter clogged.
4. Wash the filter with 3 successive 10 mL volumes of deionized water. If the graduated cylinder was emptied of sample rinse it out with the 3 successive 10 mL volumes of DI water and pull these rinsings through

the filter. This ensures that no solids were left on the sides of the graduated cylinder.

5. Once the DI water washings have been pulled through the filter rinse the sides of the filter funnel with a small amount of DI water and pull through the filter to rinse any solids down off the funnel.
6. Using tweezers, place the filter(s) into labeled aluminum dishes or foil squares.
7. Put the filters in the drying oven and dry for one hour at 103-105°C.
8. Remove the filters from the drying oven and place them in the dessicator until their temperature stabilizes. We have determined that 15 min. is sufficient time.
9. Zero the balance and weigh filters. Record their weights appropriately on the bench sheet. Remember to check the balance zero after every 4 filters are weighed. ALWAYS USE TWEEZERS TO TRANSFER FILTERS.
10. Repeat steps 7-9 until the weighings are consistent. (The difference between weighings is ≤0.0004 g or <4%, whichever is less.

**VSS**

Be sure to record the weight of the filter + residue prior to ignition in muffle furnace!

11. Turn on muffle furnace and allow temperature to stabilize at 550 ± 50°C.
12. Place filter in muffle furnace for 15 min. Filter can be placed in an aluminum weighing dish or in a clean porcelain crucible during ignition.
13. Remove the filter from the muffle furnace USING TONGS and place in the dessicator until it cools to a stable temperature. We have determined that 15 min. is sufficient.
14. Zero the balance and weigh the filter. Record the post-volatile weight on the bench sheet.
15. Repeat steps 7-9 until the weighings are consistent. . (The difference between weighings is ≤0.0004 g or <4%, whichever is less.

**TS (Total Solids)**

Total solids are performed on process control samples on an as needed basis. A mixed sample is evaporated and dried in a weighed dish in a 103-105˚C oven. The increase in weight over that of the empty dish after evaporation represents the total solids .

1. Weigh and record the weight of a cleaned evaporating dish.
2. Mix sample well and transfer to dish, recording the amount of sample added in mLs.
3. Allow sample to evaporate and dry at least for one hour in the oven at 103-105˚C.

4. Place dried dish in desiccator for 15 minutes prior to weighing dish and recording weight.
5. Repeat the cycle of drying and weighing until a constant weight is obtained or until the weight change is 4% or less of the previous weight.
6. Calculation is as follows:

$$\text{mg total solids/L} = \frac{(A-B) \times 1{,}000{,}000}{\text{sample volume, mL}}$$

where: A= weight of dried residue + dish, mg
B= weight of dish, mg

**CALCULATION:**

**TSS**

$$\text{TSS, mg/L} = \frac{(A-B) \times 1{,}000{,}000}{\text{sample volume, mL}}$$

where: A = weight of filter + dried residue, g
B = "tare" weight of filter, g

Report final results to 2 significant figures.

**VSS**

$$\text{VSS, mg/L} = \frac{(A-B) \times 1{,}000{,}000}{\text{sample volume, mL}}$$

where: A = weight of filter + dried residue prior to ignition, g
B = weight of filter + dried residue following ignition, g

Report final results to 2 significant figures.

## G. Sampling and Preservation

1. Container: Polyethylene or Glass
2. Preservation: cool to 1-6°C
3. Maximum Holding Time: 7 Days
4. Samples can be composite or grab

## H. QA/QC

1. Duplicates are performed on 10% of samples.
2. Run a blank with each batch of samples.

3. Check calibration of the analytical balance monthly using class S weights.
4. Have balance professionally calibrated on a yearly basis.