We Are Columbia

Quality Assurance Project Plan

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

Prepared by City of Columbia Department of Engineering

November 2015
Revised May 2017
Revised December 2018
Revised January 2019

Lead Organization: City of Columbia
Department of Engineering
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Project Manager: Stormwater Manager (if Stormwater Manager position is vacant, the Director of Engineering or his/her designee)
City of Columbia, Department of Engineering

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

Project Manager:
City of Columbia
Stormwater Manager
or Director of Engineering
or Designee

Date: 1/18/19

Print Name: Alfred Jessee

Print Title: Storm Water Manager

QA Manager:
City of Columbia
Stormwater Plan
Reviewer or Deputy
Director of Engineering
or Designee

Andrea Bolling Date: 1/18/19

Print Name: Andrea Bolling

Print Title: Deputy Director of Engineering

Assistant City Manager:
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Columbia Water
Assistant City Manager or
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Manager for Columbia
Water

Clint E. Shealy Date: 1/18/19

Print Name: Clint E. Shealy

Print Title: Assistant City Manager

WQ Manager:
SCDHEC
Bureau of Water

David R Graves Date: 4/2/19

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QA Office:
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Nydia F Burdick Date: 3/15/19

Nydia Burdick, Manager

Laboratory Director:
Rogers and Callcott

Anne Norris Date: 1/29/18

Anne Norris

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A. Project Management

A3. Distribution List

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Anne Norris	Rogers and Callcott – Laboratory Director	anne.norris@rogersandcallcott.com

A4. Project/Task Organization

The tasks of the City of Columbia's QAPP will be to monitor four parameters at three different S.C. DHEC established water quality monitoring stations for a period of six 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:

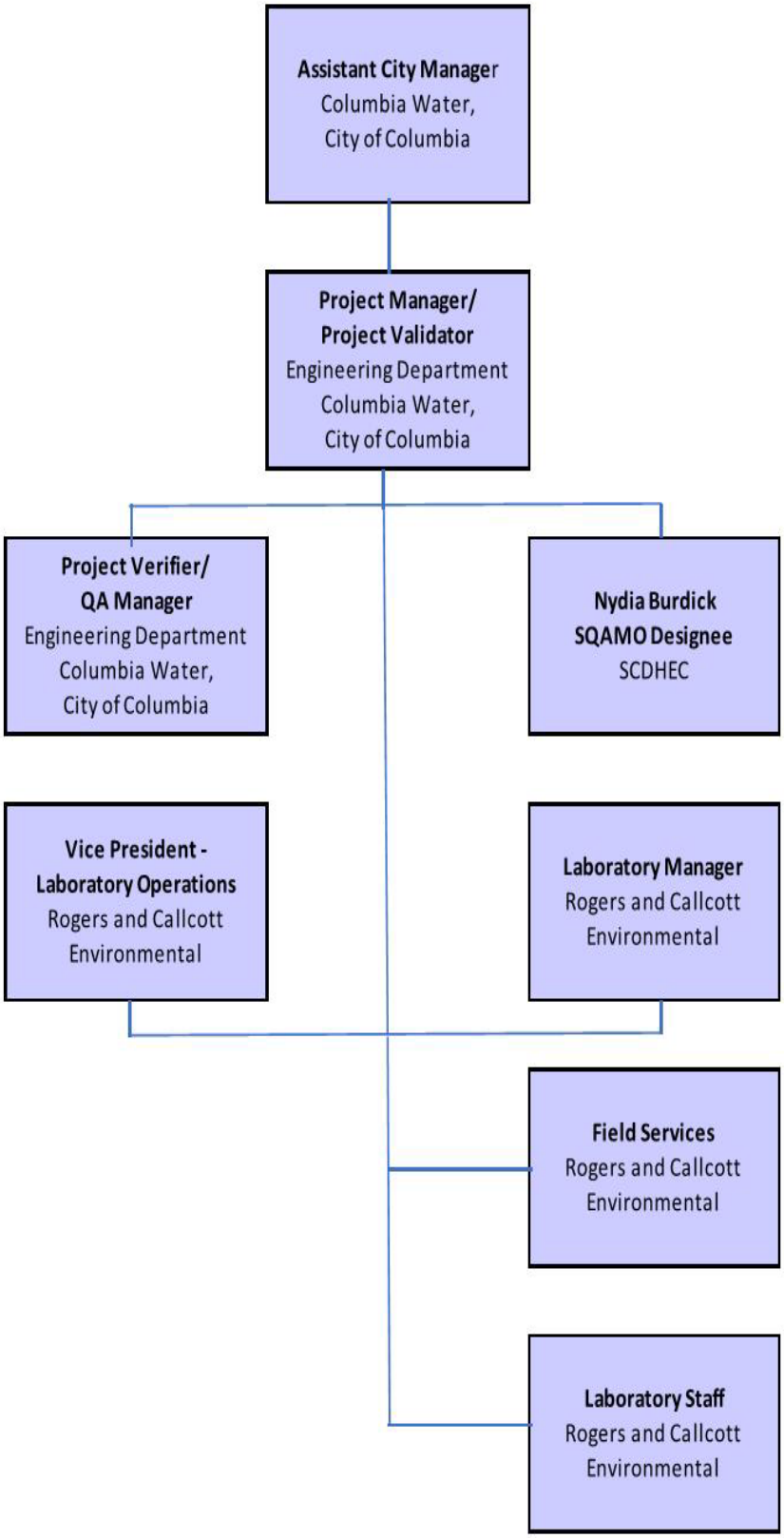
Project Manager/Project Validator (Stormwater Manager {*if Stormwater Manager position is vacant, the Director of Engineering or his/her designee*}, City of Columbia, Department of Engineering) – Will manage the project including developing and maintaining the QAPP and submitting reports to hand off to CDM Smith and EPA, per the Consent Decree Schedule, and validating data.

Project Verifier/QA Manager (Stormwater Plan Reviewer {*if Stormwater Plan Reviewer position is vacant, the Deputy Director of Engineering or his/her designee*}, City of Columbia, Department of Engineering) – Will ensure quality of laboratory analysis results, review and confirm the acceptability of data generated from work performed and verify that the work performed fulfills the specified requirements set forth in the QAPP.

Rogers and Callcott Environmental – Will perform field analysis/sampling and confirm/compile data for City reports. (Laboratory Certification Number: 40572001)

Nydia Burdick (SCDHEC) – Will review and approve the QAPP and any revisions thereto.

Figure 1: Organizational Chart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Rogers and Callcott
Environmental
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Rogers and Callcott
Environmental
Field Services
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Laboratory Staff
Rogers and Callcott
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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, as required, 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. For years 4 through 6 under the Consent Decree, monitoring will be performed monthly at Sites C-001 and B-280 and every other month at Site C-017.

II. Water Quality Stations (see attached map):

Table 1: Water Quality Monitoring Stations/Sites

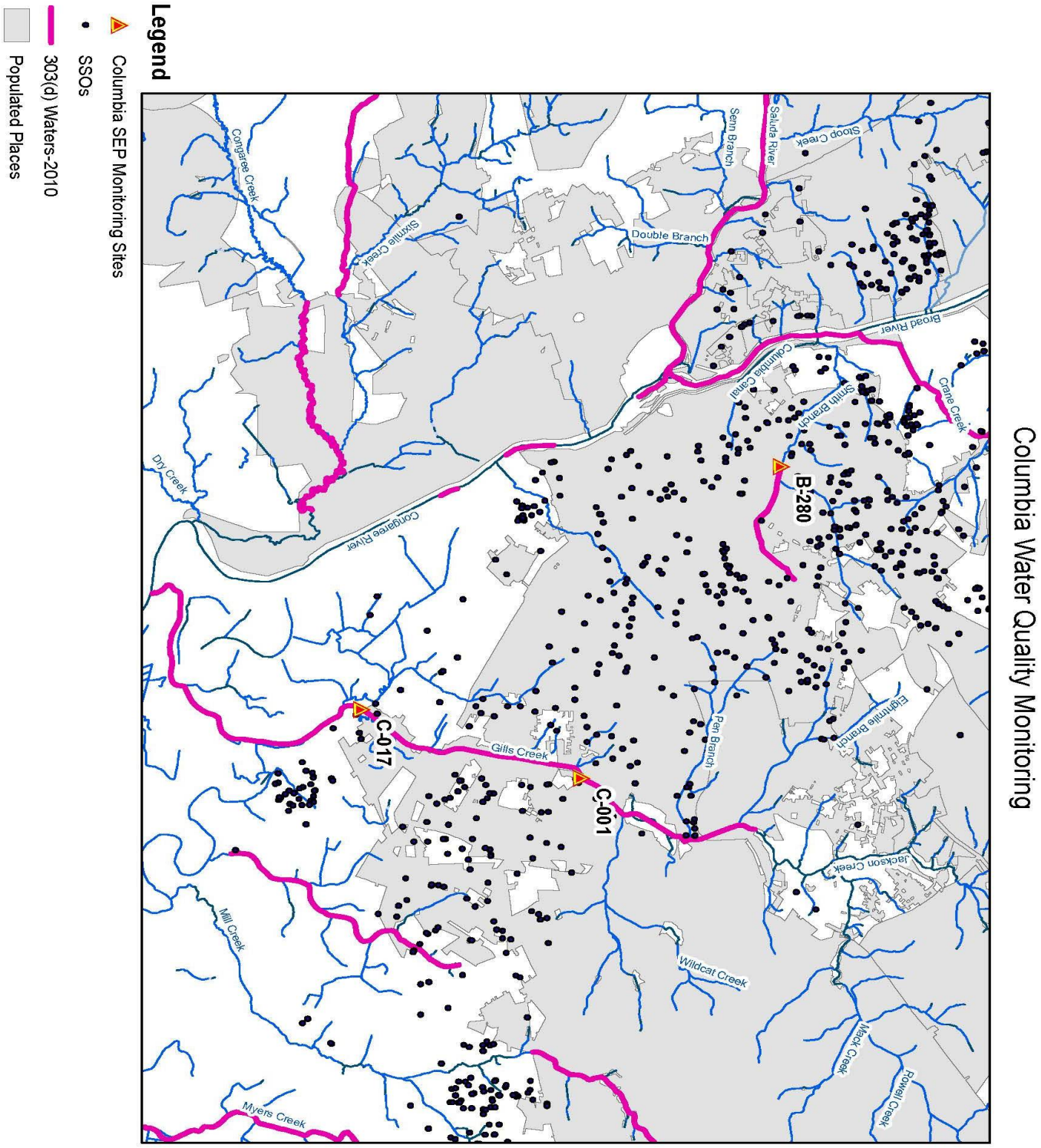
Site	Description	Impairment	TMDL	Monitoring Parameters	Frequency
C-001	Gills Creek @ Garners Ferry Road	Fecal Coliform	Yes	DO *E. coli* Temp TSS	Quarterly, 1/2016 – 12/2018; Monthly, 1/2019 – 12/2021
B-280	Smith Branch @ North Main Street	Fecal Coliform	Yes	DO *E. coli* Temp TSS	Quarterly, 1/2016 – 12/2018; Monthly, 1/2019 – 12/2021
C-017	Gills Creek @ Bluff Road	Fecal Coliform; Dissolved Oxygen	Yes	DO *E. coli* Temp TSS	Quarterly, 1/2016 – 12/2018; Every other month, 1/2019 – 12/2021

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.

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.

A6.1 Map of Monitoring Sites

Figure 2: Map of DHEC Monitoring Stations / Sampling Sites



A7.1 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 for the first 3 years under the Consent Decree and monthly (every other month at Site C-017) from years 4 through 6 under the Consent Decree.

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 0.3 meters.

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, 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.

Note: LCS/LFB will not be run for each TSS sample batch, as this is not a method requirement. The lab analyzes a weekly LCS and routine proficiency tests. TSS methods do include precision analysis by the use of sample duplicates which are required for each sample batch. E Coli and other microbiological analysis do not have precision measures because of the nature of microbiological analysis.

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. The DQI criterion for precision does not include information for bacteria and DO.

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. 100% completeness is required for this project. Re-sampling will be conducted if a collected sample is inadvertently destroyed, or if a sample is otherwise unavailable or compromised.

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. Since samples at the selected locations and parameters have been collected in the past by DHEC, the methodology used for this project will be the same as those used by DHEC. This will ensure that results obtained from this project will be directly comparable to the historical results obtained by DHEC.

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.

Table 2: Criteria for Measurement DQIs

Parameter	Units	Accuracy[a] (LCS)	Accuracy[a] (Matrix Spike)	Precision[a] (RSD or RPD)	MDL[b]	RL[c]	Complete-ness (%)
E. coli	CFU/100 ml	NA	NA	NA	1 CFU/100 mL FU	*1 CFU/100 mL 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	within ±0.2 mg/L of the published saturation at the observed temperature and altitude/barometri c pressure.	NA	NA	<0.3	*<0.3*	100
Water Temperature	°C	NA	NA	± 0.5°C	NA	*NA*	100

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.

A8. Special Training Requirements and Certifications

The Certificate issued by the SC DHEC Office of Environmental Laboratory Certification for Rogers and Callcott Environmental (Columbia) is 40572001. Rogers and Callcott Environmental has offices in Greenville and Columbia SC. Work for this project will be performed at the Columbia location – 215 Stoneridge Dr, Columbia SC 29210.

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 at 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 QC Manager 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. 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 or designee 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 under the Consent Decree and monthly (every other month at Site C-017) from years 4 through 6 under the Consent Decree. 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 as defined in the Consent Decree. Hardcopies will be bound and stored 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 pursuant to the reporting requirements of the SEP (specifically Revised Appendix I of the Consent Decree)..

The copy of the data package provided to the City and all associated raw data are kept as defined in the Consent Decree. These laboratory records are stored electronically and backed up off-site. For electronic data deliverables in Microsoft Excel or similar formats, laboratory files are also maintained electronically and backed up off-site.

Laboratory and field data for the four required parameters will be collected and evaluated in accordance with this QAPP. Analytical reports will be periodically provided to the City by the laboratory, and subsequently reviewed by the appropriate City personnel. After review and approval by the City, reports will be forwarded electronically to DHEC pursuant to the reporting requirements of the SEP (specifically Revised Appendix I of the Consent Decree).

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 will be collected and analyzed in accordance with the selected methodology and standards.

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.

Sampling will begin in the first month following approval of this Plan. The sampling schedule will generally be at regular intervals based upon the week (1^{st}, 2^{nd}, 3^{rd} 4^{th}) of the month in which the first samples were collected. For example, if the first samples were collected in the 2^{nd} week of January, subsequent quarterly samples will be collected in the 2^{nd} week of every third month thereafter (in this case April, July, October); for monthly samples collection would take place the 2^{nd} week of every month thereafter. Sample schedule may fluctuate within the quarter/month, but the sampling will occur as close as possible to this schedule. Sample collection/analysis 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 reviews this laboratory report, the report will be finalized. If a sample is destroyed or lost 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.

Variations in weather, especially precipitation, can cause variation in bacterial counts. Since this project involves collection of bacterial samples within streams, it is expected that frequency, duration, and timing of precipitation events will affect sampling data. Weather at the time of sample collection will be recorded to assist with determining data that may have been affected by variations in weather.

B2. Sampling Methods

As mentioned before, four parameters will be measured on a quarterly basis for the first 3 years under the Consent Decree. For years 4 through 6 under the Consent Decree, monitoring will be performed monthly at Sites C-001 and B-280 and every other month at Site C-017.

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.

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.**

Table 3: Sample Collection Criteria

Sample Type	Parameter Measured	Sample Container	Minimum Sample Size	Preservation Method/ Storage
Urban stream/ditch water, collected via grab samples	*E. coli*	Sterile glass or sterile plastic with sodium thiosulfate	100 mL	Field: store in cooler at 1-6 °C Lab: store in refrigerator at 1-6 °C and start analysis within 8 hours

Sample Type	Parameter Measured	Sample Container	Minimum Sample Size	Preservation Method/ Storage
Urban stream/ditch water, collected via grab samples	Total Suspended Solids (TSS)	plastic	1000 mL	Field: store in cooler at 1-6 °C Lab: store in refrigerator at 1-6 °C and start analysis within 7 days

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 Technician or Field Sampling Technicians. 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 from the stream immediately downstream of the 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 are carefully filled with river water 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 container at the end which you can place the sample bottle. The equipment/technician is required to simply dip the bottle 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.

Equipment used for sampling includes a temperature/DO meter.

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 provide more specifics on both field 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.
- Name of sampler
- A unique identifier will be on each sampling bottle, consisting of the site name/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 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. The laboratory will also compete a sample receipt form detailing any issues at the time of receipt.

Once the COC has been verified against the delivered samples, sample information is entered into the laboratory LIMS system.

Samples received by the laboratory are identified by unique laboratory identification numbers. The sample's laboratory number is printed on a waterproof label and attached to each bottle. Numbered samples are stored in secured areas according to aliquot preservation requirements.

At the end of the day or as soon as practical, the work order and COC for all samples received on a day is reviewed electronically for correctness. COC 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. In some cases (i.e. client request or lawsuits, etc), 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 refrigerators in the laboratory are for 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 approximately 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.

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* (MPN) by Method 9223B of *Standard Methods*
- Total Suspended Solids (TSS) by Method 2540 D of *Standard Methods*
- Dissolved oxygen by Method 4500-O G of *Standard Methods*
- Water temperature by 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)
- SM 2550B (Temperature)

eAll 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.

Laboratory turnaround time is generally associated with meeting holding times for samples for analysis but will always be within 10 working 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 pursuant to the reporting requirements of the SEP (specifically Revised Appendix I of the Consent Decree).

B5. Quality Control (QC)

B5.1 Dissemination of Quality Requirements

The objectives of the Rogers and Callcott Quality Assurance Program are to determine the quality of the results that are reported through continued monitoring and to control such quality in order to ensure that results meet or exceed the requirements of project. Refer to the Rogers and Callcott Quality Assurance Manual for a comprehensive discussion of the Quality Assurance / Control Program.

Any laboratory staff member observing any occurrence (e.g., equipment failure) that impacts laboratory capabilities or schedule of deliverables 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 5-7. Temperature is measured with a temperature sensor. 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. Temperature should be taken by a calibrated IR thermometer.

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)

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)

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, estimated quantitation limit (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).

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:

$$\mathbf{\%C = \frac{M_v}{M_t} x100}$$

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 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 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]	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 Quanti-Tray

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--24 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-24 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-24 lot..
Internal PE sample	At least one (1) per year	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
%R = percent recovery
MDL = method detection limit
RPD = relative percent difference
QC = quality control
RL = reporting limit where RL = (2.5 mg /mL filtered) x 1000 mL
RSD = relative standard deviation
PE = performance evaluation

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 < 10% RSD	Repeat until acceptable
Balance Calibration Check	Daily, Prior to weighing any sample filters Monthly – 3 weights which must include a 10 mg weight.	Daily: Weight of certified 100 mg weight: 0.998 – 0.1002 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.
Sample analysis	For all sample analyses	Total residue on filter: ≥25 mg to ≤ 200 mg	If total residue on filter < 2.5 mg report result as < RL If total residue on filter > 200 mg filter a smaller volume of sample.
Laboratory Control Sample	one (1) per week	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 Duplicates	One per batch of 10 samples	Precision: The duplicates are within ±5% of their average or 10%RPD.	Investigate. Flag duplicated sample.
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	At least one (1) per year	Determined by PE provider	Investigate all unacceptable results.

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

Table 7. Summary of QC requirements for temperature-DO 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 pH ±0.1 SU Others 75-125% R 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 (pH, DO % saturation of water saturated air) as sample pH ± 0.1 of expected, others 99-101% R	Investigate and fix any obvious problems. Repeat until acceptable.
Calibration check (Lab only)	Immediately following calibration	DO ± 0.2 mg/L expected value from the published oxygen saturation charts	Investigate and fix any obvious problems. Recalibrate and repeat until acceptable.
Thermometer accuracy verification against a NIST-traceable reference thermometer	Annually	Thermometer/thermister being checked must be within 1°C of the reference thermometer	Discard or repair.

B.5.2 Quality Control Activities / Frequency

All instrument calibrations, initial calibrations, and calibration verifications are performed at the method-required frequency. All quality control measures (duplicates, spikes, matrix spike duplicates) are performed at the method required frequency or (if not specified in the method) as specified in the Laboratory's Standard Operating Procedure for that method. These measures are documented in the raw data.

B.5.3 Contingencies for Handling Out-of-Control Data

The procedures to follow when non-conformance issues arise, including corrective action and documentation are stated in the laboratory's quality assurance manual and in the test method's SOP. To the extent possible, samples should be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measures shall be reported with the appropriate data qualifiers.

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	**1**
Autoclave	**1**
Temperature/Dissolved Oxygen/pH Field Meter	**1**
Incubator	**1**
Oven	**1**
Refrigerator/Freezer	**5**
Water deionizing system	**1**
Quanti-Tray sealer	**1**
Water Bath	**1**

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. 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 Unacceptable
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. At least monthly, check balance across entire range of weights. Daily check must be made with a weight which is most nearly the weight of the sample plus the weighing dish.	Monthly (across entire set of weights) / Daily (with weight most nearly sample + dish)	± 0.1%	Clean and autocal or repair
Quanti-Tray sealer	NA	NA	NA	NA
Dissolved Oxygen	One-point calibration with water saturated air	Daily	± 0.2 mg/L expected value, from the oxygen saturation charts	Investigate and correct problem. Repeat calibration until acceptable, if cannot recalibrate repair meter.

B8. Inspection/Acceptance Requirements for Supplies and Consumables

The quality of goods and services received from suppliers and contractors is monitored routinely. Chemicals used by the laboratory are purchased from well-known chemical manufacturers and suppliers and are reviewed by laboratory personnel upon receipt to ensure appropriate quality. The grade of chemical purchased is that specified by the procedure for which it is intended.

The inspection / acceptance requirements for supplies and consumables are documented in the laboratory's SOP for Purchasing, Receiving and Storing Laboratory Chemicals and Supplies.

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)

Historical data from DHEC, collected at the same stations and for the same parameters included in this project, is a considered a non-direct measurement. As previously stated, a goal of this project is to compare the water quality monitoring data collected during these improvement projects to applicable historical data. 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 DHEC historical data is relevant to this project, as this project will mimic the DHEC methodology. This is done intentionally to ensure that results obtained from this project will be directly comparable to the historical results obtained by DHEC.

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 QC Manager
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 we have a 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

Note: The Laboratory Director is responsible for all corrective action. For PTs (PEs), the Laboratory Director documents the success of corrective action and reports that to Laboratory Certification. For audits that are reported to Laboratory Certification, the Laboratory Director responds to Laboratory Certification with the necessary corrections.

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

The lab director will write the reports and they will be received by the Project Manager.

D. Data Validation and Usability

D1. Data Review, Verification and Validation

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

Item	Criteria	If not met sample is accepted, flagged or rejected?	Flag	Comments
Sample not analyzed within hold time	For *E. coli*: Sample analysis should begin as soon as possible after receipt; sample incubation must be started no later than 8 hours from time of collection.	Rejected	N/A	Out of holding time
Lost sample	Proper COC documentation not followed and sample is misplaced	(Unable to analyze)	N/A	N/A
Unable to Collect Sample	Various circumstances (i.e., weather, lost sampling container) cause sample to not be collected	(Unable to analyze)	N/A	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)	Client Contacted	N/A	Noted on sample receipt form
Temperature blank not placed within cooler during sample transport	Unknown receipt temperature	Sample Receipt from notation	N/A	Noted on sample receipt form
Incorrect sampling container used for sample collection	Incorrect sampling container used for sample collection	Sample Receipt from notation	N/A	Noted on sample receipt form
Improper preservation	Improper preservation (i.e., acidification, filtering)	Sample Receipt from notation	N/A	Noted on sample receipt form

D.1.1 Data Review Process

The Laboratory has a process in place for data review. This process is discussed in detail in the Laboratory's Quality Assurance Manual.

The analyst that performed the analysis is responsible for assessing and evaluating all quality control elements associated with the batch before reporting results. To the extent possible, samples should be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples

associated with the failed quality control measures shall be reported with the appropriate data qualifiers.

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.

The Project Verifier will be responsible for Data Verification. The Project Validator will be responsible for Data Validation.

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.

If the Project Validator approves of the report as received, the report will be signed and dated to validate. Should an issue arise during the validation process, the Project Validator will contact the Project Verifier stating the issue. The Project Verifier will provide a written explanation of the issue, take appropriate steps to rectify the issue, and submit an amended report to DHEC via email. The amended report will be also reviewed by the Project Validator, and validated with a signature/date, when appropriate.

E. Revision History

E1. May 2017

Drew Stroud, QA Manager and Project Verifier, is no longer employed by the City of Columbia. Therefore, Ralana Wilson will be assuming these roles. This change affects all the information related to Mr. Stroud, specifically found on pages 2, 5, 6 and 33.

E2. December 2018

Mike Jaspers, Stormwater Manager and QAPP Project Manager/Project Validator, is no longer employed by the City of Columbia. This change removes all references to Mike Jaspers (previously found on pages 2, 5, 6, and 33) and replaces them with the titles only.

- References to Mike Jaspers are now either Stormwater Manager or Project Manager/Project Validator

Ralana Wilson, Stormwater Plan Reviewer and Project Verifier/QA Manager, is no longer employed by the City of Columbia. This change removes all references to Ralana Wilson (previously found on pages 2, 5, 6, and 33) and replaces them with the titles only.

- References to Ralana Wilson are now either Stormwater Plan Reviewer or Project Verifier/QA Manager

Table of Contents was updated to include a List of Tables and a List of Figures.

Distribution List and Organization Chart have been updated with current contact information.

Minor formatting and typographical edits were made throughout.

E3. January 2019

Access Analytical replaced with Rogers and Callcott Environmental. Laboratory procedures, equipment, and quality control were updated as applicable. Rogers and Callcott COC was added as Appendix A.

Section	Starting Page	Revision Description
A3	6	Added the Lab director, responsible for the analysis to the distribution list
A4	6	Removed On-Line Environmental from the Project List Removed Access Analytical from the Project List

		Added Rogers and Callcott
A4	7	Updated Figure 1 – Organizational Chart
A6	10	Updated Table 1 Frequency column to include dates associated with years of the consent decree.
A7.1	12	Updated language under category a.
A7.3	13	Updated language under "Note"
A7.8	15	DO and Water Temperature Accuracy (LCS). Changed DO water accuracy to the correct requirement as follows: Within ±0.2 mg/L of the published saturation at the observed temperature and altitude/barometric pressure. This is in Rogers & Callcott DO SOP.
A8	15	Special Training Requirements and Certification update for Rogers and Callcott
A9	16	Updated language in second paragraph of section. Added "or designee" to those reviewing the QAPP. Revised from reading S.C. DHEC's Quality Assurance Manager.
A9.1	17	Revised data delivery and laboratory record keeping based on Rogers and Callcott methods
B2	18	Removed specification of manufacturer of "calibrated field probes"
B2.1	19	Increased minimum sample side from 500 to 1000mL for TSS Revised language of paragraphs following Table 3
B3	21	Removed "Example for a river sample" from the bulleted information that must be recorded by the sampler Removed "Laboratory Master Technician" from those providing a sample receipt
B3.1	21	Revised language to better reflect Rogers and Callcott equipment and procedures
B3.2	22	Revised language to better reflect Rogers and Callcott equipment and procedures
B3.3	22	Sample Disposal – Destruction of samples are noted on internal COC forms was deleted

B4.1	23	The standard operating procedures documents were adjusted to match the tittles from Rogers and Callcott laboratory Removed comment regarding used or destroyed samples Revised the laboratory turnaround time from 10 days to 10 working days after receipt of samples Added (MPN) to the *E.coli* method
B5.1	22	Updated language to better reflect Rogers and Callcott's procedures and processes Section B 5.1 referred to tables 4-6 for QC. Since Table 4 is statistics, the appropriate tables 5 through 7 were corrected. Table 6 specified that the balance is checked daily with 1 weight and monthly with 3 –one must be a 10 mg. Table 6 did not include sample duplicates. I added. Table 7 includes pH and LDO requirements. Did not include annual accuracy verification for thermometers or temperature sensing devices. Table 7 included LCS. Not required for DO by SM 4500 OG. Only applies to LDO meters. Updated accuracy calculation procedure to better reflect laboratory methods Edited corrective action to reflect which Colilert was being used in Table 5 Revised the TSS acceptance criteria balance calibration check to tighter standards in Table 6 tp: weight of certified 100 mg weight: 0.998-0.1002g Removed the specification of manufacture and specified probe parameters Increased the laboratory control sample minimum frequency from one

		year to one week
B5.2 & B5.3	30	Revised B5.2- Quality Control Activities / Frequency and B5.3 Contingencies for Handling Out-of-Control Data
B6	30	Updated the number of units for each instrument in Table 8
B7	30	Removed the due date for next calibration from the information recorded in the current calibration log
B8	32	Revised Inspection/Acceptance Requirements for Supplies and Consumables
C	33	Deleted paragraph before C1
C1	33	Changed "Laboratory Director" to "Laboratory QC Manager"
D1	34	Updated table to reflect Rogers and Callcott Lab's criteria and the flags that are used for various criteria. Added an overview paragraph (D.1.1) about Rogers and Callcott's Data Review Process
E3	36	Added to revision history
	41	Replaced laboratory SOPs with Rogers and Callcott's DO, *E. coli* , TSS and Temp SOPs

Appendices

A. Chain of Custody (COC) Form

Rogers & Callcott ENVIRONMENTAL

CHAIN OF CUSTODY RECORD

WORK ORDER _____

Mailing Address: PO Box 5655, Greenville, SC 29606, Phone (864) 232-1556
Shipping Address: 426 Fairforest Way, Greenville, SC 29607, Fax (864) 232-6140
215B Stoneridge Drive, Columbia, SC 29210, Phone (803) 509-8999

Client Name _____
Address _____
Report To: _____
Email Address _____
Telephone # _____
PO # _____ Project # _____

R & C WORK ORDER	YR___ DATE	TIME	SAMPLE DESCRIPTION	Total Number of Containers	Parameter(s) →								COMMENTS

Filtered (Yes/No)
Cooled (Yes/No)
Container Type (Plastic/Glass)
Container Volume (mL)
Sample Type (Grab/Composite)
Sample Source (WW, GW, DW, SW, S, Other)

Preservation Code(s)
A – None; E – HCl; I – Zn Acetate
B – HNO_3; F – $Na_2S_2O_3$; J – H_3PO_4
C – H_2SO_4; G – Boric Acid; K – NaH2PO4
D – NaOH; H – Ascorbic Acid; L - _____

SAMPLER – RELINQUISHED BY: 1.	DATE/TIME:	RECEIVED BY: 2.	DATE/TIME:
RELINQUISHED BY: 3.	DATE/TIME:	RECEIVED BY: 4.	DATE/TIME:
RELINQUISHED BY: 5.	DATE/TIME:	RECEIVED BY: 6.	DATE/TIME:
RELINQUISHED BY: 7.	DATE/TIME:	RECEIVED BY: 8.	DATE/TIME:

Composite Start Date: _____
Composite Start Time: _____
Time or Flow (Circle one) Initials: _____

Temperature of blank or representative sample
At time of collection _____ °C
At time of lab receipt _____ °C

Samples remain the property of the Client at all times. In the event samples contain or may contain hazardous materials, Rogers and Callcott shall, after completion of testing and at Client's expense, return such samples to Client or make such samples available for disposal by Client's agent.

Revised June 2018

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B. Standard Operating Procedures (SOPs)

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

E. COLI
COLILERT®
SM 9223B - 2004

1. APPLICABLE MATRIX

This method is approved by the U.S. EPA for monitoring drinking water under the Safe Drinking Water Act. This method is approved for monitoring under the Clean Water.

2. LIMIT OF DETECTION AND QUANTITATION

Reporting limit for e.coli is 1 MPN/100 mLs.

3. SCOPE AND APPLICATION

The enzyme substrate test utilizes hydrolysable substrates for the simultaneous detection of total coliform bacteria and *β-D*-galactosidase, which cleaves the chromogenic substrate, resulting in release of the chromogen. *Escherichia coli* are defined as bacteria giving a positive total coliform response and possessing the enzyme *β*-galactosidase, which cleaves a fluorogenic substrate, resulting in the release of the fluorogen.

Escherichia coli: A fluorogenic substrate, such as 4-methylumbelliferyl- *β-D*-glucuronide (MUG), is used to detect the enzyme *β*-galactosidase, which is produced by *E. coli*. The *β*-glucuronidase enzyme hydrolyzes the substrate and produces a fluorescent product when viewed under long-wavelength (366-nm) ultraviolet (UV) light. The presence of fluorescence indicates a positive test for *E. coli*. Some strains of *Shigella* spp. also may produce a positive fluorescence response. Because *Shigella* spp. are overt human pathogens, this is not considered a detriment for testing the sanitary quality of water.

4. SUMMARY OF METHODS

Total coliforms and E. coli are specifically and simultaneously detected in 24-28 hours, by inoculating the reagent with the water sample and incubating it for 24-28 hours at 35°C ± 0.5°C. The reagent formula contains salts, nitrogen, and carbon sources that are specific for total coliforms. It provides the specific indicator nutrients ONPG (ortho-nitrophenyl-B-D-galactophyranoside) and MUG (4-methylmbelliferyl-B-Dglucuronide) for the target microbes, total coliforms and E. coli. As these nutrients are metabolized, yellow color (from ONPG) and fluorescence (from MUG) are released confirming the presence of total coliforms and E. coli, respectively. Non-coliform bacteria are suppressed and cannot metabolize in indicator nutrients. Consequently, they do not interfere with the specific identification of the target microbes during the test incubation period.

5. DEFINITIONS

Accuracy: the degree of agreement between an observed value and an accepted reference value.

Holding Time: the maximum times that samples may be held prior to analysis and still be considered valid.

Sterility Blank (laboratory reagent blank): an aliquot of sterilized deionized water that is treated exactly as a sampling, including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The sterility blank is used to determine if bacteria are present in the laboratory environment, reagents, or apparatus.

6. INTERFERENCES

Water samples containing humic or other material may be colored. If there is background color, compared inoculated vessel to a control tube containing only water ample. In certain waters, high calcium salt content can cause precipitation, but this should not affect the reaction.

Do not pre-filter samples and place filters in the chromogenic substrate containing vessel. Filtration can concentrate coliforms, but also non-coliforms, heterotrophs, particulates, and certain chemicals (divalent cations, heavy metals, etc.) which can overlay and suppress coliforms, adversely affecting the sensitivity of the test. Coliform bacteria can become trapped in the filter restricting their access to indicator nutrients in the chromogenic substrate reagent and subsequent growth and detection.

Samples are not to be diluted in buffered water. The Colilert® reagent is already buffered and additional buffer compounds can adversely affect the growth of target microbes and test performance.

7. SAFETY

General good laboratory practices are required. Refer to the company's Health and Safety Plan.

As with all polluted waters, treat each sample as a potential health risk. Never mouth pipette. Wash hands before eating.

Consult the SDS for all chemicals and standards used in the analytical process for health hazards and appropriate handling techniques.

All chemicals are to be returned to the proper storage area after use.

Housekeeping is an important aspect of maintaining a safe work environment. All work areas should be cleaned at the end of each workday. All spills should be cleaned up immediately.

Broken glass should be cleaned up and placed in the broken glass bins.

Disinfect lab benches before and after analysis.

Observe strict personal hygiene. Wash hands thoroughly with germicidal soap.

Keep hands away from face.

Place contaminated items (positive samples, cultures, etc.) in bags to be autoclaved prior to disposal.

8. EQUIPMENT AND SUPPLIES

- Incubator able to maintain 35 ± 0.5°C
- Thermometer(s) with 0.2°C graduations for incubation unit(s) immersed in liquid
- High-clarity, sterile, 120 ml polystyrene vessels with 100 ml fill line containing Sodium Thiosulfate
- Color comparator with manufacturer recommended shelf life
- 6 watt 365nm long wave fluorescent UV lamp (Replace lamp annually)
- Quanti-tray 2000
- Quanti-tray sealer

All instruments and apparatus are maintained by Rogers and Callcott employees or outside personnel as appropriate. All such maintenance documented.

9. REAGENTS AND STANDARDS

Colilert for 100 mL samples

Colilert® reagent is stored in a cool (2-30°C), dry place out of direct sunlight per manufacturer requirements. Colilert® medium is used before the manufacturer's stated expiration date. The expiration date for the Colilert® medium is 12 months from the date of manufacture. (Note: Do not use Colilert® – 18 with Quanti-Tray for *E. coli*.)

- Sterility Control – Sterile Deionized Water
- Positive/Positive Control – Eschenichia Coli (ATCC 25922 or 11775)
- Positive/Negative Control – Klebsiella Pneumoniae (ATCC 31488)
- Negative Control – Pseudomonas Aeruginosa (ATCC 10145 or 27853)

All reagents and standards must be stored in appropriately labeled containers.

Commercially prepared reagents and standards are labeled as follows:

Date Received
Date Opened (and analyst's initials)
Expiration Date
Chemical Inventory Number

All commercially prepared standards and reagents from which standards are prepared must have a Certificate of Analysis. The Certificate of Analysis are maintained.

10. SAMPLE COLLECTION, PRESERVATION, SHIPMENT AND STORAGE

Collect at least 100 mL of representative sample in a sterile sample bottle containing sodium thiosulfate. Open the sample container carefully just prior to collection, and close immediately following collection. Do not lay the lid or cap down, and avoid contact near the mouth of the container. Do not "rinse" the container with sample. Do not fill the sample container completely. Maintain enough air space to allow adequate space for mixing the sample prior to analysis.

E. coli samples should be analyzed as soon as possible after collection. Such samples are to be kept at < 10°C during transport and storage. The holding time between sample collection and sample being placed in the incubator is eight hours.

Upon receipt, the samples are checked for proper preservation. If any sample is found to be improperly preserved or handled, the sample custodian will notify the client and initiate corrective action if applicable.

11. QUALITY CONTROL, ACCEPTANCE CRITERIA, DATA ASSESSMENT AND CORRECTIVE ACTION

Rogers and Callcott operates a formal quality control program. Rogers and Callcott maintains performance records to define the quality of the data that is generated.

The temperatures of the incubator, water bath, and refrigerator should be checked twice each working day with at least four hours elapsing between readings. The temperature reading and initials of the analyst are recorded on a sheet appropriate for the device.

Each lot of Colilert® reagent is subjected to positive, negative and sterility control tests before use. Recommended organisms are *E. coli* (ATCC 25922 or 11775), *Klebsiella*-pneumoniae (ATCC 31488), and *Pseudomonas aeruginosa* (ATCC 10145 or 27853).

Each week (or each time that the MMO-MUG media is used if used less than once each week), the laboratory is to run a known positive (*E.coli*) and sterility control using the Colilert® procedure. (The Columbia facility may use a municipal influent as a weekly known positive).

Commercially prepared media that has a shelf life of more than 90 days must be verified every 90 days with known organisms to ensure that media is working properly.

Before use, one sample container and one Quanti-Tray from each lot number is tested for sterility by adding 100 ml sterile water (NOT sterile buffer solution), one packet of Colilert® reagent, mixing and incubating per the procedure described in Section 13. After 24 hours incubation, it is examined for growth. If growth is detected, all sample containers in that lot number are rejected and returned to the supplier for replacement

NOTE: Refer to Microbiology QC Summary SOP for specific quality control requirements for:

- **Equipment** such as Thermometers, Balances, Incubators, Waterbaths, Refrigerators, Freezers, Autoclaves, Volumetric equipment, Glassware, Quantitray sealer, etc.
- **Supplies / Reagents** such as Dilution water, Reagent water, Sample containers, Media, Reference organisms, etc
- **Quality control** such Duplicate counts, Sterility checks, Positive / Negative controls, etc.

The quality control requirements contained in the Microbiology QC Summary SOP are an integral and mandatory part of this method.

The Micro QC SOP summarizes requirements from Standard Methods for the Analysis of Water and Wastewater, EPA Microbiological Methods for Monitoring the Environment, EPA Manual for the Certification of Laboratories Analyzing Drinking Water, TNI Standards, and additional state specific requirements.

Duplicates

Duplicate samples will be treated in exactly the same manner as are samples.

Refer to QC frequency in Summary of Micro Quality Control Summary (Greenville or Columbia facility).

The precision is calculated for duplicate results and compared to the appropriate limits. If the precision limit is exceeded, corrective action must be taken. Corrective action includes qualification of the data.

Precision is calculated by the following:

- Convert the sample and duplicate results to a log.
  - If either value is <1, add 1 to both values before calculating the log
- Calculate the difference between the log values
- Compare the difference to the precision limit

Precision limit = 0.873

12. CALIBRATION AND STANDARDIZATION

Not applicable.

13. PROCEDURE

Before and after analyses, disinfect the work area with 70% alcohol and allow surface to dry.

1) Turn on the power on the Quanti-Tray Sealer. The amber Power Light should illuminate.

2) Allow the Sealer to warm up and the green Ready Light to come on (up to 10 minutes). Sealer will not operate until both the amber power light and the green Ready Light are illuminated, indicating that the unit has reached operation temperature.

3) Aseptically open one unit dose of Colilert® reagent.

4) Add contents of one pack to a 100-ml sample in a sterile vessel.

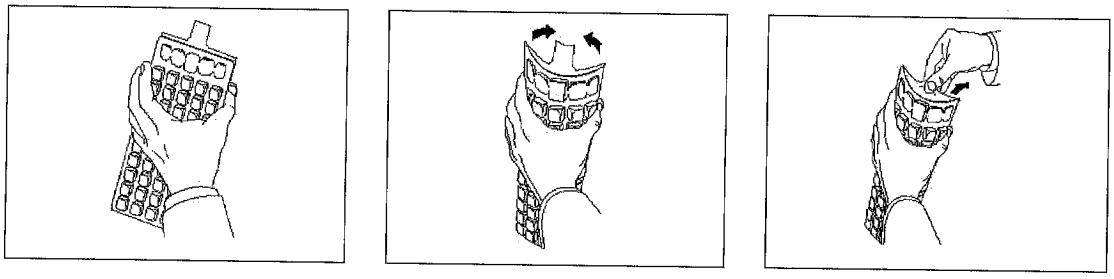
Samples are not to be diluted in buffered water. Samples are to be diluted in sterile deionized water only. The Colilert reagent is already buffered and additional buffer compounds can adversely affect the growth of target microbes and test performance.

5) Cap vessel and shake until dissolved. Allow foam to settle.

6) Use one hand to hold a Quanti-Tray upright with the well side facing the palm.

7) Squeeze the upper part of the Quanti-Tray so that the Quanti-Tray bends toward the palm.

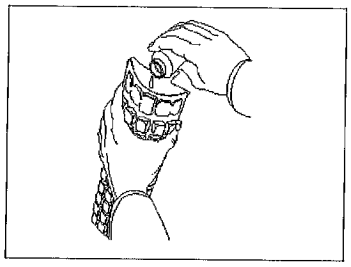
8) Open the Quanti-Tray by pulling the foil tab away from the well side. Avoid touching the inside of the foil or tray.

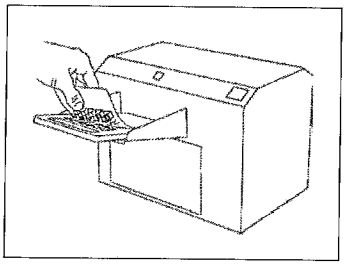


9) Pour the reagent/sample mixture directly into the Quanti-Tray, avoiding contact with the tab. Allow them to settle.

10) Place an empty Quanti-Tray 2000 Rubber Insert on the Input Shelf with the large cut-out facing away from the Sealer.

11) Place a Quanti-Tray 2000 filled with sample and reagent onto the Rubber Insert, making sure that the Tray is properly seated in the Rubber Insert, and with each well of the Tray in its corresponding Rubber Insert hole.





12) Slide the Rubber Insert with Tray into the Sealer until the motor grabs the Rubber Insert and begins to draw it into the Sealer.

13) In approximately 15 seconds, the Tray will be sealed and partially ejected from the rear of the Sealer. Remove the Rubber Insert and Tray from the rear of the Sealer.

14) If at any time, you wish to reverse the motor drawing the Rubber Insert into the Sealer (for example, if a misaligned Tray is accidentally fed into the Sealer), press and hold the Reverse Button. However, do not reverse the motor once the Rubber Insert has been drawn fully into the Input Slot.

15) Multiple Rubber Inserts can be run consecutively without pausing.

Note: Turn off Sealer when not in use.

16) Place sealed tray(s) in incubator at 35°C ± 0.5 °C for 24 hours.
Note: Do not stack more than 10 trays high. Do not arrange in a box pattern.

17) Read results according to the Result Interpretation table below. Count the number of positive wells and refer to the MPN table provided with the trays to obtain a Most Probable Number.

Result Interpretation

Appearance	Result
Less yellow than the comparator	Negative for total coliforms and *E. coli*
Yellow equal to or greater than the comparator	Positive for total coliforms
Yellow and fluorescence equal to or greater than the comparator	Positive for *E. coli*

Note: Look for fluorescence with a 6-watt, 365-nm UV light within 5 inches of the sample in a dark environment. Face light away from your eyes and towards the sample.

When analyzing samples for e. coli, mark the yellow wells and then observe the marked wells

for fluorescence under UV light.

Colilert® results are definitive at 24-28 hours. In addition, positives for both total coliforms and *E. coli* observed before 24 hours and negatives observed after 28 hours are also valid.

The comparator is the lowest level of yellow and fluorescence that can be considered positive. A typical positive test is much more intense than the comparator.

If a sample is yellow after 24 hours for Colilert®-24 of incubation, but slightly less than the positive comparator or indeterminate, it may be incubated up to an additional 4 hours (but no more than 4 hours). If the sample is coliform positive, the color will intensify. If it does not intensify, record the sample as negative. If the sample color remains indeterminate, the laboratory must consider the sample as invalid and request another sample from the same location. 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.

If an inoculated Colilert® vessel is inadvertently incubated more than 28 hours for Colilert®-24, the test is invalid and another sample from the same location must be collected and tested.

14. DATA ANALYSIS AND CALCULATION

Quanti-Tray 2000 – Count the number of large and small positive wells and use IDEXX MPN Generator to calculate results.

The Quanti-Tray 2000 counts from 1 to 2,419.

15. METHOD PERFORMANCE

Refer to Performance Testing results.

16. POLLUTION PREVENTION

Tubes or plates which have been used for microbial analysis may contain concentrated amount of potentially harmful bacteria. Sterilize used tubes of medium in an autoclave before disposal.

Reagents and known positive/negative microorganisms are prepared in quantities consistent with laboratory use to minimize the amount of expired reagents and microorganisms to be disposed.

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

To the extent possible, samples should be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measures shall be reported with

the appropriate data qualifiers.

If quality control measures are not within quality control limits, and it is appropriate the data will be qualified. If data qualification is not appropriate, the samples will be re-analyzed or another sample will be collected if possible.

18. WASTE MANAGEMENT

Samples and wastes may be disposed of on-site or returned to the client for disposal.

Non-hazardous aqueous samples and waste: Non-hazardous aqueous waste may be flushed down the sink with water. Metal samples and digestates are neutralized with sodium bicarbonate before being flushed down the sink with water.

Non-hazardous soil samples: Non-hazardous soil samples may be disposed of in the dumpster.

Hazardous samples: Hazardous samples are returned to the client (with a copy of the laboratory report) for disposal unless prior arrangements have been made.

Hazardous / Regulated Waste: Hazardous and regulated waste shall be disposed of by the hazardous waste contractor. Refer to the Waste Disposal Standard Operating Procedure.

Plates with microbial growth and microorganism cultures are autoclaved and then disposed.

19. REFERENCES

Clesceri, L.S., M.A.H. Franson, A.E. Greenberg, and R.R. Trussell, Eds., APHA, AWWA, WPCF, Standard Methods for the Examination of Water and Wastewater, SM9223B - 2004.

IDEXX Laboratories, Inc., Colilert® Instruction.

20. TABLES, DIAGRAMS, FLOWCHARTS

None

SOP Revision History

Method:	E.Coli - MPN	
Method Reference:	SM 92223B	
Revision	**Revision Date**	**Revision**
Typographical/formatting corrections.	March 2011	1
Update Method Reference	December 2012	2
Clarify Test Organisms Update quality control section	January 2016	3
Correct typographical error in lamp wavelength	March 2016	4
Update Quality Control - Duplicates	January 2017	5
Update wording of Quality Control Section	March 2017	6

OXYGEN, DISSOLVED
MEMBRANE ELECTRODE
STANDARD METHODS 4500-OG - 2011

1. APPLICABLE MATRIX

This method is applicable to wastewaters and streams and other aqueous matrices.

2. LIMIT OF DETETION AND QUANTITATION

The reporting limit is 1.0 mg/L (0.1 mg/L for field analysis).

3. SCOPE AND APPLICATION

Standard Methods 4500O-OG- 2001 with 2011 editorial revisions is approved for Clean Water Act Monitoring.

The probe method for dissolved oxygen is recommended for those samples containing materials which interfere with the modified Winkler procedure such as sulfite, thiosulfate, polythionate, mercaptans, free chlorine or hypochlorite, organic substances readily hydrolyzed in alkaline solutions, free iodine, intense color or turbidity and biological flocs.

The probe method is recommended as a substitute for the modified Winkler procedure in monitoring of streams, lakes, outfalls, etc., where it is desired to obtain a continuous record of the dissolved oxygen content of the water under observation.

The probe method may be used under any circumstances as a substitute for the modified Winkler procedure provided that the probe itself is standardized against the Winkler method on samples free of interfering materials.

4. SUMMARY OF METHOD

The most common instrumental probes for determination of dissolved oxygen in water are dependent upon electrochemical reactions. Under steady-state conditions, the current or potential can be correlated with DO concentrations. Interfacial dynamics at the probe-sample interface are a factor in probe response and a significant degree of interfacial turbulence is necessary. For precision performance, turbulence should be constant.

5. DEFINITIONS

Accuracy: the degree of agreement between an observed value and an accepted reference value.

Batch: environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents.

A **preparation batch** is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours.

An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Corrective Action: the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent the recurrence.

Holding Time: the maximum times that samples may be held prior to analysis and still be considered valid.

Precision: measure of the degree of agreement among duplicate analyses of a sample.

6. INTERFERENCES

Plastic films used with membrane electrode systems are permeable to a variety of gases besides oxygen, although none is depolarized easily at the indicator electrode. Prolonged use of membrane electrodes in waters containing such gases as hydrogen sulfide (H_2S) tends to lower cell sensitivity. Eliminate this interference by frequently changing and calibrating the membrane electrode.

7. SAFETY

General good laboratory practices are required. Refer to the company's Health and Safety Plan.

As with all polluted waters, treat each sample as a potential health risk. Never mouth pipette. Wash hands before eating.

Consult the MSDS for all chemicals and standards used in the analytical process for health hazards and appropriate handling techniques.

All chemicals are to be returned to the proper storage area after use.

Housekeeping is an important aspect of maintaining a safe work environment. All work areas should be cleaned at the end of each workday. All spills should be cleaned up immediately.

Broken glass should be cleaned up and placed in the broken glass bins.

8. EQUIPMENT AND SUPPLIES

Oxygen-sensitive membrane electrode, polarographic or galvanic, with appropriate meter.

Instrument Operation:

Note: Refer to manufacturer's instructions for proper use, calibration, and storage.

Fisher Scientific Digital Barometer

Preparing the instrument

1. Enter Rogers and Callcott Laboratory altitude (Greenville, SC is1006 ft./310 meters)
2. The millibars Hg reading is multiplied by 0.750062 to convert to mm Hg for entering data into dissolved oxygen meter.

YSI Model 5905 BOD Probe

Probe Preparation

All YSI probes are shipped dry. You must follow these instructions when preparing a new probe or changing membranes. Prepare the electrolyte by dissolving the KCl crystals in the dropper bottle filled to the neck with deionized water. Then proceed as follows:

Using YSI 5906 Cap Membranes for the YSI 5905 BOD Probe:

1. Remove the stirring mechanism by pulling it straight out.
2. Fill the membrane cap with KCl electrolyte.
3. Position the sensor over the cap and slowly push the sensor into the membrane while tightening at the same time.
4. Make sure no air bubbles are visible under membrane cap.
5. Replace stirring mechanism. Probe is now ready for use.

YSI Model 5000 Dissolved Oxygen Meter

Preparing the Instrument

It is important that the instrument be placed in the intended operating position - vertical, tilted, or on its back - before it is prepared for use and calibrated. Readjustment may be necessary when the instrument operating position is changed. After preparing the probe, proceed as follows:

1. As specified in the dissolved oxygen meter manual, enter the salinity value for the

calibration environment (0.0 ppt for air calibration).
2. Enter the barometric pressure obtained from barometer (millibars Hg from barometer converted to mm Hg by multiplying by 0.750062).
3. Before calibrating, allow 15 minutes for optimum probe stabilization. Repolarize whenever the instrument has been OFF or the probe has been disconnected.

Orion Star Meter Series (4, A326, A111)

Prior to calibration, the probe must be prepared and polarized.

1. The DO probe is continuously polarized when connected to the meter. When first connected, or if more than 60 minutes has elapsed with the probe disconnected, re-connect the probe and allow 30 to 60 minutes for polarization. If the probe readings are stable, probe disconnections of less than one hour will require 5 to 25 minutes for polarization.
2. The probes are shipped dry. Fill the membrane cap with KCl electrolyte.
3. Position the sensor over the cap and slowly push the sensor into the membrane while tightening at the same time.
4. Make sure no air bubbles are visible under membrane cap.

9. REAGENTS AND STANDARDS

Deionized Water.

10. SAMPLE COLLECTION, PRESERVATION, SHIPPING AND STORAGE

Because membrane electrodes offer the advantage of analysis in situ, they eliminate errors caused by sample handling and storage.

Samples should be analyzed within 15 minutes of collection.

11. QUALITY CONTROL, DATA ASSESSMENT, ACCEPTANCE CRITERIA, AND CORRECTIVE ACTION

The DO meter should be verified weekly against the Modified Winkler Method (Refer to Winkler SOP). The meter and the Winkler Method should agree within ± 0.2 mg/L, if not, corrective action must be taken.

Corrective Action may include:
Cleaning DO probe (refer to manufacturer's instructions)
Re-analysis by Winkler Method
Re-calibration of DO meter
Change probe membrane
Preparation of new Winkler reagents
Repair of DO meter

Temperature Calibration

Perform a temperature probe calibration check against an NIST traceable thermometer annually. Document the check.

Precision Control (Duplicates):

Not applicable

12. CALIBRATION AND STANDARDIZATION

Air Calibration – YSI Meter

1. Gently wipe any moisture off the sensor with a Chem-Wipe. Place the probe in a BOD bottle containing about 1" of water to provide a 100% relative humidity.
2. Press [0] to turn instrument on.
3. Allow the probe to polarize and the temperature to stabilize for at least 15 minutes. If calibration is performed prematurely, the values will drift and may be out of specification.
4. Press the [CALIBRATE] soft key to change to calibration mode.
5. Press the [DO CAL] soft key.
6. Press the [NEXT] soft key 3 times to achieve a “blinking” barometric pressure reading.
7. Enter the barometric pressure by using the [UP] and [DOWN] soft keys. The barometric pressure reading (in millibars) is taken from the digital barometer and converted to mm Hg by multiplying by 0.750062.
8. Press [ENTER] to save setting. (“Pressure Setting Saved” should be displayed at bottom of screen for a few seconds).
9. Press [AUTO CAL] soft key (“D.O. Calibration Saved” should be displayed at bottom of screen for a few seconds). If an error message is displayed, then the probe has not stabilized and needs more time, or there is a membrane problem.
10. Press [MODE] to return to the main mode. The instrument is now calibrated and ready to measure dissolved oxygen and temperature. Calibration can be disturbed by physical shock, touching the membrane, or drying out of the electrolyte.
11. A beginning and end calibration, as well as a continuing calibration verification after every ten samples (including temperature and dissolved oxygen reading) are recorded in a workbook. The dissolved oxygen and temperature readings are obtained from a full BOD bottle of dilution water (without nutrients), called the Calibration Blank, which is changed weekly. The end calibration and continuing calibration verification dissolved oxygen readings ideally should be within 0.2 mg/l of initial reading.

Air Calibration – Orion Star Meter (4 Star)

1. Inspect the DO membrane.
2. In the set-up mode select DO.
3. Moisten the sponge in the calibration sleeve with deionized water and insert the probe

into the sleeve, but without touching the water saturated material.
4. Select Air calibration.
   Air calibration is performed in water saturated air using the air calibration sleeve. This is the simplest and most accurate calibration method. The air saturation standard is set to 102.3% saturation.
5. Press calibrate. Wait for the reading to stabilize.
6. After calibration the meter will return to the measurement mode.

Air Calibration – Orion Star Meter (A326, A111)

1. Inspect the DO membrane.
2. In the select DO channel.
3. Moisten the sponge in the calibration sleeve with deionized water and insert the probe into the sleeve, but without touching the water saturated material.
4. Select Air calibration.
   Air calibration is performed in water saturated air using the air calibration sleeve. This is the simplest and most accurate calibration method. The air saturation standard is set to 102.3% saturation.
5. Press start. Wait for the reading to stabilize.
6. After calibration, press cal done, and then press measure.

13. PROCEDURE

Sample measurement:

Follow all precautions recommended by manufacturer to insure acceptable results. Take care in changing membrane to avoid contamination of sensing element and also trapping of minute air bubbles under the membrane, which can lead to lowered response and high residual current. Provide sufficient sample flow across membrane surface to overcome erratic response. Rinse the probe with deionized water between each sample reading.

Dissolved Oxygen Measurement:

With the instrument prepared for use and the probe calibrated, place the probe in the sample to be measured and turn on the stirrer (if applicable). Read dissolved oxygen in mg/L from the display.

Field

The DO meter must be calibrated in the laboratory to ensure that the meter is working properly. Upon arrival at each site, the meter must be calibrated to account for changes in altitude. (Both the lab and field calibration are recorded in the calibration log.)

14. DATA ANALYSIS AND CALCULATION

Read the dissolved oxygen from the meter in mg/L.

15. METHOD PERFORMANCE

Refer to Standard Methods 4500 – OG-2011.

16. POLLUTION PREVENTION

Reagents and standards are prepared in quantities consistent with laboratory use to minimize the amount of expired reagents and standards to be disposed.

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

To the extent possible, samples should be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measures shall be reported with the appropriate data qualifiers.

If quality control measures are not within quality control limits, and it is appropriate the data will be qualified. If data qualification is not appropriate, the samples will be re-analyzed or another sample will be collected if possible.

18. WASTE MANAGEMENT

Samples and wastes may be disposed of on-site or returned to the client for disposal.

Non-hazardous aqueous samples and waste: Non-hazardous aqueous waste may be flushed down the sink with water. Metal samples and digestates are neutralized with sodium bicarbonate before being flushed down the sink with water.

Non-hazardous soil samples: Non-hazardous soil samples may be disposed of in the dumpster.

Hazardous samples: Hazardous samples are returned to the client (with a copy of the laboratory report) for disposal unless prior arrangements have been made.

Hazardous / Regulated Waste: Hazardous and regulated waste shall be disposed of by the hazardous waste contractor. Refer to the Waste Disposal Standard Operating Procedure.

19. REFERENCES

U.S. EPA Methods for Chemical Analysis of Water and Wastes, EPA-600/4-70-020, Revised

March, 1983, Method 360.1

Standard Methods for the Examination of Water and Wastewater, Method 4500-OG-2001 with 2011 editorial revisions.

20. TABLES, DIAGRAMS, FLOWCHARTS

Refer to Table 1.

Table 1

Dissolved oxygen saturation values

Solubility of oxygen in water at various temperatures and pressures
[In milligrams per liter. Values based on Weiss (1970). C, degrees Celsius; mmHg, millimeters of mercury]

Temp. C	Atmospheric pressure, mmHg																					
	760.0	750.0	740.0	730.0	720.0	710.0	700.0	690.0	680.0	670.0	660.0	650.0	640.0	630.0	620.0	610.0	600.0	590.0	580.0	570.0
0	14.6	14.4	14.2	14.0	13.8	13.6	13.4	13.2	13.0	12.8	12.7	12.5	12.3	12.1	11.9	11.7	11.5	11.3	11.1	10.9
1	14.2	14.0	13.8	13.6	13.4	13.2	13.1	12.9	12.7	12.5	12.3	12.1	11.9	11.7	11.6	11.4	11.2	11.0	10.8	10.6
2	13.8	13.6	13.4	13.3	13.1	12.9	12.7	12.5	12.3	12.2	12.0	11.8	11.6	11.4	11.2	11.1	10.9	10.7	10.5	10.3
3	13.4	13.3	13.1	12.9	12.7	12.5	12.4	12.2	12.0	11.8	11.7	11.5	11.3	11.1	10.9	10.8	10.6	10.4	10.2	10.0
4	13.1	12.9	12.7	12.6	12.4	12.2	12.0	11.9	11.7	11.5	11.3	11.2	11.0	10.8	10.7	10.5	10.3	10.1	10.0	9.8
5	12.7	12.6	12.4	12.2	12.1	11.9	11.7	11.6	11.4	11.2	11.1	10.9	10.7	10.5	10.4	10.2	10.0	9.9	9.7	9.5
6	12.4	12.3	12.1	11.9	11.8	11.6	11.4	11.3	11.1	10.9	10.8	10.6	10.4	10.3	10.1	9.9	9.8	9.6	9.5	9.3
7	12.1	12.0	11.8	11.6	11.5	11.3	11.1	11.0	10.8	10.7	10.5	10.3	10.2	10.0	9.9	9.7	9.5	9.4	9.2	9.1
8	11.8	11.7	11.5	11.3	11.2	11.0	10.9	10.7	10.6	10.4	10.2	10.1	9.9	9.8	9.6	9.5	9.3	9.1	9.0	8.8
9	11.5	11.4	11.2	11.1	10.9	10.8	10.6	10.5	10.3	10.2	10.0	9.8	9.7	9.5	9.4	9.2	9.1	8.9	8.8	8.6
10	11.3	11.1	11.0	10.8	10.7	10.5	10.4	10.2	10.1	9.9	9.8	9.6	9.5	9.3	9.2	9.0	8.9	8.7	8.6	8.4
11	11.0	10.9	10.7	10.6	10.4	10.3	10.1	10.0	9.8	9.7	9.5	9.4	9.2	9.1	9.0	8.8	8.7	8.5	8.4	8.2
12	10.8	10.6	10.5	10.3	10.2	10.0	9.9	9.8	9.6	9.5	9.3	9.2	9.0	8.9	8.7	8.6	8.5	8.3	8.2	8.0
13	10.5	10.4	10.2	10.1	10.0	9.8	9.7	9.5	9.4	9.3	9.1	9.0	8.8	8.7	8.5	8.4	8.3	8.1	8.0	7.8
14	10.3	10.1	10.0	9.9	9.7	9.6	9.5	9.3	9.2	9.0	8.9	8.8	8.6	8.5	8.4	8.2	8.1	7.9	7.8	7.7
15	10.1	9.9	9.8	9.7	9.5	9.4	9.3	9.1	9.0	8.8	8.7	8.6	8.4	8.3	8.2	8.0	7.9	7.8	7.6	7.5
16	9.8	9.7	9.6	9.5	9.3	9.2	9.1	8.9	8.8	8.7	8.5	8.4	8.3	8.1	8.0	7.9	7.7	7.6	7.5	7.3
17	9.6	9.5	9.4	9.3	9.1	9.0	8.9	8.7	8.6	8.5	8.3	8.2	8.1	8.0	7.8	7.7	7.6	7.4	7.3	7.2
18	9.4	9.3	9.2	9.1	8.9	8.8	8.7	8.6	8.4	8.3	8.2	8.0	7.9	7.8	7.7	7.5	7.4	7.3	7.2	7.0
19	9.3	9.1	9.0	8.9	8.8	8.6	8.5	8.4	8.3	8.1	8.0	7.9	7.8	7.6	7.5	7.4	7.3	7.1	7.0	6.9
20	9.1	8.9	8.8	8.7	8.6	8.5	8.3	8.2	8.1	8.0	7.8	7.7	7.6	7.5	7.4	7.2	7.1	7.0	6.9	6.7
21	8.9	8.8	8.6	8.5	8.4	8.3	8.2	8.1	7.9	7.8	7.7	7.6	7.5	7.3	7.2	7.1	7.0	6.9	6.7	6.6
22	8.7	8.6	8.5	8.4	8.2	8.1	8.0	7.9	7.8	7.7	7.5	7.4	7.3	7.2	7.1	7.0	6.8	6.7	6.6	6.5
23	8.6	8.4	8.3	8.2	8.1	8.0	7.9	7.7	7.6	7.5	7.4	7.3	7.2	7.0	6.9	6.8	6.7	6.6	6.5	6.4
24	8.4	8.3	8.2	8.0	7.9	7.8	7.7	7.6	7.5	7.4	7.3	7.1	7.0	6.9	6.8	6.7	6.6	6.5	6.3	6.2
25	8.2	8.1	8.0	7.9	7.8	7.7	7.6	7.5	7.3	7.2	7.1	7.0	6.9	6.8	6.7	6.6	6.4	6.3	6.2	6.1
26	8.1	8.0	7.9	7.8	7.6	7.5	7.4	7.3	7.2	7.1	7.0	6.9	6.8	6.7	6.5	6.4	6.3	6.2	6.1	6.0
27	7.9	7.8	7.7	7.6	7.5	7.4	7.3	7.2	7.1	7.0	6.9	6.7	6.6	6.5	6.4	6.3	6.2	6.1	6.0	5.9
28	7.8	7.7	7.6	7.5	7.4	7.3	7.2	7.1	6.9	6.8	6.7	6.6	6.5	6.4	6.3	6.2	6.1	6.0	5.9	5.8
29	7.7	7.6	7.5	7.3	7.2	7.1	7.0	6.9	6.8	6.7	6.6	6.5	6.4	6.3	6.2	6.1	6.0	5.9	5.8	5.7
30	7.5	7.4	7.3	7.2	7.1	7.0	6.9	6.8	6.7	6.6	6.5	6.4	6.3	6.2	6.1	6.0	5.9	5.8	5.7	5.6
31	7.4	7.3	7.2	7.1	7.0	6.9	6.8	6.7	6.6	6.5	6.4	6.3	6.2	6.1	6.0	5.9	5.8	5.7	5.6	5.5
32	7.3	7.2	7.1	7.0	6.9	6.8	6.7	6.6	6.5	6.4	6.3	6.2	6.1	6.0	5.9	5.8	5.7	5.6	5.5	5.4
33	7.2	7.1	7.0	6.9	6.8	6.7	6.6	6.5	6.4	6.3	6.2	6.1	6.0	5.9	5.8	5.7	5.6	5.5	5.4	5.3
34	7.0	6.9	6.8	6.7	6.7	6.6	6.5	6.4	6.3	6.2	6.1	6.0	5.9	5.8	5.7	5.6	5.5	5.4	5.3	5.2
35	6.9	6.8	6.7	6.6	6.5	6.4	6.3	6.3	6.2	6.1	6.0	5.9	5.8	5.7	5.6	5.5	5.4	5.3	5.2	5.1

SOP Revision History

Method:	Oxygen Dissolved
Method Reference:	4500-OG

Revision	**Revision Date**	**Revision**
Clarification to Section 12 – Air Calibration procedure.	March 2011	2
Updated Standard Methods Reference Update Quality Control Section	November 2012	3
Updated to include A326 and A111 meter	January 2016	4
Removed previous SC DHEC duplicate requirement for samples that are not measured in-situ per SC DHEC January 2016 audit	March 2016	5

TEMPERATURE
LABORATORY AND FIELD METHOD
STANDARD METHODS 2550B

1. APPLICABLE MATRIX OR MATRICES

This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes.

2. DETECTION LIMIT

The reporting limit is dependent upon the thermometer used.

3. SCOPE AND APPLICATION

Temperature readings are used in the calculation of various forms of alkalinity, in studies of saturation and stability with respect to calcium carbonate, in the calculation of salinity, and in general laboratory operations. In limnological studies, water temperatures as a function of depth often are required. Elevated temperatures resulting from discharges of heated water may have significant ecological impact. Identification of source of water supply, such as deep wells, often is possible by temperature measurements alone. Industrial plants often require data on water temperature for process use or heat-transmission calculations.

This method is approved for Safe Drinking Water Act monitoring and Clean Water Act monitoring.

4. SUMMARY OF METHOD

Temperature measurements may be made with any good grade of non-mercury or dial type centigrade thermometer, or a thermistor.

5. DEFINITIONS

Accuracy: the degree of agreement between an observed value and an accepted reference value.

Batch: environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents.

A **preparation batch** is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours.

An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch

can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Corrective Action: the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent the recurrence.

Duplicate Analysis: the analyses or measurements performed identically on two sub-samples of the same sample. The results of the duplicate analyses are used to evaluate analytical or measurement precision.

Holding Time: the maximum times that samples may be held prior to analysis and still be considered valid.

Material Safety Data Sheet (MSDS): written information provided by the vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

Precision: measure of the degree of agreement among duplicate analyses of a sample.

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.

6. INTERFERENCES

None.

7. SAFETY

General good laboratory practices are required. Refer to the company's Health and Safety Plan.

As with all polluted waters, treat each sample as a potential health risk. Never mouth pipette. Wash hands before eating.

Consult the MSDS for all chemicals and standards used in the analytical process for health hazards and appropriate handling techniques.

All chemicals are to be returned to the proper storage area after use.

Housekeeping is an important aspect of maintaining a safe work environment. All work areas should be cleaned at the end of each workday. All spills should be cleaned up immediately.

Broken glass should be cleaned up and placed in the broken glass bins.

8. EQUIPMENT AND SUPPLIES

Normally, temperature measurements may be made with any good non-mercury filled Celsius thermometer. As a minimum, the thermometer should have a scale marked for every 0.1°C, with markings etched on the capillary glass. The thermometer should have a minimal thermal capacity to permit rapid equilibration. For field operations use a thermometer having a metal case to prevent breakage.

9. REAGENTS AND STANDARDS

None.

10. SAMPLE COLLECTION , PRESERVATION AND HANDLING

Temperature should be analyzed immediately.

11. QUALITY CONTROL, ACCEPTANCE CRITERIA, AND CORRECTIVE ACTION

Rogers and Callcott operates a formal quality control program.

Precision Control (Duplicates):

Not applicable.

Accuracy Control (Spikes):

Not applicable.

12. CALIBRATION AND STANDARDIZATION

Annually check the thermometer against a precision thermometer certified by the National Institute of Standards and Technology (NIST) that is used with its certificate and correction chart. NIST thermometer must be verified by an external company once every 5 years.

13. PROCEDURE

Calibrate annually any temperature measurement devices with an NIST-certified thermometer before field use. Make readings with the thermometer or device immersed in water long enough to permit complete equilibration. Report results to the nearest 0.1 or 1.0°C, depending on need.

Note: Any thermometer that has a correction factor >1°C must be replaced.

14. CALCULATION

Not applicable.

15. METHOD PERFORMANCE

Refer to annual calibration records.

16. POLLUTION PREVENTION

Reagents and standards are prepared in quantities consistent with laboratory use to minimize the amount of expired reagents and standards to be disposed.

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

To the extent possible, samples should be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measures shall be reported with the appropriate data qualifiers.

If quality control measures are not within quality control limits, and it is appropriate the data will be qualified. If data qualification is not appropriate, the samples will be re-analyzed or another sample will be collected if possible.

18. WASTE MANAGEMENT

Samples and wastes may be disposed of on-site or returned to the client for disposal.

Non-hazardous aqueous samples and waste: Non-hazardous aqueous waste may be flushed down the sink with water.

Hazardous samples: Hazardous samples are returned to the client (with a copy of the laboratory report) for disposal unless prior arrangements have been made.

19. REFERENCES

Standard Methods for the Examination of Water and Wastewater, Method 2550B - 2000.

Standard Methods for the Examination of Water and Wastewater, Method 2550B - 2010.

U.S. EPA Methods for Chemical Analysis Water and Wastes, EPA-600/4-79-020, Revised March, 1983, Method 170.1.

20. TABLES, DIAGRAMS, FLOWCHARTS

Not applicable.

SOP Revision History

Method:	Temperature
Method Reference:	2550B

Revision	**Revision Date**	**Revision**
Update Standard Method Reference	November 2012	2
Update Standard Method Reference for SDWA	August 2013	3
Added Method Reference	January 2016	4
Added NIST calibration frequency Added correction factor criteria	March 2016	5

RESIDUE, NON-FILTERABLE
(TOTAL SUSPENDED SOLIDS DRIED AT 103°C – 105°C)
STANDARD METHODS 2540D - 2011

1. APPLICABLE MATRIX

This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes. This method is approved by the U.S. EPA for Clean Water Act monitoring (1997 with 2011 editorial revisions).

2. LIMIT OF DETECTION AND QUANTITATION

The practical range of the determination is 1 mg/L to 20,000 mg/L. The reporting limit is dependent upon the amount of sample filtered. If 1 liter of sample is filtered, the reporting limit is 1.0 mg/L. (For NC: If 1 liter of sample is filtered, the reporting limit is 2.5 mg/L.)

3. SCOPE AND APPLICATION

Solids refer to matter suspended or dissolved in water or wastewater. Solids may affect water or effluent quality adversely in a number of ways. Waters with high dissolved solids generally are of inferior palatability and may induce an unfavorable physiological reaction in the transient consumer. For these reasons, a limit of 500 mg dissolved solids/L is desirable for drinking waters. Highly mineralized waters also are unsuitable for many industrial applications. Waters high in suspended solids may be esthetically unsatisfactory for such purposes as bathing. Solids analyses are important in the control of biological and physical wastewater treatment processes and for assessing compliance with regulatory agency wastewater effluent limitations.

4. SUMMARY OF METHOD

A well-mixed sample is filtered through a standard glass fiber filter, and the residue retained on the filter is dried to a constant weight at 103-105°C. The increase in weight of the filter represents the total suspended solids. If the suspended material clogs the filter and prolongs filtration, it may be necessary to increase the diameter of the filter or decrease the sample volume.

5. DEFINITIONS

Batch: environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents.

An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Corrective Action: the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent the recurrence.

Duplicate Analysis: the analyses or measurements performed identically on two sub-samples of the same sample. The results of the duplicate analyses are used to evaluate analytical or measurement precision.

Holding Time: the maximum times that samples may be held prior to analysis and still be considered valid

Method Blank (laboratory reagent blank): an aliquot of deionized water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, and internal standards that are used with other samples. The method blank is used to determine if method analytes or other interferences are present in the laboratory environment, reagents, or apparatus.

Precision: measure of the degree of agreement among duplicate analyses of a sample.

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.

6. INTERFERENCES

Filtration apparatus, filter material, pre-washing, and drying temperature are specified because these variables have been shown to affect the results.

Exclude large floating particles or submerged agglomerates of non-homogeneous materials from the sample if it is determined that their inclusion is not representative. Because excessive residue on the filter may form a water-entrapping crust, limit the sample size to that yielding no more than 200 mg/L residue.

Samples high in Filterable Residue (dissolved solids) such as saline waters, brines and some wastes, may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing of the filter and any dissolved solids in the filter minimizes this potential interference.

7. HEALTH AND SAFETY

General good laboratory practices are required. Refer to the company's Health and Safety Plan.

As with all polluted waters, treat each sample as a potential health risk. Never mouth pipette. Wash hands before eating.

Consult the SDS for all chemicals and standards used in the analytical process for health

hazards and appropriate handling techniques.

All chemicals are to be returned to the proper storage area after use.

Housekeeping is an important aspect of maintaining a safe work environment. All work areas should be cleaned at the end of each workday. All spills should be cleaned up immediately.

Broken glass should be cleaned up and placed in the broken glass bins.

8. EQUIPMENT AND SUPPLIES

- Glass fiber filter discs - Environmental Express Proweigh (47 mm and 90 mm) filter
- Membrane filter funnel with reservoir (suction flask) and a coarse fritted disc as filter support.
- Drying oven (103-105°C)
- Desiccator with color indicating dessicant.
- Tongs or forceps.
- Analytical balance, capable of weighing to 0.1 mg.
- If volatile suspended solids are to be determined also: Furnace capable of attaining 550°C

9. REAGENTS AND STANDARDS

Sigma Cell Cellulose Type 20

10. SAMPLE COLLECTION , PRESERVATION, SHIPMENT AND STORAGE

Upon receipt, the samples are checked for proper preservation. If any sample is found to be improperly preserved or handled, the sample custodian will notify the client and initiate corrective action if applicable.

Sample must be preserved within 15 minutes of collection by cooling. Sample must be shipped and stored at ≤ 6°C and not frozen.

Maximum holding time is 7 days.

Sample may be collected in either glass or plastic (polyethylene or fluoropolymer) containers.

11. QUALITY CONTROL, ACCEPTANCE CRITERIA, DATA ASSESSMENT, AND CORRECTIVE ACTION

Rogers and Callcott operates a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, the analysis of laboratory reagent blanks, fortified blanks and other laboratory solutions as a continuing

check on performance. Rogers and Callcott maintains performance records to define the quality of the data that is generated.

Demonstration of Capability

An initial demonstration of capability is performed prior to the analysis of samples and with a significant change in the instrument, personnel, or test method.

An IDOC is performed by analyzing four standards prepared from Sigma Cell in same manner as LCS.

IDOC Acceptance Limits:

Average % Recovery – 80-120
RPD Limit – 15

Ongoing demonstration of capability is performed by routine quality control such as Laboratory Control Samples and Performance Testing.

Method Blank / Laboratory Reagent Blank

Method blanks are performed at a frequency of at least one per preparation batch. The method blank is processed along with the samples to include all steps of the analytical procedure. The method blank is used to assess the preparation batch for possible contamination during the preparation and processing steps. Blank values that exceed the MRL indicate laboratory or reagent contamination and should be suspected and corrective actions must be taken. Corrective actions may include reanalysis of the batch or data qualification (as appropriate).

Laboratory Control Sample (Blank Spike / Laboratory Fortified Blank)

Once a week, the method is verified using Sigma Cell Cellulose Type 20 by weighing approximately 0.05 g (record exact weight) of the standard, and dissolving in 100 mL of DI water, analyzing in the same manner as a sample. The final result should recover within ±20% of the amount initially weighed.

If the recovery is not within the control limits, the batch should be reanalyzed. If re-analysis is not appropriate due to issues such as insufficient sample or holding time, etc., the data should be qualified or a re-sample obtained.

Quality Control Sample

An external blind quality control sample is analyzed annually.

Precision Control (Duplicates):

A well-mixed sample will be split for duplication for precision control, prior to any sample pretreatment of method process. Duplicate samples will be treated in exactly the same manner as are samples.

At a minimum, one sample per matrix from every analysis batch will be duplicated for precision control. Total duplicated samples will equal or exceed a rate of 10 percent of samples analyzed.

Duplicates should agree within ±5% of the average weight. If the TSS result is less than 25 mg/L then the sample / duplicate result should agree within ± 2 mg/L of the average.

If the duplicate exceeds these limits, corrective action must be taken which may include reanalysis of the batch. If the cause can be documented why the duplicate exceeded limits and no other sample analyses were affected, sample batch reanalysis may be omitted with supervisor's approval. Samples which have precision control problems will be documented for reporting purposes.

Continuing Calibration Verification

A weight in the working range is to be weighed when the tare weights and gross weights of samples are taken. This weight should be within acceptance criteria of ±2% as specified in the Gravimetric Analysis: Balance Calibration Check SOP (unless other acceptance criteria are specified in the reference method). If the weight is not within the acceptance criteria, then corrective action must be taken. Corrective action may include: re-zeroing the balance, re-weighing the weight, checking the weight for damage, leveling the balance, and notifying a manager that the balance needs servicing.

12. CALIBRATION AND STANDARDIZATION

The analytical balance calibration is checked with at least three weights each day used, and recorded in the balance logbook. The balance calibration is also checked at the first of each month using seven or more weights covering the working range. The weights are recorded in the balance logbook. All check weights are NIST traceable. The check weights should not vary by more than 2% from the weight.

A service contract is maintained on the balances. Each balance is serviced and calibrated annually or more often if needed, by an outside firm.

The analyst will check the oven thermometer annually against an NIST traceable thermometer.

13. PROCEDURE

Selection of Sample Volume

Choose sample volume to yield between 2.5 mg and 200 mg dried residue. If the volume filtered fails to meet the minimum yield, increase sample volume up to 1L. If complete

filtration takes more than 10 minutes, increase filter diameter or decrease sample volume.
Assemble the filtering apparatus and begin suction. Wet the filter with a small volume of deionized water to seat it against the fritted support.
Shake the sample vigorously and quantitatively transfer the predetermined sample volume selected to the filter using a graduated cylinder. Remove all traces of water by continuing to apply vacuum after sample has passed through.

With suction on, wash the graduated cylinder, filter, non-filterable residue and filter funnel wall with three portions of 10 mLs of deionized water allowing complete drainage between washing. Remove all traces of water by continuing to apply vacuum for about 3 minutes after water has passed through. (Note: for a 9.0 cm filter – wash, with three portions of 20 mLs of deionized water.)

NOTE: Samples with high dissolved solids may require additional washing.

For sample volume less than 100 mLs, use a 100 mL graduated cylinder to measure volume, for sample volume less than 25 mLs, use a 25 mL graduated cylinder to measure volume, and for sample volume less than 10 mLs, put aliquot of sample in beaker and stir, use a 10 mL or 5 mL wide-tipped pipette to measure volume. Do not pipette volume less than 1ml, perform a primary dilution prior to sample analysis.

NOTE: If sample is high in dissolved solids, it should be filtered using a 9.0 cm filter.

Carefully remove the filter from the filter support. Dry at least one hour at 103-105°C. Cool in a desiccator for at least one hour and weigh. Repeat the drying / dessicating cycle until a constant weight is obtained (weight change is less than 0.5 mg).

If Volatile Solids are to be determined, refer to SOP for EPA 160.4 / SM 2540E - 2011.

14. DATA ANALYSIS AND CALCULATION

Calculate Total Suspended Solids (Non-filterable Residue) as follows:

$$TSS = \frac{(A - B)}{C} * 1000 * 1000$$

Where: A = weight of filter + residue in grams
B = weight of filter in grams
C = mL of sample filtered

15. METHOD PERFORMANCE

Method performance is monitored through blanks, laboratory control standards, and performance testing.

16. POLLUTION PREVENTION

Reagents and standards are prepared in quantities consistent with laboratory use to minimize the amount of expired reagents and standards to be disposed.

17. CONTINGENCIES FOR HANDLING OUT-OF-CONTROL OR UNACCEPTABLE DATA

To the extent possible, samples should be reported only if all quality control measures are acceptable. If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measures shall be reported with the appropriate data qualifiers.

If quality control measures are not within quality control limits and it is appropriate, the data will be qualified. If data qualification is not appropriate, the samples will be re-analyzed or another sample will be collected if possible.

18. WASTE MANAGEMENT

Samples and wastes may be disposed of on-site or returned to the client for disposal.

Non-hazardous aqueous samples and waste: Non-hazardous aqueous waste may be flushed down the sink with water.

Hazardous samples: Hazardous samples are returned to the client (with a copy of the laboratory report) for disposal unless prior arrangements have been made.

Hazardous / Regulated Waste: Hazardous and regulated waste shall be disposed of by the hazardous waste contractor. Refer to the Waste Disposal Standard Operating Procedure.

19. REFERENCES

U.S. EPA Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March, 1983, Method 160.2.

Standard Methods for the Examination of Water and Wastewater, Method 2540D – 1997 with 2011 editorial revisions.

20. TABLES, DIAGRAMS, FLOWCHARTS

Not applicable to this method.

SOP Revision History

Method:	Residue, Non-Filterable (Total Suspended Solids, TSS)
Method Reference:	2540 D

Revision	Revision Date	Revision
Update Quality Control Section 11 Update Standard Method Reference	November 2012	2
Update duplicate frequency	October 2014	3
Remove reference to filter preparation since pre-washed / pre-weighed filters are used.	April 2015	4
Update Quality Control Section	November 2015	5
Added "prepared in same manner as LCS" to DOC per SC DHEC.	March 2016	6