



**Procedures for Chain-of-Custody,
SOP Development, and Common Field
Parameter Measurements
(DO, pH, Chlorine, Temperature)**

**Office of Environmental Laboratory
Certification**

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TABLE OF CONTENTS

STANDARD OPERATING PROCEDURES (SOPS) FORMAT 3

TRAINING – DEMONSTRATION OF CAPABILITY 5

PROFICIENCY TESTING (PT) REQUIREMENTS 5

CHAIN-OF-CUSTODY 6

DISSOLVED OXYGEN (DO)..... 7

HYDROGEN-ION CONCENTRATION (pH) – SM 4500H+B-2011 10

RESIDUAL CHLORINE – SM 4500Cl G-2011 13

TEMPERATURE - SM 2550B-2010 20

RECORDS AND TRACEABILITY 22

REFERENCES 22

EXAMPLE RECORDS

Thermometer Accuracy Verification Record..... 23

Temperature Record Chart..... 24

Hydrogen-Ion Concentration (pH) Calibration..... 25

Hydrogen-Ion Concentration (pH) Sample Analysis..... 26

Residual Chlorine Initial Calibration Verification..... 27

Total / Free Residual Chlorine1 Calibration and Sample Analysis 28

Dissolved Oxygen Calibration and Sample Analysis for SM 4500 OG-2011 29

STANDARD OPERATING PROCEDURES (SOPS) FORMAT

Technical SOPs are written instructions on how to perform a specific analytical method and/or task in the field or laboratory. SOPs are also used to explain how to collect a sample and preserve it to maintain the sample integrity. The SOP must explain all aspects associated with an analysis. More information concerning technical SOPs can be found at <https://www.epa.gov/sites/production/files/2015-06/documents/g6-final.pdf>

Include on the title page and in the header of each SOP the following information:

Laboratory Name:
SOP ID Number:

Revision Date:
Revision Number:
Issue Date:

Use the following format for developing a technical SOP:

Parameter Name
EPA Approved Method Reference

- A. Scope and Applicability
Describes the purpose of the process or procedure, types of samples analyzed, and any organization or regulatory requirements, as well as any limits to the use of the procedure.
- B. Summary of the Method
Provides a brief explanation of the procedure.
- C. Definitions
Defines any acronyms, abbreviations, or specialized terms used in the methodology and SOP.
- D. Health & Safety
Includes operations and/or chemicals that could result in personal injury or loss of life and explains what will happen if the procedure is not followed or is followed incorrectly.
- E. Interferences
Describes any component of the process that may interfere with the accuracy of the final product. Includes physical or chemical properties of the sample.
- F. Sample Handling, Preservation, and Storage
Documents the sampling and collection requirements for each type of sample (NPDES, Drinking Water, and Solid and/or Hazardous Waste). Specifies the maximum required holding time from the time of collection along with the temperature and chemical preservative. Documents the storage conditions for sample analysis.
- G. Instrumentation and Equipment
Lists all instrumentation and equipment that are necessary to perform the analysis/test. Specifies the manufacturer and model number of all instruments.

- H. Reagents, Standards, and Consumable Materials
Lists all reagents, standards, and materials needed to perform the analysis/test. Specifies the source by documenting the manufacturer, catalog number, concentration, and any other pertinent information.
- I. Standard and/or Reagent Preparation
Documents the preparation procedure for all standards and reagents. Specifies the concentration at which all standards and/or reagents are prepared and the storage conditions and maximum holding times for all standards and reagents. Specifies the procedure for necessary standardization of reagents and the frequency performed.
- J. Instrument Calibration Procedures
Documents the calibration procedures and frequency of calibration for each instrument. Includes initial calibration and calibration verification procedures along with the acceptance criteria for a valid calibration.
- K. Sample Analysis Procedures
Documents step-by-step procedures for analyzing routine and quality control samples.
- L. Quality Control
Includes all laboratory quality control practices along with the frequency and acceptance criteria for all QC samples and calibration verifications. Describes the initial Demonstration of Capability (DOC) and on-going demonstration of capability for all analysts. Includes the procedures for the determination of Method Detection Limits (MDLs), if applicable.
- M. Data Reduction, Validation, and Reporting
Describes data reduction, calculations, and reporting procedures.
- N. References
Includes the references used to develop the SOP and any pertinent instrument manuals.
- O. Waste Management
Provides information concerning proper waste disposal procedures for samples and/or reagents and supplies.
- P. Revision History
Documents the section(s) that have been modified since the last revision. Provides an ongoing history of the SOP modifications.

The revision history should include the following information:

SOP Revision #	Revision Date	Section Modified	Modification	Reason Changed

- Q. Appendix
Include all appendices, tables, diagrams, etc.

TRAINING – DEMONSTRATION OF CAPABILITY

Training records must be maintained for all personnel. These records should include all job-related formal education and training taken by the analyst that pertains to any aspect to his/her responsibilities, including but not limited to analytical methodology, SOP review, laboratory safety, sampling, quality assurance, and data analysis.

An initial demonstration of capability (IDOC) must be performed for each method and each analyst. It is a demonstration of the precision and accuracy required by the method. As stated in SM 1020B.1, each analyst must conduct an IDOC at least once before analyzing any sample to demonstrate proficiency in performing the method and obtaining acceptable results for each parameter .

An ongoing or continuing demonstration of capability (CDOC) is used to document an analyst's continued proficiency with a method by meeting the precision and accuracy requirements. (Refer to SM 1020B.3 for guidance)

PROFICIENCY TESTING (PT) REQUIREMENTS

Proficiency Testing Requirements can be found on our website at www.scdhec.gov/labcert. The required proficiency testing parameters are also specified. If you have any questions, contact the Office of Environmental Laboratory Certification at (803)896-0970 or by email at labcerthelp@dhec.sc.gov.

CHAIN-OF-CUSTODY

Each certified laboratory must have written instructions for sample collection and handling in its standard operating procedures manual. To ensure sample integrity, it is required that an accurate written record (chain-of-custody) be available to trace the possession and handling of samples from the moment of collection through analysis and final disposition. This is referred to as chain-of-custody and is important to ensure sample integrity and will be used in the event of litigation involving the sample results. Chain-of-custody records must include the following:

- 1) Sample collection date and time.
- 2) Sample collector's signature.
- 3) Unique sample identification number (Assigned by the laboratory upon receipt).
- 4) Sample location and description.
- 5) Sample type - grab or composite.
- 6) Number of containers per analysis received.
- 7) Container type - glass or plastic.
- 8) Analyses required.
- 9) Preservatives used - H₂SO₄, NaOH, ice, etc.
- 10) Program area - WW, DW, GW, etc.
- 11) Sample matrix - liquid, soil, waste, etc.
- 12) Transfer signatures w/dates and times for relinquishing and laboratory receipt. This includes relinquishment to a laboratory refrigerator for temporary storage or other secure location.
- 13) Receipts maintained when shipped by common carrier (FEDEX, UPS, etc.).
- 14) Temperature upon receipt in the laboratory.
- 15) Contracted analyses must be documented with the name of contract laboratory.

For composite samples, the starting and ending date and time for the compositing period must be noted on the chain-of-custody record. Composite samples must be maintained at $\leq 6^{\circ}\text{C}$ during the sampling period. Many facilities use refrigerated samplers. If a refrigerated sampling device is not utilized, ice must be used and replenished as needed to ensure that the sampling environment is maintained at $\leq 6^{\circ}\text{C}$ during the 24 hour composite sampling period. The temperature of the sampling environment must be documented at the time the sample is harvested either in field records or on the chain-of-custody form.

Grab and composite samples must be preserved properly within 15 minutes of collection. The required containers, preservation techniques, and holding times for specific NPDES parameters are listed in Table II of 40 CFR Part 136. All chemical preservatives must be traceable to the sample container and analysis requested. If proper preservation cannot be determined from the chain-of-custody and laboratory verification (e.g. pH and chlorine), the analytical results will be invalid.

Chain-of-custody is not required for field analyses (pH, DO, residual chlorine, and temperature) or any other analysis performed at the collection site (such as specific conductance), since the analyses are performed at the same time that the samples are collected. The instrumentation (brand/model) and reagents used must be traceable to the field measurements performed. Dates and times of collection and analysis must also be traceable to field analysis results.

DISSOLVED OXYGEN (DO)

Membrane Electrode Method SM 4500OG-2011
Luminescence Method ASTM D888-09C and HACH 10360 Rev. 1.2

Separate certification is required for the membrane electrode and luminescence DO Methods.

A. Equipment and Supplies:

- Meter and membrane electrode probe or LDO probe.
- DO or LDO probe must be stored according to the manufacturer's instructions.

B. Holding Time:

Samples must be analyzed within 15 minutes of collection (40 CFR Part 136 Table II). It is strongly recommended that samples be analyzed *in situ* or analyzed immediately upon collection.

C. Calibration:

Membrane Electrode Method: Follow the manufacturer's calibration procedures to obtain acceptable precision and accuracy. The DO meter must be calibrated daily (each work shift) at the certified laboratory location prior to sample analysis in the field or laboratory. Calibration records must reflect the calibration procedures required by the manufacturer. Documentation of all calibrations must be maintained by the laboratory and must be readily available for review.

The records must, at a minimum, include the following information provided it is applicable to the instrument in use:

- Laboratory name
- Laboratory ID #
- Meter ID
- Date/Time of calibration
- Air temperature during calibration
- Adjusted/Final DO value
- Theoretical DO value - obtained from DO tables at the observed temperature and corrected for altitude/pressure
- Analyst's initials
- Membrane check – indicates the laboratory has inspected the membrane and it is in operable condition (no tears, bubbles, folding, etc...)
- Comment/Maintenance – issues, membrane replacement, etc...

Additional information may be required, such as meters that require the operator to input the local altitude, barometric pressure, and/or the salinity of the water:

- Red-line check
- Altitude
- Altitude adjustment
- Barometric pressure
- Salinity
- % Saturation

Luminescent Dissolved Oxygen (LDO) Method: Follow the calibration procedures in the approved method. The following information must be recorded:

- Laboratory name
- Laboratory ID #
- Meter ID
- Date/Time of Calibration
- Theoretical DO value - obtained from DO tables and corrected for altitude/pressure
- Calibration Verification (LCS/LCSD) % recovery - The LCS/LCS duplicate samples are water-saturated air (or air-saturated water) samples that must be analyzed with each sample batch. Refer to the approved method for the acceptance criteria and analytical requirements.
- Analyst's initials
- Comments/maintenance – membrane cap, probe, and battery replacement

D. Sample Analysis:

The sample analysis records must include, at a minimum, the following information:

- Laboratory name
- Laboratory ID#
- Date/Time of sample collection
- Date/Time of sample analysis (sample collection and analysis time would be the same for samples analyzed *in situ*).
- Sample ID
- Dissolved oxygen (DO) reading in mg/L
- Meter ID
- Analyst's initials
- Comments

E. Quality Control:

- Zero check - Performed if required by the manufacturer. Document the results in the calibration records.
- The DO probe's temperature sensor must be checked for accuracy at least annually using a NIST-traceable reference thermometer. This is required even if the meter is not used for reporting temperature. If the temperature difference from the reference thermometer is more than 1.0°C, then the probe must be repaired or replaced.
- If the meter has an internal barometer, the barometer must be verified for accuracy monthly.
- Calibration verification - verify the instrument's calibration by using the original calibration value and using the instrument supplied solubility tables to confirm that the instrument reads within $\pm 2\%$ or ± 0.2 mg/L.
- Initial Demonstration of Capability (IDC): Required for new analysts to demonstrate proficiency in performing a new method. The IDC is also used to demonstrate proficiency with new equipment.

HYDROGEN-ION CONCENTRATION (pH) – SM 4500H+B-2011

This procedure is for the analysis of pH in drinking water or wastewater using a pH meter capable of reading 0 - 14 standard units (SU), in 0.1 SU increments with a hydrogen-ion electrode.

A. Equipment and Supplies

- pH meter and pH electrode

B. Reagents:

Standard Buffer Solutions: Commercially or laboratory prepared buffer solutions of known value.

- Commercially prepared buffer containers are labeled with received date, opened date, expiration date, and analyst initials.
- Laboratory prepared buffer containers are labeled with pH value, lot #, prepared date, expiration date, and preparer's initials.
- Buffers are used within the manufacturer's expiration date. If the manufacturer fails to provide an expiration date, the laboratory must assign an expiration date of one year after opening in the laboratory.
- Buffer solutions prepared by the laboratory from pH powder pillows or liquid concentrates can be used up to one year from the date that they are prepared. Pillow liquid concentrates must not have exceeded the manufacturer's expiration date.
- Buffers transferred to secondary containers are labeled with pH value, lot number, and expiration date.
- Buffers are not used more than once, a fresh aliquot is used each time the meter is calibrated or if a buffer is checked between calibrations.

Wash solution: Deionized or distilled water, preferably in a squeeze bottle.

C. Holding Time:

Samples must be analyzed within 15 minutes of collection (40 CFR Part 136 Table II). It is strongly recommended that samples be analyzed *in situ* or analyzed immediately upon collection.

D. Calibration:

Daily Calibration: The pH meter must be calibrated daily (each work shift) at the certified laboratory location prior to sample analysis in the field or laboratory. The instrument must be calibrated according to the manufacturer's instructions using a minimum of two, preferably three different pH buffer concentrations.

- The pH buffers used must not differ by more than 3 Standard Units (SU). (For example, pH 4 and 10 buffers are more than 3 SU apart. To cover the range of pH 4 to 10, the meter should be calibrated with 4, 7, and 10 pH buffers.)
- The pH buffers used for calibration must bracket the expected sample pH value. (For example, if the analyst expects the pH of the sample to be 8.9, then pH 7.0 and 10.0 buffers should be used for the calibration.)

Calibration Verification: The meter's calibration must be verified after each calibration with a fresh aliquot of pH buffer. The buffer used to check the calibration will be analyzed in the same manner as a sample and must read within 0.1 SU of the buffer's true value. For example, after the meter is calibrated (standardized) with pH buffers 4, 7, and 10, a pH 7.0 buffer is analyzed and the result must be between 6.9 and 7.1 SU. Otherwise the calibration is not valid.

Calibration records must document, at a minimum, the following information:

- Laboratory name
- Laboratory ID #
- Meter ID
- Date/time of calibration
- pH buffers used for calibration/standardization
- Lot numbers of pH buffers
- Electrode slope or efficiency value (if displayed by the meter) – refer to electrode manufacturer's manual
- Reading of the calibration verification pH buffer check (must be within 0.1 SU of the true value)
- Comments (problems, maintenance, electrode replacement, etc.)
- Analyst's initials

E. Sample Analysis:

Sample analysis records must be traceable to the daily calibration records. The sample analysis records must include, at a minimum, the following information:

- Laboratory name
- Laboratory ID #
- Meter ID
- Date of analysis
- Time of sample collection
- Time of sample analysis (sample collection and analysis time would be same for samples analyzed *in situ*)
- Sample ID
- pH value of sample
- pH value of sample duplicate (see Quality Control section below)
- Analyst's initials
- Comments

F. Quality Control

- Calibration – Refer to Section D.
- Calibration Verification – Refer to Section D.
- Initial Demonstration of Capability (IDC) – Required for new analysts to demonstrate proficiency in performing a new method. The IDC is also used to demonstrate proficiency with new equipment.
- Proficiency Testing (PT) Sample – Required annually.
- Sample Duplicates – Collect an aliquot of sample and split the aliquot into two separate containers for analysis. The first aliquot is the regulatory compliance sample, the results of which are recorded and used for reporting purposes. The second aliquot is analyzed and results recorded for quality control purposes. A duplicate sample is performed with each batch of samples. If however, the facility only analyzes one sample per day, it is acceptable to perform only one duplicate sample per week. If the pH samples are analyzed *in situ*, a duplicate is not performed. Sample duplicate results must not be reported or averaged with the original sample result. Duplicate acceptance criteria is typically ± 0.2 S.U.
- Temperature Sensor Calibration Verification – If the instrument is equipped with an automatic temperature compensator (ATC), it is recommended that the device be checked for accuracy against a certified NIST-traceable reference thermometer. This is required if the laboratory is reporting temperature directly from the pH meter. See the “TEMPERATURE” section of this document for details.

G. Continuous Monitoring pH – EPA 150.2

Some laboratories may be using continuous monitoring instruments for the measurement of sample pH. The use of such instruments is approved for compliance monitoring as long as they are operating in accordance with the accepted EPA analysis criteria. Therefore, such instrumentation must be calibrated at least daily to ensure the accuracy of the reported values. Appropriate records of the daily calibrations must be kept to verify that the checks have been performed.

Many of the continuous monitoring instruments now on the market can easily be directly calibrated on a daily basis. Direct calibration of any instrument is always preferred. However, the Department acknowledges that direct daily calibration of some of the continuous monitoring devices currently marketed can be difficult, if not impossible.

As an alternative to direct daily calibration, it will be acceptable for a laboratory to compare a sample value from the continuous monitoring instrument to a sample value obtained from a benchtop instrument that has been properly calibrated each day. For pH analysis, the value obtained from the continuous monitor must be within ± 0.2 S.U. of the value obtained from the benchtop instrument.

RESIDUAL CHLORINE – SM 4500Cl G-2011

This method is used for the analysis of free and/or total residual chlorine for drinking water or wastewater samples using the DPD spectrophotometric or colorimetric method. The South Carolina total residual chlorine reporting limit (practical quantitation limit-PQL) for wastewater is 0.05 mg/L.

A. Equipment

- Spectrophotometer or colorimeter
- Class A volumetric glassware and pipets
- Analytical balance (accurate to 0.0001g)

B. Standards – Refer to Section H. for examples of standard preparation.

Potassium Permanganate (KMnO₄) Primary Stock Standard - laboratory or commercially prepared 1000mg/L (0.891g KMnO₄/L) standard solution.

Laboratory Prepared:

- Prepare at least every 6 months
- Store in a glass container protected from light
- Store in a cool dry place
- Label with reagent name, concentration, date prepared, and analyst's initials

Commercially Prepared:

- Store in a container protected from light
- Once opened, stock standard solution shelf-life is six months or manufacturer's expiration date, whichever comes first
- Label with date received, date opened, six month expiration date, and analyst's initials
- Store as recommended by manufacturer

Potassium Permanganate Secondary Solution - prepare a 100mg/L solution by diluting 10ml of the primary stock to 100 ml with DI water.

- 100mg/L secondary solution must be prepared at least weekly
- Store in a glass container protected from light
- Store in a cool dry place
- Label with reagent name, concentration, date prepared, and analyst's initials

Potassium Permanganate Working Standard Solutions (< 100mg/L) - prepare initial and daily calibration verification standards using the secondary solution.

- Prepare daily, preferably use immediately
- A minimum of two working standards used for daily calibration verification
- A minimum of five working standards used for initial calibration verification
- Class A volumetric glassware and pipettes (or mechanical pipettes) must be used.

Chlorine Standards - commercially prepared liquid chlorine standard solutions may be used to prepare working standards in place of the potassium permanganate standards. Chlorine standards must be prepared according to the manufacturer's instructions using Class A volumetric glassware and pipettes and used immediately. The preparation of chlorine standards will differ from the preparation of potassium permanganate standards.

Commercially obtained secondary gel standards ($\geq 1.0\text{mg/L}$) – The concentration established by the manufacturer must be used. The gel standards are not as accurate as potassium permanganate or chlorine primary standards, therefore gel standards (at the concentration of $\geq 1.0\text{mg/L}$) may **ONLY** be used for calibration verification after every 10 samples and at the end of the sample analysis batch. **Potassium permanganate (or liquid chlorine) standards must always be used for daily (each work shift) calibration verification prior to sample analysis.**

C. Holding Time

Samples must be analyzed within 15 minutes of collection (40 CFR Part 136 Table II).

D. Calibration

Note: When using potassium permanganate standards, the meter must be zeroed using reagent water, not the standard.

Initial Calibration Verification: Initial calibration verification is performed with a blank and at least 5 standards (potassium permanganate or chlorine liquid standards only, gel standards cannot be used for the ICV) covering the concentration range for sample analyses. One of the standards must be at the reporting limit concentration. Initial calibration verification is required for each meter on an annual basis or when a new lot number of DPD reagent is used, whichever comes first.

Separate initial calibration verifications must be performed for free and total DPD reagents for each meter in use.

A blank is required to be analyzed with the initial and daily calibration verification. The same source of DI water that is used to prepare the standards must be used to zero the meter prior to the analysis of the standards. For the blank, ensure the DPD reagent is added to the DI water. The reading of the blank after this process must be documented on the records.

All observed meter readings for the 5 standards must be within $\pm 10\%$ of the standard's true value (or $\pm 20\%$ for 0.05 mg/L standard). If the standards do not meet the acceptance criteria, new secondary and/or primary standard solutions must be prepared and a new calibration verification performed.

Each initial calibration verification record must be labeled with the following:

- Laboratory name
- Laboratory ID #
- Identify analysis as free or total residual chlorine
- Date and time of calibration
- Analyst's initials
- Instrument ID
- Lot number of DPD reagent
- True concentration of standards, (mg/L)
- Observed concentration (meter readings) or absorbance of standards, (mg/L)
- Blank reading, (mg/L)
- Wavelength (if adjustable)
- Cell path length (if interchangeable)
- Stock Standard ID
- Intermediate Standard ID

If chlorine gel standards are in use, these must be verified with each initial calibration verification and read within $\pm 10\%$ of the manufacturer's assigned value.

Daily Calibration Verification Criteria: The instrument's calibration must be verified daily (each work shift) at the certified laboratory location prior to sample analysis in the field or laboratory, after each batch of 10 samples, and at the end of the run.

The daily calibration verification must be performed before any samples are analyzed using a minimum of two standards and a blank (potassium permanganate or chlorine liquid standards only, gel standards cannot be used for the daily calibration verification performed prior to sample analysis). For wastewater analyses, one of the standards must be at the reporting limit of 0.05mg/L.

After each batch of 10 samples, and at the end of the run verify the calibration with a single mid-level standard (potassium permanganate, chlorine liquid standards, and chlorine gel standards may be used).

The daily calibration verification must be traceable to the initial calibration verification and sample analysis records. Traceability is established by the date, time, meter ID, standard ID, and DPD reagent lot number.

All observed meter readings must be within 10% of the standard's true value (20% for the 0.05mg/L standard). If standards do not meet the acceptance criteria, freshly prepared

secondary and/or primary standard solutions must be used to perform a new calibration verification.

Each daily calibration verification record must be labeled with the following information:

- Laboratory name
- Laboratory ID #
- Identify analysis as free or total residual chlorine
- Instrument ID
- Lot number of DPD Reagent
- Date of initial calibration verification
- Date and time of daily calibration verification
- Blank reading, (mg/L)
- True concentration of standards (mg/L)
- Observed concentration of the standards (mg/L)
- Analyst's initials
- Stock Standard ID
- Intermediate Standard ID

E. Sample Analysis:

Sample analysis records must be traceable to the daily calibration verification records. The instrument's calibration must be verified each work shift before conducting sample analysis.

Documentation of all sample analyses must be readily available for review and must, at a minimum, include the following information:

- Laboratory name
- Laboratory ID #
- Identify analysis as free or total residual chlorine
- Instrument ID
- Lot number of DPD Reagent (must be traceable to initial and daily calibration verification)
- Sample Identification
- Date and time of collection
- Date and time of analysis
- Sample residual chlorine value, mg/L
- Sample duplicate result, mg/L
- Analyst's initials
- Comment and/or sample dilution section

F. Sample Reporting:

When reporting sample results, the laboratory cannot report values that are lower than the lowest standard concentration used to generate the initial calibration verification. For example, if the lowest standard used to perform the initial calibration verification is 0.05 mg/L and if the meter reads less than 0.05 mg/L, then the analyst must report the value as "<0.05 mg/L". The South Carolina minimum reporting level for wastewater is 0.05 mg/L.

Samples with results above the highest standard concentration used to generate the initial calibration verification must be diluted.

G. Quality Control:

- Daily Calibration Verification – See Section D
- Initial Calibration Verification – See Section D
- Initial Demonstration of Capability (IDC) – Required for new analysts to demonstrate proficiency in performing a new method. The IDC is also used to demonstrate that the laboratory allowed modifications to a method will produce results as accurate and precise as the reference method.
- Proficiency Testing (PT) Sample – Required annually.
- Quality Control Sample (QCS) – Externally generated, blind QCS (unknown concentration) at least annually, preferably semi-annually, or quarterly.
- Sample Duplicates – Collect an aliquot of sample and split the aliquot into two separate containers for analysis. The first aliquot is the regulatory compliance sample, the results of which are recorded and used for reporting purposes. The second aliquot is analyzed and results recorded for quality control purposes. A duplicate sample is performed with each batch of samples. If however, the facility only analyzes one sample per day, it is acceptable to perform only one duplicate sample per week. Sample duplicate results must not be reported or averaged with the original sample result. Typical acceptance criteria is $\pm 10\%$.
- Mechanical Pipette Calibration Verification – If using a mechanical pipette for preparation of standards and/or samples must be verified for accuracy annually.
- Analytical Balance Calibration Verification – If using an analytical balance for preparation of the potassium permanganate the following requirements must be met and documentation maintained for the analytical balance:
 - An annual service contract is required to calibrate the balance and ensure its accuracy. It is recommended that the service contract be obtained with an ISO17025 accredited company for this service.
 - Monthly calibration verification of the analytical balance is required using at least three ASTM Type 1, Class 1 or 2 (or equivalent) weights covering the range of measurements being performed.
 - Daily calibration verification of the analytical balance is required on the day of use with at least one ASTM Type 1, Class 1 or 2 (or equivalent) weight in the range of the measurements being performed.
 - ASTM Type 1, Class 1 or 2 (or equivalent) weights must be recertified every 5 years or more frequently if damage has occurred. The South

Carolina Department of Agriculture Metrology Lab can perform this recertification of weights.

- Method Detection Limit (MDL) Study – An annual MDL study is required for spectrophotometers when using absorbance and concentration to construct a calibration curve. Refer to 40 CFR Part 136, Appendix B, Revision 2.

H. Examples of Standard Preparation of Potassium Permanganate Standards for Residual Chlorine Calibration Verification

Stock Standard (1000mg/L): This standard is good for six months from the date of preparation.

To make (mg/L)	Use this standard:	Use this much	Final volume
1000mg/L	Potassium Permanganate Crystals	0.891g	1000 ml

Intermediate Standard (100mg/L): This standard is good for one week after preparation.

To make (mg/L)	Use this standard:	Use this much	Final volume
100mg/L	Stock Standard (1000mg/L)	10mL	100 ml

Daily Standard Preparation: These standards must be prepared fresh immediately prior to use. These are example dilutions only, other dilutions can be utilized. Laboratories are not limited to the following concentrations.

To make (mg/L)	Use this standard:	Use this much	Final volume
8 mg/L	Intermediate Standard (100mg/L)	8 ml	100 ml
7 mg/L	Intermediate Standard (100mg/L)	7 ml	100 ml
6mg/L	Intermediate Standard (100mg/L)	6 ml	100ml
5 mg/L	Intermediate Standard (100mg/L)	5 ml	100 ml
4 mg/L	Intermediate Standard (100mg/L)	4 ml	100 ml
3 mg/L	Intermediate Standard (100mg/L)	3 ml	100 ml
2 mg/L	Intermediate Standard (100mg/L)	2 ml	100 ml
1.5 mg/L	Intermediate Standard (100mg/L)	1.5 ml	100 ml
1 mg/L	Intermediate Standard (100mg/L)	1 ml	100 ml
0.5 mg/L	Intermediate Standard (100mg/L)	0.50 ml	100 ml
0.2 mg/L	2 mg/L Daily Standard	10 mL	100 mL
0.1 mg/L	1 mg/L	10 ml	100 ml
0.05 mg/L	0.5 mg/L	10 ml	100 ml

Notes:

- 1) All glassware that is used to make standards should be thoroughly rinsed with distilled water prior to use. If it becomes discolored from permanganate, the glassware can be washed with a dilute acid solution.
- 2) All standards must be prepared with either Class volumetric pipettes or by an automatic pipetter which has been verified for accuracy at the volumes used. The accuracy verification must be performed annually. All volumes must be within $\pm 2.5\%$ of the expected volume.

TEMPERATURE - SM 2550B-2010

A. Equipment

Thermometers and temperature measuring devices used for monitoring or reporting temperature must be checked for temperature accuracy against a NIST-traceable reference thermometer. Thermometers must be graduated with the appropriate increments for the analysis or test for which temperature is being measured (0.1, 0.2, 0.5, or 1.0°C). It is not acceptable to extrapolate between the graduated lines.

Laboratory Glass Thermometers and Temperature Measuring Devices: These must be checked for temperature accuracy using a NIST-traceable reference thermometer at least annually. See exceptions below:

Electronic Thermometers and Temperature Measuring Devices: Digital thermometers, thermocouples, and other similar electronic temperature measuring devices must be verified for accuracy against a NIST-traceable reference thermometer at least annually. Digital thermometers must be checked for temperature accuracy along with its temperature probe.

Dial Bimetallic Thermometers: These types of thermometers must be checked for temperature accuracy using a NIST-traceable reference thermometer at least quarterly. Dial thermometers that cannot be calibrated must not be used.

Laboratory Infrared (IR) Thermometers: IR thermometers must be verified for accuracy over the full temperature range of use at least every six months using a NIST-traceable reference thermometer. This would include ambient (20-30°C), iced (4°C) and frozen (0 to ≤10°C). Each day of use, a single check of the IR thermometer or device must be made by checking the temperature of a bottle of water at the temperature of interest that contains a calibrated thermometer. Agreement between the two must be within 0.5°C, or the IR device must be recalibrated.

NIST-Traceable Reference Thermometer: A liquid in glass reference thermometer or digital thermometer (meter and probe as one unit). NIST-traceable liquid in glass thermometers must be verified for accuracy every five years with NIST metrological traceability and measurement uncertainty documented. Digital NIST-traceable reference thermometers and probes must be verified for accuracy annually with NIST metrological traceability and measurement uncertainty documented. Documentation of NIST metrological traceability and measurement uncertainty must be available for review.

B. Holding Time

Samples must be analyzed within 15 minutes of collection (40 CFR Part 136 Table II).

C. Temperature Accuracy Verification Procedure

- Immerse laboratory thermometer (or other temperature sensing device) into test sample at the normal range of use for the thermometer or device.
- Immerse the reference thermometer into the test sample. The thermometer manufacturer will note the immersion depth, e.g., full immersion or partial immersion.
- Allow both thermometers to stabilize for at least 30 minutes.
- Record the readings from both thermometers and calculate the difference.

Document the temperature accuracy verification of each thermometer with the following:

- NIST-traceable reference thermometer ID
- Laboratory thermometer ID
- Date of accuracy verification
- NIST-traceable reference thermometer reading
- Laboratory thermometer reading
- Temperature correction factor = NIST-traceable reference thermometer reading minus the laboratory thermometer reading
The temperature correction factor must not exceed $\pm 1.0^{\circ}\text{C}$ or the thermometer must be replaced.
- Analyst's initials

Each thermometer or temperature measuring device must be tagged with the current temperature correction factor, date of accuracy verification, and analyst initials. The analyst must take the correction factor into account when monitoring and recording the corrected temperature.

D. Temperature Monitoring Records

The temperature of all equipment used for sample storage and analysis must be recorded twice daily at least four hours apart. These records should include:

- Instrument or equipment ID
- Thermometer ID
- Thermometer correction factor
- Date and Time of each reading
- Temperature in $^{\circ}\text{C}$
- Analyst initials
- Temperature adjustments (\uparrow) (\downarrow)

RECORDS AND TRACEABILITY

Laboratory traceability is the ability to ‘trace’ sample, standard, and analytical data back to a point of origin, receipt, or generation. It is necessary to document the IDs for all meters, balances, standards, and reagents used during the analysis on calibration records and sample analysis records. Including this information on the calibration and sample analysis records greatly enhances the legal defensibility of laboratory-generated data.

Laboratories are encouraged to perform an annual review of their Quality Assurance Plans, SOPs, bench sheets, and documentation procedures to seek out any documentation/traceability discrepancies and perform the necessary corrective actions. The Standard Operating Procedures (SOPs) MUST mirror the actual analytical procedures taking place in the laboratory.

REFERENCES

1. Standard Methods for the Examination of Water and Wastewater, 22nd Edition. American Public Health Association.
2. 40 CFR Parts 141 and 143 for Safe Drinking Water Act.
3. 40 CFR Part 136 for Clean Water Act.
4. EPA’s Manual for the Certification of Laboratories Analyzing Drinking Water, EPA 815-R-05-004, January 2005, Fifth Edition.

Thermometer Accuracy Verification Record

- A. National Institute of Standards and Technology (NIST)-Traceable Reference Thermometer identification number (Reading A): _____

- B. Identification number of thermometer that is being verified (Reading B). Thermometers must be checked in the range of use. For example: If the thermometer is used in the refrigerator to monitor $\leq 6^{\circ}\text{C}$, the thermometer must be checked against the NIST-traceable thermometer at 1- 6°C .

Date	Thermometer ID Number (B)	Reading A °C	Reading B °C	A – B °C	Analyst Initials

Temperature Record Chart

Instrument: _____
 Thermometer ID Number: _____ Correction Factor: _____
 Month: _____
 Year: _____

Day	AM Time	Temp ¹ °C	Initials	PM Time	Temp ¹ °C	Initials	Day	PM Time	Temp ¹ °C	Initials	PM Time	Temp ¹ °C	Initials
01							17						
02							18						
03							19						
04							20						
05							21						
06							22						
07							23						
08							24						
09							25						
10							26						
11							27						
12							28						
13							29						
14							30						
15							31						
16													

1 Temperature recorded reflects adjustment for the temperature correction factor.

Hydrogen-Ion Concentration (pH) Calibration

Method: _____

Meter ID: _____

Lab Name and ID: _____

Calibration Date Time		pH Buffer _____	pH Buffer _____	pH Buffer _____	Slope (%) or Efficiency (mV)	pH Buffer Check	Comments	Analyst's Initials
						pH Buffer _____		

4.0 pH Buffer Lot Number: _____

7.0 pH Buffer Lot Number: _____

10.0 pH Buffer Lot Number: _____

Laboratory Name:

Laboratory ID #:

Hydrogen-Ion Concentration (pH) Sample Analysis

Method: _____

Meter ID: _____

Date	Time		Sample ID	pH Value	Sample Duplicate	Analyst's Initials
	Collected	Analyzed			pH Value	

Laboratory Name: _____ Laboratory ID #: _____

Residual Chlorine Initial Calibration Verification

Total or Free Residual Chlorine (Circle One)

Date/Time: _____

Instrument ID: _____

Analyst: _____

DPD Lot#: _____

Cell Path Length¹: _____

Standard ID: _____

Wavelength¹: _____

True Value of Standards mg/L	Observed Value ³ (Meter Reading) mg/L	PASS / FAIL acceptance criteria ⁴
Blank = 0.00mg/L		
Standard 1 (PQL ²) =		
Standard 2 =		
Standard 3 =		
Standard 4 =		
Standard 5 =		

- 1 Required to be recorded if the meter cell path length and/or wavelength are adjustable on the instrument used for analysis.
- 2 The lowest standard on the initial calibration verification is the Practical Quantitation Limit (PQL) or reporting limit. This concentration is used as the reporting limit. For example: The lowest non-zero standard on the initial calibration verification is 0.05mg/L. The laboratory obtains a sample reading of 0.02mg/L. The laboratory would record the sample value as <0.05mg/L on the sample analysis record, since 0.05mg/L is the lowest standard on the initial calibration verification.
For NPDES (wastewater) analyses, the PQL is 0.05 mg/L.
- 3 The observed readings must be within 10% of the true value. If the meter readings are different by more than 10%, maintenance must be performed. An acceptance range of 80-120% is permitted for the 0.05 mg/L standard because most colorimeters display results to only two decimal places. Maintenance includes cleaning the sample cells, remaking the standard, and possibly returning to the manufacturer for repair.
- 4 If any standards fail to meet the acceptance criteria discussed in footnote 3 above. The standard must be reanalyzed. Refer to the Field Measurement Document for assistance.

Laboratory Name: _____

Laboratory ID #: _____

Total / Free Residual Chlorine Calibration and Sample Analysis

DPD lot # _____

Stock Standard ID: _____

Meter/Model#: _____

Intermediate Standard ID: _____

Initial Calibration Verification Date: _____

Calibration Verification Date Time		Blank Reading mg/L	Standard 1 _____mg/L ²	Standard 2 _____mg/L ²	Sample ID	Time ³ Collected Analyzed		Sample Reading mg/L	Sample Duplicate Reading mg/L	End of Run Check Standard _____mg/L ²	Analyst	Comments and/or Sample Dilutions

Standard 1 Preparation: _____mL of intermediate standard into _____mL DI Water.

Standard 2 Preparation: _____mL of intermediate standard into _____mL DI Water.

End of Run Standard Preparation: _____mL of intermediate standard into _____mL DI Water

¹ Indicate whether the analyses are total residual chlorine or free residual chlorine by circling the Total or Free at the top of the page.

² Daily meter readings for the standards must be within 10% of the true value of the standards, 20% is allowed for the 0.05mg/L standard.

³ 15 minute maximum holding time between sample collection and analysis.

Laboratory Name: _____

Laboratory ID # _____

Dissolved Oxygen Calibration and Sample Analysis for SM 4500 OG-2011

Meter ID _____

Analyst initials	Date	Calibration		Altitude or Pressure	DO reading after cal, mg/L	Saturation Chart, mg/L	Pass? (± 0.2 mg/L or 2%) √	Sample ID	Time of		Sample Reading, mg/L	Membrane/Battery change? Comments
		Time	Temp						Collection	Analysis*		

* Samples analyzed *in-situ* will mean that the collection and analysis time are the same. All samples must be analyzed within 15 minutes of collection.

Unless otherwise noted samples are collected: In-situ or Grab (circle)