*Name of Facility*

Standard Operating Procedure

for the analysis of

Total Residual Chlorine, Low Level

Spectrophotometric, DPD

Method: Hach 10014 ULR

Effective Date:

Supervisor Signature: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Date: \_\_\_\_\_\_\_\_\_\_

Supervisor Name (print): \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

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 *Blue text is replaceable instructional language to be customized for your facility.*

1. Summary of Method
	1. The ULR chlorine buffer and DPD indicator are added to a sample and a colorimetric procedure is used to determine the concentration of Total Residual Chlorine (TRC).
	2. This method is approved by EPA as an Alternate Test Procedure (ATP) and all steps in the method must be followed. Reference Appendix 3 for the approval letter from EPA.
	3. *State what type of samples are analyzed, e.g., wastewater effluent, ground water monitoring well, etc. and the permit limits if applicable*
	4. *State what your minimum reporting limit and working range is (based on the standards that are used for the factory-set calibration curve verification or construction of a lab-prepared calibration curve)*
2. Definitions
	1. Calibration blank: Deionized or Distilled water, without chlorine and without DPD/buffer, that is used to zero the meter when a laboratory-prepared standard is used.
	2. Reagent blank: Deionized or Distilled water, from the same source used to make calibration and calibration verification standards, to which 1.0 mL of Blanking Reagent is added. The Blanking Reagent removes chlorine and chloramines from the water. It is then analyzed like a sample (i.e., with DPD/buffer added). The concentration of the reagent blank must not exceed 5 µg/L.
	3. Method Blank: Deionized or Distilled water, from the same source used to make calibration and calibration verification standards that is analyzed like a sample (i.e., with DPD/buffer added). The concentration of the Method Blank, after the Reagent Blank concentration is subtracted, must be ≤ ½ the concentration of the lowest calibration, or calibration verification, standard.
	4. Factory-set Calibration Curve: An internal calibration curve, generated and stored as meter programs by the instrument manufacturer.
	5. Laboratory-generated Calibration Curve: A linear regression equation generated from the analysis of a series of laboratory-prepared liquid standards. Sample results are obtained by plugging sample absorbance values into the linear regression formula. This is usually performed automatically by direct read-out meters.
	6. Second-source Standard: A standard prepared from a source independent (e.g., different vendor, different lot #, etc.) from that used to prepare the calibration standards. When using a factory-set calibration curve, all other standards are considered second source.
	7. Daily Check Standard: A laboratory-prepared standard of known concentration of the analyte of interest. A Daily Check Standard is used to evaluate laboratory performance and analyte recovery in a blank matrix.
	8. Post-Analysis Calibration Verification Standard: A Daily Check Standard that is analyzed after all sample analyses.
	9. µg/L: Units for the low-level measurement of TRC.
	10. Safety Data Sheet (SDS): Document that lists information relating to occupational safety and health for the use of various substances and products.
	11. NC WW/GW LC: North Carolina Wastewater Groundwater Laboratory Certification
	12. *Add any other applicable acronyms used by your facility*
3. Safety and Waste Handling
	1. *Items that would be included in this section are things such as:*
* *Precautionary measures (list here and at the critical steps in the procedure)*
* *Personal protective equipment (e.g., gloves, eye protection, lab coat, work in a hood, etc.)*
* *Hazardous chemicals/reagents (include sodium arsenite if used)*
* *Storage and disposal of samples and reagents (include sodium arsenite if used)*
* *Reference to Chemical Hygiene Plan, if applicable*
* *Location of SDSs*
1. Apparatus, Equipment and Reagents

*Note: Include storage conditions and expiration dates for standards and reagents. It is recommended catalog numbers also be included*

* 1. *List your meter with make and model*
	2. *List your pour-through cell and filtering apparatus*
	3. Filters: *List types of filters used.*
	4. Stock Calibration standard: *what is the source of the stock calibration standard for the calibration verification or construction of a curve. (delete if contracting curve verification to another lab)*
	5. Check standard: *state if it is purchased; or if prepared, how it is made. If using a laboratory-generated calibration curve, the check standard must be second source*
	6. Reagent water: *state what type of water is used e.g., purchased deionized water, etc.*
	7. Liquid buffer: *state manufacturer*
	8. Liquid DPD indicator: *state manufacturer*
	9. Blanking Reagent*: state manufacturer*
	10. Volumetric flask *(state volumes used)*
	11. Volumetric pipet *(state volumes used)*
	12. Mechanical pipet *(if used)*
	13. 5.25 *N* Sulfuric Acid: *state manufacturer*
	14. *If performing manganese, chromium interference adjustment, add 1N Sulfuric Acid, Potassium Iodide, and Sodium Arsenite*
1. Interferences *If interferences are present at your facility, document how to treat samples in the applicable section.*
	1. The DPD methods are subject to interference by oxidized forms of Manganese (Mn4+, Mn7+) and Chromium (Cr6+). Perform the following steps to correct for these elements:
		1. Adjust sample to 6-7 S.U. with 1 *N* Sulfuric Acid
		2. Add 9 drops Potassium Iodide (30 g/L) to an 80-mL sample.
		3. Mix and wait 1 minute.
		4. Add 9 drops of Sodium Arsenite (5 g/L) and mix. **Note: Samples that are treated with sodium arsenite will contain arsenic and may require special disposal consideration. Refer to current SDS for safe handling and disposal instructions.**
		5. Analyze the treated sample as described in Section 8.
		6. Subtract the result of this test from the original analysis to obtain the correct concentration.
	2. Sample color and turbidity may interfere in all colorimetric procedures. This procedure uses filtering and sample blanking to counteract these interferences.
	3. If the sample contains a dechlorinating agent (e.g., sulfite or sulfur dioxide), the sample result (corrected for the reagent blank) will read “0” or a slightly negative value.
	4. Bromine, Chlorine Dioxide, Ozone and Iodine interfere at all levels.
	5. Organic Chloramines and Peroxides can interfere.
	6. Samples with extreme pH or those that are highly buffered are to be adjusted to 6-7 S.U.
	7. Copper (Cu2+) and Iron (Fe3+) interfere at levels above 1000 µg/L.
	8. Nitrite, NO2- (uncommon in clean waters)

|  |  |
| --- | --- |
| mg/L Nitrite | Apparent µg/L chlorine |
| 2.0 mg/L | 3 µg/L |
| 5.0 mg/L | 5 µg/L |
| 10.0 mg/L | 7 µg/L |
| 15.0 mg/L | 16 µg/L |
| 20.0 mg/L | 18 µg/L |

1. Sample Collection, Preservation and Holding Time
	1. *State what containers samples are collected in. Samples must be collected in glass or polyethylene containers.*
	2. There is no preservation requirement for Total Residual Chlorine.
	3. The holding time is 15 minutes. Holding time is defined as the time from sample collection to the addition of DPD.
	4. *State where the sample is generally analyzed (e.g., immediately at the sampling site, in the lab within holding time, etc.)*
2. Calibration *Either the factory-set curve is verified (sections 7.1-7.6 follow Option 1 of the Approved Procedure for the Analysis of Total Residual Chlorine (DPD Colorimetric by Hach 10014 ULR)) or a laboratory-generated calibration curve is programmed into the meter (sections 7.8-7.16 follow Option 3 of the Approved Procedure for the Analysis of Total Residual Chlorine (DPD Colorimetric by Hach 10014 ULR)).* ***Delete the section that is not applicable or update to describe Options 2 or 4 (three standards daily).***

* 1. The factory-set calibration curve on program 86 must be verified initially, every 12 months or any time the optics of the instrument are serviced.
	2. The following standard concentrations are used: *list your standard concentrations here (5 are required for annual calibration). One must be below your permit limit*
		1. See Appendix 2 for example standard preparation instructions *or state here if this is contracted to another lab.*
	3. A Reagent Blank must be analyzed with the curve verification and have a value ≤ 5 µg/L. The Reagent Blank absorbance is subtracted from all Method Blank and standard absorbances.
	4. A Method Blank must be analyzed with the curve verification and have a value ≤ ½ the concentration of the lowest calibration verification standard.
	5. Standards with concentrations < 50 µg/L must have a recovery of 75-125%.
	6. Standards with concentrations ≥ 50 µg/L must have a recovery of 90-110%.
	7. The Excel file shown in Appendix 1 is used to evaluate the acceptability of the curve verification. The Excel file may be downloaded from the NC WW/GW LC website here: <https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch/technical-assistance-policies>
	8. *Use the following sections 7.7 through 7.16 and delete 7.1-7.6 if constructing a laboratory-generated calibration curve (Option 3):* A calibration curve must be constructed and programmed into the meter initially, every 12 months and any time the optics of the instrument are serviced.
	9. *State which program on the instrument the constructed curve is saved in*
	10. The following standard concentrations are analyzed: *list your standard concentrations here (required to have 5 every 12 months and one must be below your permit limit)*
		1. See Appendix 2 for example standard preparation instructions *or state here if this is contracted to another lab.*
	11. A Reagent Blank must be analyzed with the curve and have a value ≤ 5 µg/L. The Reagent Blank absorbance is subtracted from all Method Blank and standard absorbances.
	12. A Method Blank must be analyzed with the calibration curve and have a value ≤ ½ the concentration of the lowest calibration standard.
	13. Insert the absorbance value of each standard analyzed into the calibration curve equation to back-calculate the value of each standard.
	14. For standards with concentrations < 50 µg/L, the back-calculated value must be between 75% and 125% of the true value.
	15. For standards with concentrations ≥ 50 µg/L, the back-calculated value must be between 90% and 110% of the true value.
	16. The correlation coefficient of the curve must be ≥ 0.995.
	17. A second source standard must be analyzed and have a recovery of 75-125% for concentrations < 50 µg/L and have a recovery of 90-110% for concentrations ≥ 50 µg/L.
	18. The excel file shown in Appendix 1 is used to evaluate the acceptability of the curve verification. The Excel file may be downloaded from the NC WW/GW LC website here: <https://deq.nc.gov/about/divisions/water-resources/water-resources-data/water-sciences-home-page/laboratory-certification-branch/technical-assistance-policies>
1. Procedure
	1. Use a new filter for each test.
	2. Check condition of sample cells prior to use. They must be clean, clear of fingerprints and free of scratches.
	3. Ensure proper placement of indexed cells. Note that there are two options for Pour-Thru Cell placement. One gives a 1-inch cell path length and when rotated 90°, gives a ½-inch cell path length. Ensure the 1-inch cell path length is used.
	4. Determine the reagent blank value. See the acceptance criterion in Section 12.
		1. Start program 86 (or if a user-generated curve was saved in the instrument, use that program).
		2. Flush the Pour-Thru Cell with at least 50-mL of deionized water.
		3. Collect approximately 100 mL of deionized or tap water in a clean, 250-mL beaker.
		4. Add 1.0 mL of Blanking Reagent to the beaker. Swirl to mix. The Blanking Reagent removes chlorine and chloramines from the water. Use this solution in step 8.4.11.
		5. Start a timer for 5 minutes.
		6. Open one ampule of ULR Chlorine Buffer Solution
		7. Use a clean pipette to add 1 mL of buffer from the ampule to a clean and prepared 50-mL mixing cylinder.
		8. Open one ampule of DPD Indicator Solution for ULR Chlorine
		9. Use a clean pipette to add 1 mL of indicator from the ampule to the same mixing cylinder.
		10. Swirl to mix. Continue to the next step within 1 minute.
		11. Fill the cylinder to the 50-mL mark with dechlorinated water from step 8.4.4. Keep the remaining dechlorinated water for step 8.4.14.
		12. Put the stopper on the mixing cylinder. Invert the mixing cylinder two times to mix.
		13. Start the instrument timer. A 3-minute reaction time starts.
		14. During the reaction period, flush the Pour-Thru Cell with the remaining dechlorinated water from step 8.4.4.
		15. When the flow stops, push ZERO. The display shows 0 µg/L Cl2.
		16. When the timer expires, pour the contents of the cylinder into the Pour-Thru Cell.
		17. Push READ. Results show in µg/L Cl2.
		18. Subtract this value from the sample results received in this procedure. Refer to the instrument documentation for more information on blank adjustment.
		19. Flush the Pour-Thru Cell with at least 50-mL of deionized water immediately after use.
	5. A daily check standard must be analyzed each day before sample analysis using the same procedure as for samples in steps 8.6 through 8.28. See acceptance criteria in Section 12.
	6. Start program 86. (or if a user-generated curve was saved in the instrument, use that program)
	7. Flush the Pour-Thru Cell with at least 50-mL of deionized water.
	8. Remove the cap from the OriFlo plunger assembly. Make sure that the O-ring is seated correctly in the cap.
	9. Install a new, 3-micron filter (white) into the cap recess. Wet the filter with drops of deionized water. Reassemble and hand-tighten the cap onto the plunger.
	10. Open one ampule of ULR Chlorine Buffer Solution.
	11. Use a clean pipette to add 1 mL of buffer from the ampule to a clean and prepared 50-mL mixing cylinder.
	12. Open one ampule of DPD Indicator Solution for Ultra Low Range Chlorine.
	13. Use a clean pipette to add 1 mL of indicator from the ampule to the same mixing cylinder.
	14. Swirl to mix. Continue to the next step within 1 minute.
	15. Prepared Sample: Prevent extra agitation while carefully filling the cylinder to the 50-mL mark with sample.
	16. Put the stopper on the mixing cylinder. Carefully invert the mixing cylinder twice to mix.
	17. Start the instrument timer. A 3-minute reaction time starts. Complete steps 8.18-8.24 during this time. Note: The reacted sample is to be measured 3-6 minutes after mixing the sample reagents.
	18. Push the valve button on the OriFlo barrel assembly in ("closed" position). Put the barrel assembly into its stand.
	19. Pour approximately 50 mL of the original sample into the barrel. The lower ring on the barrel assembly shows about a 50-mL volume.
	20. Insert the plunger into the barrel and slowly push the plunger down with even pressure until the plunger is fully seated.
	21. Pour the filtered, unreacted sample from the plunger reservoir into the Pour-Thru Cell.
	22. When the flow stops, push ZERO. The display shows 0 µg/L Cl2.
	23. Pull the barrel valve button out to the “open” position. Pull the plunger up to separate it from the barrel assembly. Discard the remaining unfiltered sample. A new membrane may be necessary for very turbid samples.
	24. Push the barrel valve button to the “closed” position. Put the barrel assembly into its stand.
	25. When the timer expires, pour the contents of the mixing cylinder into the barrel.
	26. Insert the plunger into the barrel and slowly push the plunger down with even pressure, until the plunger is fully seated.
	27. Pour the filtered, reacted sample from the plunger reservoir into the Pour-Thru Cell.
	28. When the flow stops, push READ. Results show in µg/L Cl2. If the sample contains a dechlorinating agent (e.g. sulfite or sulfur dioxide), the sample result (corrected for the reagent blank) will read "0" or a slightly negative value.
	29. *This section would be for analysts who are taking measurements at multiple sites away from the lab’s primary location.* A post analysis calibration verification using the check standard must be analyzed after the last compliance sample. See acceptance criteria in Section 12.
2. Documentation

The following must be documented in indelible ink whenever sample analysis is performed:

* 1. Date and time of sample collection
	2. Date and time of sample analysis (i.e., time reagents are added) to verify the 15-minute holding time is met [Alternatively, one time may be documented for collection and analysis with the notation that samples are measured immediately at the sample site.]
	3. Facility name, sample site (ID or location), and permit number
	4. Collector’s/analyst’s name or initials
	5. Preparation procedure and true values of laboratory-prepared standards, when applicable
	6. Daily Check Standard analysis time
	7. True value of the Daily Check Standard
	8. Value obtained and recovery for the Daily Check Standard
	9. Value obtained for the Reagent Blank (verification of ≤ 5 µg/L)
	10. Value obtained for the Method Blank (must be ≤ 1/2 concentration of the lowest calibration curve, or calibration curve verification, standard)
	11. All data must be documented and reported in units of measure as specified in the permit (e.g., µg/L)
	12. Time analyzed, true value and value obtained for the Post-analysis Calibration Verification Standard(s), where applicable
	13. Traceability for chemicals, reagents, standards and consumables
	14. Instrument identification (serial number preferred)
	15. Date of most recent calibration curve generation or calibration curve verification
	16. Statement that samples were filtered
	17. Final value to be reported
	18. Parameter analyzed
	19. Method reference (refer to Certified Parameters Listing (CPL) for correct method description)
	20. Data qualifier(s), when applicable
	21. Equipment maintenance (recommended)
1. Proficiency Testing Procedure
	1. Analysis of a blind (PT) sample is required at least once during every 9-month PT calendar year (January 1- September 30).
		1. A list of approved PT providers may be found on the NELAC website at <http://nelac-institute.org/content/NEPTP/ptproviders.php>. Check this list yearly to assure the chosen provider is approved.
		2. A PT sample can be analyzed as early as January 1 and the graded result must be reported to NC WW/GW LC office from the PT vendor no later than September 30.
	2. PT samples must be analyzed in accordance with the routine testing, calibration and reporting procedures, unless otherwise specified in the instructions supplied by the PT Sample Provider.
		1. PT Samples are logged in and analyzed using the same staff, sample tracking systems, standard operating procedures including the same equipment, reagents, calibration techniques, analytical methods, and the same quality control acceptance criteria.
		2. PT Samples shall not be analyzed with additional quality control. They are not to be replicated beyond what is routine for Compliance Sample analysis.
		3. PT Sample analysis must be documented on the laboratory’s daily benchsheet.

* 1. The PT provider’s instructions for preparing the PT Sample must be followed and the practice documented by the analyst. The instruction sheet will be initialed and dated when the PT sample is prepared and retained for 5 years.
	2. The following information must be included when reporting the PT Samples.
		1. EPA Lab Code: *(enter here so it is easy to retrieve)*
		2. State Lab Certification number: *(enter here so it is easy to retrieve)*
		3. Method description (refer to CPL for current method description):
		4. Mailing address for NC WW/GW LC: 1623 Mail Service Center, Raleigh, NC 27699-1623
1. Calculations and Reporting
	1. Percent Recovery

% Recovery = Value Obtained x 100

 True Value

* 1. Report in units of µg/L
	2. *Describe number of significant figures and rounding procedures*
1. Quality Assurance and Quality Control
	1. Reagent Blanks must be ≤ 5 µg/L, or corrective action must be taken (See Section 14.0).
	2. Method Blanks must be ≤ ½ the concentration of the lowest calibration or calibration verification standard.
	3. Daily check standards and post-analysis check standards (if applicable) must read within ±10% of the known value for concentrations ≥ 50 µg/L and within ±25% for concentrations < 50 µg/L, or corrective action must be taken (See Section 14.0).
	4. Any sample greater than *(state upper reporting limit)* must be diluted to fall within the range of the calibration curve. Any sample less than (*state lower reporting limit)* will be reported as < *(lower reporting limit)*
	5. *If mechanical pipets are used for critical measurements (preparing standards) they must be calibrated every 12 months. State the frequency and steps for performing the calibration or state what lab is contracted to perform it.*
	6. *State who is transcribing the data to the eDMR and whether anyone peer reviews (checks) it. Peer review is recommended, but if that is not possible, it is recommended that the analyst rechecks their own transcription for errors after a certain amount of time has passed.*
2. Preventative Maintenance
	1. *State if a maintenance log or record is maintained*
	2. *Include instructions for cleaning pour through cell and how often it is done.* *The Pour-Thru Cell can collect a buildup of products with color, especially if the reacted solutions stay in the cell for long periods of time after measurement. Rinse the Pour-Thru Cell with 5.25 N Sulfuric Acid to remove the color. Fully rinse with deionized water. Put a cover on the Pour-Thru Cell funnel when it is not in use.*
3. Troubleshooting and Corrective Action
	1. *State what will be done if a meter does not pass the daily or post analysis calibration check or reagent blank criterion. (e.g.,* *reanalyze the reagent blank or standard once*, *recalibrate meter and reanalyze previous samples, qualify samples on the DMR, etc.)*
4. Employee Training

Employee training must be documented and kept on file.

* 1. *Include required education, training, experience and/or demonstrated skills required for the position*
	2. Employee must have read this SOP *– may also include reading the Approved Procedure for the Analysis of Total Residual Chlorine (DPD Colorimetric)*

*Employee must obtain acceptable results on Proficiency Testing samples or other demonstrations of proficiency (e.g., Initial Demonstration of Capability (IDOC), side-by-side comparison with established analyst, etc.) before analyzing compliance samples for reporting.*

1. References
	1. Hach Water Analysis Handbook, Method 10014, 03/2014, Edition 8.
	2. North Carolina Wastewater/Groundwater Laboratory Certification Approved Procedure for the Analysis of Total Residual Chlorine (DPD Colorimetric by Hach 10014 ULR), Revision 11/26/2019.
	3. 15A NCAC 02H .0800
2. Revision History

|  |  |  |
| --- | --- | --- |
| Type: Review or Revision | Date | Summary of Changes Made if Revision |
|  |  |  |
|  |  |  |
|  |  |  |
|  |  |  |
|  |  |  |
|  |  |  |

Appendix 1(delete the spreadsheet that isn’t used)





*Appendix 2* *(delete the standard preparation instructions that are not applicable)*

**TOTAL RESIDUAL CHLORINE CURVE PREPARATION**

(For Facilities with a limit of 13 µg/L)

**Note:** The use of **Class A** Volumetric flasks and pipettes is critical to the accurate preparation of these low-level standards.

**Needed Materials**

Chlorine–Free Water

Potassium Permanganate

1-Liter Class A Volumetric Flask (Quantity = 1)

100-mL Class A Volumetric Flask (Quantity = 7)

50-mL Class A Volumetric Pipette (Quantity = 1)

25-mL Class A Volumetric Pipette (Quantity = 1)

10-mL Class A Volumetric Pipette (Quantity = 2)

5-mL Class A Volumetric Pipette (Quantity = 2)

2-mL Class A Volumetric Pipette (Quantity = 1)

1-mL Class A Volumetric Pipette (Quantity = 1)

**800 mg/L Stock Standard** – Prepare by dissolving 712.8 mg KMnO4 (potassium permanganate) in 1 liter of chlorine-free water.

**NOTE:** **If using a purchased 1000 mg/L Standard as your stock**, the following Intermediate Standard should be prepared by diluting 8 mL of the 1000 mg/L standard to 100 mL with chlorine-free water.

**80 mg/L Intermediate Standard** – Prepare by diluting 10 mL of the 800 mg/L Stock Standard to 100 mL with chlorine-free water.

**0.8 mg/L (800 µg/L) Working Standard** - Prepare by diluting 1 mL of the 80 mg/L Intermediate Standard to 100 mL with chlorine-free water.

The **Working Standard** will be used to prepare your **5-Point Curve**.

Add 50 mL of the Working Standard to a 100-mL volumetric flask and dilute to volume with chlorine-free water. **This is your 400 µg/L Standard.**

Add 25 mL of the Working Standard to a 100-mL volumetric flask and dilute to volume with chlorine-free water. **This is your 200 µg/L Standard.**

Add 10 mL of the Working Standard to a 100-mL volumetric flask and dilute to volume with chlorine-free water. **This is your 80 µg/L Standard.**

Add 5 mL of the Working Standard to a 100-mL volumetric flask and dilute to volume with chlorine-free water. **This is your 40 µg/L Standard.**

Add 5 mL of the **200 µg/L Standard** to a 100-mL volumetric flask and dilute to volume with chlorine-free water. **This is your 10 µg/L Standard.**

*Appendix 2* *(delete the standard preparation instructions that are not applicable)*

**TOTAL RESIDUAL CHLORINE CURVE PREPARATION**

(For Facilities with a limit of 17 µg/L)

**Note:** The use of **Class A** Volumetric flasks and pipettes is critical to the accurate preparation of these low-level standards.

**Needed Materials**

Chlorine–Free Water

Potassium Permanganate

1-Liter Class A Volumetric Flask (Quantity = 1)

100-mL Class A Volumetric Flask (Quantity = 7)

50-mL Class A Volumetric Pipette (Quantity = 1)

25-mL Class A Volumetric Pipette (Quantity = 1)

10-mL Class A Volumetric Pipette (Quantity = 2)

5-mL Class A Volumetric Pipette (Quantity = 1)

2-mL Class A Volumetric Pipette (Quantity = 1)

1-mL Class A Volumetric Pipette (Quantity = 1)

**800 mg/L Stock Standard** – Prepare by dissolving 712.8 mg KMnO4 (potassium permanganate) in 1 liter of chlorine-free water.

**NOTE:** If using a purchased 1000 mg/L Standard as your stock, the following Intermediate Standard should be prepared by diluting 8 mL of the 1000 mg/L standard to 100 mL with chlorine-free water.

**80 mg/L Intermediate Standard** – Prepare by diluting 10 mL of the 800 mg/L Stock Standard to 100 mL with chlorine-free water.

**0.8 mg/L (800 µg/L) Working Standard** - Prepare by diluting 1 mL of the 80 mg/L Intermediate Standard to 100 mL with chlorine-free water.

The **Working Standard** will be used to prepare your **5-Point Curve**.

Add 50 mL of the Working Standard to a 100-mL volumetric flask and dilute to volume with chlorine-free water. **This is your 400 µg/L Standard.**

Add 25 mL of the Working Standard to a 100-mL volumetric flask and dilute to volume with chlorine-free water. **This is your 200 µg/L Standard.**

Add 10 mL of the Working Standard to a 100-mL volumetric flask and dilute to volume with chlorine-free water. **This is your 80 µg/L Standard.**

Add 5 mL of the Working Standard to a 100-mL volumetric flask and dilute to volume with chlorine-free water. **This is your 40 µg/L Standard.**

Add 2 mL of the Working Standard to a 100-mL volumetric flask and dilute to volume with chlorine-free water. **This is your 16 µg/L Standard.**

*Appendix 2* *(delete the standard preparation instructions that are not applicable)*

**TOTAL RESIDUAL CHLORINE CURVE PREPARATION**

(For Facilities with a limit of 28 µg/L)

**Note:** The use of **Class A** Volumetric flasks and pipettes is critical to the accurate preparation of these low-level standards.

**Needed Materials**

Chlorine–Free Water

Potassium Permanganate

1-Liter Class A Volumetric Flask (Quantity = 1)

100-mL Class A Volumetric Flasks (Quantity = 7)

40-mL Class A Volumetric Pipette (Quantity = 1)

20-mL Class A Volumetric Pipette (Quantity = 1)

10-mL Class A Volumetric Pipette (Quantity = 2)

4-mL Class A Volumetric Pipette (Quantity = 1)

5-mL Class A Volumetric Pipette (Quantity = 1)

2-mL Class A Volumetric Pipette (Quantity = 1)

1-mL Class A Volumetric Pipette (Quantity = 1)

**1000 mg/L Stock Standard** – Prepare by dissolving 891 mg KMnO4 (potassium permanganate) in 1 liter of chlorine-free water. (This standard can be purchased pre-made.)

**100 mg/L Intermediate Standard** – Prepare by diluting 10 mL of the Stock Standard to 100 mL with chlorine-free water.

**1.0 mg/L (1000 µg/L) Working Standard** - Prepare by diluting 1 mL of the Intermediate Standard to 100 mL with chlorine-free water.

The **Working Standard** will be used to prepare your **5-Point Curve**.

Add 40 mL of the Working Standard to a 100-mL volumetric flask and dilute to volume with chlorine-free water. **This is your 400 µg/L Standard.**

Add 20 mL of the Working Standard to a 100-mL volumetric flask and dilute to volume with chlorine-free water. **This is your 200 µg/L Standard.**

Add 5 mL of the Working Standard to a 100-mL volumetric flask and dilute to volume with chlorine-free water. **This is your 50 µg/L Standard.**

Add 4 mL of the Working Standard to a 100-mL volumetric flask and dilute to volume with chlorine-free water. **This is your 40 µg/L Standard.**

Add 2 mL of the Working Standard to a 100-mL volumetric flask and dilute to volume with chlorine-free water. **This is your 20 µg/L Standard.**

***Appendix 3: EPA Acceptance Letter***

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