Please remove any shaded areas in final copy. They are present as a guide in development of SOP, showing where editing should occur.

Field Analysis

Standard Operating Procedure

(SOP)

{Facility name here}

Prepared By: NAME / TITLE _____ Date _____

Date

Approved By: NAME / TITLE _____ Date _____

Approved By: NAME / TITLE _____ Date _____

Effective Date: mm/dd/yyyy

Revision and Annual Review History

This page documents the annual review and the revisions over time to the SOP. The most recent iteration should be listed in the first row, with consecutive versions following. Signatures may be required for revised documents.

It is a requirement that this be signed annually.

Date of Revision and Annual Review	Page(s)/Section(s) Revised	Revision Explanation

2. Table of Contents

3. Procedures

a. Scope and Applicability

This SOP will outline procedures to conduct field analysis for pH, temperature, conductivity, total residual chlorine (TRC), turbidity, and dissolved oxygen (DO) as related to the analysis of wastewater samples. Remove methods not performed.

Individual Reference Methods	
pH	SM 4500 H^+ B – 2011
Temperature	SM 2550 B – 2010
Conductivity	SM 2510 B – 2011
TRC	SM 4500 Cl G – 2011 or Hach 8167
Turbidity	SM 2130 B – 2011
DO	SM 4500 O G – 2016

Individual scopes & applications	
pH	This method is used to measure the intensity of the acidic or basic nature of an aqueous solution and is expressed as pH or hydrogen ion activity. pH is one of the most common tests in environmental conditions and can frequently affect other parameters.
Temperature	This method will measure temperature in °C by utilizing a liquid in glass or electronic thermometer.
Conductivity	This method is to be used to determine the ability of a material to conduct electrical current. Conductivity is dependent on the type, concentration and valence of ions present at the time of measurement. Conductivity provides an excellent reference for solids, anion and cation analyses.
Total Residual Chlorine (TRC)	This method describes the use of a pocket Colorimeter for the measurement of total chlorine. This test is one of the most common tests used in water chemistry that frequently affects other water quality parameters.
Turbidity	Turbidity in water is caused by suspended and colloidal matter. Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted with no change in direction or flux level through the sample.
Dissolved Oxygen (DO)	A thin semi-permeable membrane, stretched over the sensor, isolates the electrodes, while allowing gases to enter. When a polarizing voltage is applied to the sensor electrodes, oxygen that has passed through the membrane reacts at the cathode causing a current that is measured as dissolved oxygen.

b. **Summary of Method** Remove methods not performed.

Individual Method Summaries	
pH	A sample is collected, and the pH is measured by inserting the calibrated pH and temperature probe into the sample and recording the reading.
Temperature	Temperature is measured using any standard liquid in glass or electronic thermometer with an analog or digital readout.
Conductivity	Conductivity is measured by inserting a calibrated conductivity probe into a sample and recording the reading
TRC	The way the meter detects the concentration of the chlorine is due to the intensity of the color produced when the reagent is added to the sample. Chlorine can be present in water as free chlorine and as combined chlorine. Both forms can co-exist in the same solution and can be determined together as total chlorine. The iodine and free chlorine react with DPD (N,N-diethyl-p-phenylenediamine) to form a red solution. The color intensity is proportional to the total chlorine concentration.
Turbidity	The turbidity is reported in Nephelometric Turbidity Units (NTU). Turbidity is a measurement of the optical property of water – a measure of the amount of light that is scattered and absorbed by particles in the sample. It requires the use of a Nephelometer or turbidimeter to compare a formazin solution to the sample.
DO	DO is measured by inserting a calibrated DO probe into a sample and recording the reading

c. Definitions

1. MB – Method Blank – A sample that is intended to contain none of the analytes of interest and which is subjected to the usual analytical or measurement process to establish a zero baseline or background value.
2. CV – Calibration Verification – A standard containing the target analytes that is used to verify the calibration curve. The concentration of continuing standards may be varied, when prescribed by the reference method, so that the range of the calibration curve is verified on a regular basis.
3. QCS – Quality Control Sample – A solution of method analytes of known concentrations that is used to fortify an aliquot of MB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards ("second source'). The QCS is

used to check laboratory performance with externally prepared test materials.

4. DUP – Duplicate – Two aliquots of the same sample taken in the laboratory and analyzed separately with identical procedures. An analysis of a DUP indicates precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.
5. RL – Reporting Limit – The minimum concentration level reported by the laboratory.
6. RPD – Relative Percent Difference – The measure of the precision or reproducibility of an analysis result.
7. DI water – Deionized water – Water that has been through a deionization process and meets ASTM standard for Type I or II water. May substitute distilled water if it meets ASTM standard for Type I or II water.
8. COC – Chain of Custody – An unbroken trail of accountability that ensures the physical security of samples, data, and records.

d. Health & Safety Warning

Analysts should always utilize all appropriate personal protective equipment during analysis. All chemicals should be handled and stored in accordance with their Material Safety Data Sheet (MSDS).

e. Cautions

Instruments and reagent/standards should be stored inside in a dry room that is at a constant temperature. Storing instruments outside in a vehicle could cause damage to the meter and degrade reagents/standards.

f. Interferences Remove methods not performed.

Individual Method Interferences	
pH	pH measurements are affected by temperature and organic material within the sample. Eliminate their interferences by frequently cleaning probe and report the temperature at which the pH was measured.
Temperature	N/A
Conductivity	N/A
TRC	Color and turbidity may give false high readings. These are compensated for by using a sample blank to zero the photometer.
Turbidity	True color that absorbs light alters the turbidity reading. This effect is usually not significant in treated water.
DO	Prolonged use of membrane electrodes in water containing gases such as H_2S tend to lower cell sensitivity. Eliminate this interference by frequently changing the membrane.

g. Personnel Qualifications / Responsibilities

Analysts should be familiar with the requirements established within this SOP and display proficiency with the techniques contained herein.

h. Equipment and Supplies Remove methods not performed.

Equipment Needed	
pH	pH meter, pH probe, temperature probe
Temperature	Thermometer or temperature probe
Conductivity	Conductivity meter, conductivity probe
TRC	Pocket Colorimeter, sample cells, stopwatch/timer
Turbidity	Turbidimeter, sample cell
DO	DO meter, DO Probe, calibration bottle

Reagents Needed	
pH	DI Water
Temperature	DI Water
Conductivity	DI Water
TRC	DI Water, DPD pillows
Turbidity	DI Water, Silicon oil
DO	DI Water

Standards Needed	
pH	4 buffer, 7 buffer, 10 buffer, CV std
Temperature	N/A
Conductivity	Calibration std, CV std
TRC	CV std
Turbidity	Calibration Std, CV std
DO	N/A

i. Step by Step Procedure Remove methods not performed.

- **Instrument or Method Calibration and Standardization**:
  - Calibrate all meters daily or with each use following the manufacturer's instructions.
- **Collection and Preservation**

- All analyses are preserved at $\leq 6\ ^{o}C$ without freezing in either glass or plastic
- All analyses other than Conductivity are to be analyzed ASAP (within 15 minutes)
- Conductivity has a hold time of 28 days.

pH & Temperature

Calibration:

1) Calibrate the meter daily or before each use according to the manufacturer's instructions using the pH 4, 7, and 10 buffers.
2) Verify calibration with CV analysis.
3) Record data.

Analysis:

1) Rinse the probe thoroughly with the DI water and blot dry
2) Shake the sample and then place the probe into the sample continuing to gently swirl the bottle.
3) Wait for the pH and Temperature readings to stabilize and record data.
4) Record the temperature of the sample next to the pH reading in the logbook and on the COC.
5) Rinse the probe thoroughly with the DI water and blot dry
6) When not in use store the electrode according to the manufacturer's instructions, usually in the electrode storage solution.

Conductivity

Calibration:

1) Calibrate the meter daily or before each use according to the manufacturer's instructions using a standard nearest the expected range of analysis.
2) Record data.

Analysis:

1) Rinse the probe thoroughly with the DI water.
3) Shake the sample and then place the probe into the sample.
   Note: Make sure the working portion of the probe remains completely immersed in the sample.
4) Wait for the reading to stabilize and record sample data.
5) Rinse the probe thoroughly with the DI water

6) Always store the electrode according to the manufacturer's instructions when not in use.

Total Residual Chlorine using HACH Pocket Colorimeter

Replace these steps with other appropriate ones if different instrument or method is used.

Calibration check:

1) Turn on instrument ensuring that the instrument is in the Low Range setting.
2) Wipe and insert blank from standards kit and place meter cap over the cell compartment to cover the cell.
3) Press the blue zero/scroll key.
4) Wipe, insert the selected standard and replace the meter cap.
5) Press green read/enter key.
6) Record data

Analysis - LOW RANGE (0.02 to 2.00 mg/L Cl2):

1) Fill a cell to the line with sample.
2) Wipe and place in colorimeter, place meter cap and press the blue zero/scroll key. Proceed to step 3 for Free Chlorine or step 6 for Total Chlorine.
3) **FREE CHLORINE:** Fill a cell to the line with sample and add 1 DPD free chlorine packet.
4) Cap and gently shake for 20 seconds, and wipe vial to remove excess water.
5) Insert in colorimeter, replace meter cap and press green read/enter key.
6) **TOTAL CHLORINE:** Fill a cell to the line with sample and add 1 DPD total chlorine packet.
7) Cap and shake for 20 seconds, and wipe vial to remove excess water.
8) **Wait 3-6 minutes**, insert in colorimeter, replace meter cap and press green read/enter key.
9) Record sample data.

Analysis - HIGH RANGE (0.1 to 8.0 mg/L Cl2):

1) Press black Menu (lines) key, press green read/enter key until instrument is changed to HR (High Range). Press black Menu (lines) key again.
2) Use the larger plastic matching cells.
3) Fill a cell to the 5ml (lower) line with sample.
4) Wipe and place in colorimeter, place meter cap and press the blue zero/scroll key.
5) **FREE CHLORINE:** Fill a cell to the line with sample and add **2** DPD free chlorine packets.
6) Cap and shake for 20 seconds, and wipe vial to remove excess water.
7) Insert in colorimeter, replace meter cap and press green read/enter key.
8) **TOTAL CHLORINE:** Fill a cell to the line with sample and add **2** DPD total chlorine packets.
9) Cap and shake for 20 seconds, and wipe vial to remove excess water.
10) **Wait 3-6 minutes**, insert in colorimeter, place meter cap green read/enter key.

11) Record sample data.

Turbidity

Calibration:

1) Calibrate the meter daily or before each use according to the manufacturer's instructions using a standard for each range of analysis.
2) Record data.

Analysis:

1) Rinse the sample cell thoroughly with the DI water.
3) Shake the sample and then pour into the sample cell.
4) Allow the sample to equilibrate to air temperature and allow all gases/bubbles to escape.
5) Wipe sample cell with soft, lint free cloth to ensure that the outside is dry, clean, and free from smudges
6) Apply a thin film of silicon oil to the sample cell & wipe sample cell with soft, lint free cloth.
7) Place sample cell with sample into turbidimeter and cover with light shield cap.
8) Record sample data.
9) Rinse the sample cell thoroughly with the DI water
10) Always store the sample cell according to the manufacturer's instructions when not in use.

Dissolved Oxygen

Calibration:

1) Check membrane and replace it according to the manufacturer's recommendations.
2) Calibrate the meter daily or before each use according to the manufacturer's instructions using a standard for each range of analysis.
3) Record data.

Analysis:

1) Rinse the probe thoroughly with the DI water.
2) Do not shake the sample.
3) Place probe in sample while stirring gently.
4) Allow the sample to stabilize.
5) Record sample data.
6) Rinse the probe thoroughly with the DI water.
7) Always store the sample cell according to the manufacturer's instructions.

j. Data and Records Management

Formula for Calculating Duplicate Relative Percent Difference (RPD)

$$RPD = \frac{|Dup_1 - Dup_2|}{(Dup_1 + Dup_2)/2} \times 100$$

Where Dup_1 = concentration of sample
Dup_2 = concentration of duplicate of sample

Formula for Calculating Percent Recovery for a Known (% Rec)

$$\%\ Rec = \frac{\text{Analyzed Value}}{\text{True Value}} \times 100$$

Formula for Calculating Percent Recovery for a Spiked Sample (% Rec)

$$\%\ Rec = \frac{(A - B)}{C} \times 100$$

Where A = measured concentration in the fortified sample
B = measured concentration in the unfortified sample
C = fortification concentration

- All data must be retained for five (5) years or until the laboratory's next audit, whichever is longer.

4. Quality Control and Quality Assurance Section

QC								
	MB		CV		QCS		DUP	
	Frequency	Acceptance Criteria ±	Frequency	Acceptance Criteria [1]	Frequency	Acceptance Criteria [1]	Frequency	Acceptance Criteria
pH	N/A	N/A	Daily	±0.1 SU	Quarterly[3]	5%	1/20[4]	20% RPD
Temperature	N/A	N/A	N/A	N/A	N/A	N/A	1/20[4]	20% RPD
Conductivity	N/A	N/A	Daily	10%	Quarterly	10%	1/20[4]	20% RPD
TRC	Daily	< RL	Daily	10%	Quarterly[2]	10%	1/20[4]	20% RPD
Turbidity	Daily	< RL	Daily	10%	Quarterly[2]	10%	1/20[4]	20% RPD
DO	N/A	N/A	N/A	N/A	N/A	N/A	1/20[4]	20% RPD

[1] May utilize acceptance criteria listed on the manufacture's certificate of analysis.
[2] Secondary standards (e.g. gel, etc.) are not allowed for QCS determination.
[3] Not Required if CV used was not one of the calibration buffers (second source).
[4] Duplicates are to be analyzed every 20 samples or quarterly, whichever comes first.

- If the results are outside of the established acceptance range repeat once, if outside the acceptance range on the second analysis, do not use the meter and investigate the cause of the problem (i.e. user error, expired or degraded standards, etc.).

5. Reference Section

i. 40 CFR 136, Code of Federal Regulations, "Guidelines Establishing Test Procedures for the Analysis of Pollutants"

ii. Standard Methods for the Examination of Water and Wastewater, 22nd Edition