

**TIMPANOGOS SPECIAL SERVICE DISTRICT STANDARD OPERATING PROCEDURE**

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Revision 10

Revised BY: RJF

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**TIMPANOGOS SPECIAL SERVICE DISTRICT  
STANDARD OPERATING PROCEDURE**

**BIOCHEMICAL OXYGEN DEMAND (BOD)**

**STANDARD METHODS 5210 B**

**Document Control # T-01**

**REVISION 10**

**EFFECTIVE DATE 06-15**

Signature of Approval:

Quality Assurance Officer:

Ryan J. Freeman

## Biochemical Oxygen Demand Method 5210B

### **1.0 Scope and Application**

- 1.01 The analysis of Biochemical Oxygen Demand (BOD) determines the relative oxygen requirements of municipal and industrial wastes. BOD is the amount of dissolved oxygen needed to oxidize organic components in waters with microorganisms

### **2.0 Summary of Method**

- 2.01 An aliquot of sample or a dilution of the sample is seeded, supplied nutrients and incubated at 20°C for 5 days. The reduction in dissolved oxygen at the end of the incubation period measures the biochemical oxygen demand.<sup>2</sup>

### **3.0 Method Deviations**

- 3.01 Not Applicable

### **4.0 Definition of Terms**

- 4.01 A list of terminology, with their definitions, utilized by this procedure and the laboratory staff is located in the Laboratory Quality Assurance Plan.

### **5.0 Interferences**

- 5.01 Dissolved inorganic salts, reactive compounds, and temperature may interfere with the performance of the DO probe.  
5.02 If ammonia is present in the sample it will oxidize into nitrate during incubation creating a positive interference.

### **6.0 Safety**

- 6.01 Due to the microbiological nature of samples, the following safety equipment should be worn by technicians: lab coat; safety glasses; and protective gloves.

**7.0    Equipment and Supplies**

- 7.01 Dissolved Oxygen Meter, with Probe
- 7.02 300mL BOD bottle
- 7.03 Incubator, capable of maintaining 20<sup>0</sup>C
- 7.04 Pipettes
- 7.05 Graduated cylinders

**8.0    Reagents and Standards**

- 8.01 Deionized water
- 8.02 Nutrient Phosphate Buffer: HACH Nutrient powder pillows; one pillow is dissolved in 6L of DI water.
- 8.03 Dechlorinating reagent, 0.025N Sodium Sulfite: 1.575g/L. This solution is not stable, **prepare daily**.
- 8.04 Polyseed: 1 capsule/500mL of nutrient buffer solution.
- 8.06 Hach BOD standard 400mg/L
- 8.07 Hach TRC Accuvac ampules, for chlorine screening

**9.0    Sample Collection, Preservation and Storage**

- 9.01 Composite samples are collected in plastic containers and analyzed as soon as possible. If analysis is not started immediately, samples must be stored at 4<sup>0</sup>C, and analyzed within 48 hours from collection.

**10.0    Quality Control, Acceptance Criteria, Corrective Actions & Contingencies**

- 10.01 All quality control is to be performed per batch, 20 samples or less. Quality control samples to be analyzed are; two blanks, two seed controls, two standards, and a batch duplicate.
  - 10.01.1 The blank should be less than 0.2 mg/L.
  - 10.01.2 The seed control should be between 0.6 and 1 mg/L.
  - 10.01.3 Standard recovery should be within 15% of true value.
  - 10.01.4 Duplicate samples should be within 20 RPD.
- 10.02 For a valid BOD result, at least 2mg/L of oxygen should be used, the final DO must not record less than 1mg/L.
- 10.03 Corrective Actions:
  - 10.03.1 If the above acceptance criteria is not met, the analysis must be stopped, trouble shoot the problem, and re-analyze.
  - 10.03.2 Most problems may be solved by performing probe maintenance, or adjusting seed volumes in future setups.  
**Note:** All instrument maintenance must be documented in the instrument maintenance log.

## 10.04 Contingencies:

- 10.04.1 If the problem cannot be solved, re-analysis is not possible in BOD analysis (due to sample expiration), the data must be flagged on the bench sheet and on the report.

**11.0 Calibration and Standardization**

- 11.01 Calibrate probe and meter as per manufacturer's instructions.

**12.0 Procedure**

## 12.01 Prepare samples for analysis:

- 12.01.01 Bring samples and dilution water to  $20 \pm 3^{\circ}\text{C}$  before beginning analysis.
- 12.01.02 Check effluent for residual chlorine using an Accuvac ampule. If chlorine is present, aerate sample for several and check again. Continue aeration until no residual chlorine is detected.

## 12.02 Prepare seed solution:

- 12.02.1 Samples may be seeded using commercially prepared seed such as Polyseed, or using Influent.
- 12.02.2 If commercial seed is to be used, prepare in accordance with manufacturer's instructions.
- 12.02.3 If Influent is to be used as seed, prepare a dilution using buffered dilution water. Typically 200-300mL Influent diluted to 500mL will produce the desired strength.

## 12.03 Set up enough BOD bottles to allow 2 blanks, 2 standards, 2 seed controls, and sample duplicates.

## 12.04 Place 300mL of dilution water in BOD bottles labeled blanks Add 2-4mL seed solution to each BOD sample bottle. (Influent type samples may be run without seed and calculated accordingly). The amount of seed solution used will be determined using data from current batch seed controls. Adjustments should be made periodically to obtain seed control factors between 0.6 and 1.0.

## 12.05 Place 10ml and 20ml of seed in BOD bottles labeled Seed Control, and dilute to 300mL with dilution water.

## 12.06 Place 2ml and 3ml of standard solution in BOD bottles labeled standard, and dilute to 300mL with dilution water.

## 12.07 Place the appropriate sample dilution in its labeled BOD bottle. Influentes typically require 3mL diluted to 300mL, and effluents require 150 and a 90mL dilution to 300mL

## 12.08 Place probe in blanks, seeds, standards, and samples, and allow reading to stabilize. Take care not to trap bubbles under probe.

## 12.09 Once stable record DO and temperature and continue with next analysis after rinsing probe.

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- 12.10 After the readings have been documented, place stoppers in bottles, seal with water, and cap.
- 12.11 Incubate samples, standards, blanks, and seed for 5 days at 20 +/- 1°C.
- 12.12 After incubation analyze the DO of the batch and document results.

### **13.0 Data Analysis and Calculations**

- 13.01 DO measurements are read directly in mg/L.
- 13.02 Artificially seeded samples are calculated as:

$$\text{mg/L BOD} = \frac{(D_1 - D_2) - (S)V_s}{P}$$

Where:

- $D_1$  = Initial D.O. of seeded sample  
 $D_2$  = Final 5 day D.O. of seeded sample  
 $S$  =  $\Delta$  DO/mL seed suspension  
( $S=0$  for Unseeded Samples)  
 $V_s$  = Volume of seed in respective test bottle  
 $P$  = Decimal volumetric fraction of sample used (mL sample/300)

- 13.03 Non seeded samples are calculated as:

Same as above except that  $S=0$

### **14.0 Method Detection Limit and Method Performance**

- 14.01 MDL Studies are not performed for this method. An LOQ (Reporting Limit) of 2 mg/L has been established.
- 14.02 The laboratory tracks method performance through analysis of quality control samples; blanks, standards, duplicates.

### **15.0 Pollution Prevention**

- 15.01 Unused sample is disposed through the laboratory sink.
- 15.02 Waste created by the method is flushed down the drain with plenty of water.

### **16.0 Waste Management**

- 16.01 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management.
- 16.02 The laboratory only produces and maintains chemicals necessary to perform required analysis to minimize waste.

### **17.0**

**References**

16.1      Standard Methods 22nd Edition, Method 5210B.

**18.0 Tables, Diagrams, Flowcharts**

18.1      Not Applicable

**TIMPANOGOS SPECIAL SERVICE DISTRICT**  
**STANDARD OPERATING PROCEDURE**

**E.-COLI (COLILERT)**

**STANDARD METHODS 9223 B**

**Document Control # T-07**

**REVISION 04**

**EFFECTIVE DATE 06-15**

Signature of Approval:

Quality Assurance Officer:

Ryan J. Freeman

## **E.-COLI IN WASTEWATER**

### **CHROMOGENIC SUBSTRATE METHOD (COLILERT)**

#### **Scope**

This SOP covers analysis of e.-coli in water samples under the Clean Water Act. With the use of Idexx Quantitrays, an MPN determination can be made in the range of 0-200, or 0-2000 MPN/100mL. The objective of the method is wastewater treatment plant monitoring.

#### **Discussion**

E.-coli exists in nature as part of the normal flora in the intestines of mammals and thus serves as an indicator that fecal contamination of the water has occurred.

#### **Matrices**

Water and wastewater (aqueous) samples

#### **Reference Method(s)**

Method 9223B

"Standard Methods for the Examination of Water and Wastewater", 22<sup>nd</sup> Edition, 2012

#### **Method Summary**

Two nutrient indicators, ONPG and MUG are the major sources of carbon in Colilert and can be metabolized by the coliform enzyme  $\beta$ -galactosidase, and the *E. coli* enzyme  $\beta$ -glucuronidase, respectively. As coliforms grow in Colilert media, they use  $\beta$ -galactosidase to metabolize ONPG and change it from clear to yellow. *E. coli* uses  $\beta$ -glucuronidase to metabolize MUG and create fluorescence under UV light. Since most non-coliforms do not have these enzymes, they are unable to grow and interfere. The few non-coliforms that do have these enzymes are selectively suppressed by a special formulation in the media. This reduces or eliminates the problem of "Indeterminate" results normally inherent to the membrane filtration method.

#### **Definitions**

1. Aliquot- Measured portion of sample or solution used for analysis
2. DI Water- Deionized water, laboratory grade water that has been purified by deionization
3. MPN- Most Probable Number, a statistical estimation of bacterial density

#### **Deviations from Reference Method**

None

### **Method Detection and Reporting Limits**

Range of detection:

0 to 200 MPN/100mL using regular Quantitray

0 to 2000 MPN/100mL using Quantitray 2000

Approximate MDL = Not applicable

Approximate MRL = Not applicable

### **Interference Screening and Removal**

Each sample container comes from the manufacturer with enough sodium thiosulfate added to neutralize up to 15ppm chlorine. Excess chlorine will render a test invalid. Daily TRC analysis of an aliquot of sample confirms that chlorine levels are within the range of removal for the tablet.

### **Sample Collection, Preservation and Storage**

|                       |  |
|-----------------------|--|
| Container Type        | Sterile Plastic, Idexx brand or equivalent       |
| Minimum Sample Volume | 100mL  |
| Preservative          | Sodium Thiosulfate (added by manufacturer)       |
| Storage               | Cool (Less than or equal to 6°C, but not frozen) |
| Maximum Hold Time     | 6 hours  |

### **Equipment and Supplies**

1. Incubator- operating at  $35 \pm 0.5^{\circ} \text{ C}$
2. Long Wave UV Light- 366nm, 6 watt, in light shielded viewing box
3. Sample containers, 100mL with sodium thiosulfate
4. MPN Quantitrays
5. Quantitray Sealer
6. Test tubes, or Sample cups- 10mL disposable

### **Reagents and Standards**

1. Colilert Medium
2. Colilert Comparator

### **Calibration and Standardization**

Not applicable

### **Procedure**

1. Preheat the Quantitray sealer for 20 to 30 minutes or until green light comes on.
2. Mix the sample well by shaking or inverting at least 20 times.
5. If necessary, adjust the sample volume to 100 mL by pouring out excess sample. Use the 100-mL mark on the sample container as a reference.
6. Separate 1 pack of Colilert media from the strip taking care not to accidentally open adjacent pack.
7. Record the Colilert media lot number (embossed on each pack) on log-in sheet.
8. Tap the pack to ensure that all powder is in bottom.
9. Hold the pack at arm's length and snap the top back on the score line.
10. Pour the contents of the media pack into the sample and close the lid. Be sure that fingers do not contact the inside or lip of the sample container, or the opening of media pack.
11. Mix the sample thoroughly by alternately shaking and inverting the container until the media is dissolved completely.
12. Clearly mark the sample ID on the back of the Quantitray with a permanent marker.
13. Pour the contents of sample bottle into a Quantitray, avoiding contact with foil tab.
14. Place the Quantitray in the rubber insert and run it through the sealer.

13. Place the sealed Quantitray in the 35° incubator for a minimum of 24 hours and a maximum of 28 hours.
14. Count the number of cells that have a yellow color greater than or equal to the comparator and record on the data sheet as Total Coliform (TC)
15. Place the tray in the black light viewing box and count the number of TC positive cells that show a fluorescence greater than or equal to the comparator and record on the data sheet as E.-coli (EC).
16. Count the large compartment at the top as one cell.
17. For Quantitray 2000, count and record large and small cells separately.

### **Calculation and Reporting**

1. Calculate the MPN for E. Coli using the Quantitray MPN charts, or the Idexx software.

### **Quality Control**

#### **Media**

1. Obtain the Certificate of Analysis for each lot number from Idexx. Keep on file in the laboratory. No additional media QC is necessary if certificates are on file.

#### **Sample Containers**

Each lot of bottles received will be verified for sterility, volume accuracy, and chlorine removal efficiency. If the bottle manufacturer issues CofAs that contain all of the applicable information, a copy of the CofA may be kept on file in lieu of in-house testing.

#### **Corrective Actions for Out of Control Data**

Not Applicable

#### **Contingencies for Handling Out of Control or Unacceptable Data**

Not Applicable

#### **Method Performance and Demonstration of Capability**

Proficiency may be demonstrated by successful analysis of a PT sample, or other sample of known concentration. Comparison counts to a certified analyst may also be used if the new analyst performs the counts independently. At least 4 or 5 comparisons should be documented.

**Safety**

Due to the microbiological nature of samples, the following safety equipment should be worn by technicians: lab coat; safety glasses; and protective gloves.

**Waste Management**

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management.

The laboratory only produces and maintains chemicals necessary to perform required analysis to minimize waste.

**Additional References**

"Presence / Absence Test Procedure" Colilert Test Kit, IDEXX Laboratories Inc.

**Approval**

Signature\_\_\_\_\_

Date\_\_\_\_\_

Name\_\_\_\_\_

Title\_\_\_\_\_

**Minor Revisions**

| Date | Section | Comments | Approval |
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**TIMPANOGOS SPECIAL SERVICE DISTRICT**

**STANDARD OPERATING PROCEDURE**

**DISSOLVED OXYGEN**

**STANDARD METHODS 4500-O G**

**Document Control # T-03**

**REVISION 07**

**EFFECTIVE DATE 06-15**

Signature of Approval:

Quality Assurance Officer:

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Ryan J. Freeman

## **Dissolved Oxygen Method 4500-O G**

### **1.0 Scope and Application**

- 1.01 This method uses the luminescent DO (LDO) probe.
- 1.02 The method is applicable to all wastewater effluents.

### **2.0 Summary of Method**

- 2.02 The LDO sensor is coated with a luminescent material. Blue light from an LED is transmitted to the sensor surface. The blue light excites the luminescent material. As the material relaxes it emits red light. The time from when the blue light was sent and the red light is emitted is measured. The more oxygen that is present the shorter the time it takes for the red light to be emitted. This time is measured and correlated to the oxygen concentration. Between the flashes of blue light a red LED is flashed on the sensor and used as an internal reference.

### **3.0 Method Deviations**

- 3.01 Not Applicable

### **4.0 Definition of Terms**

- 4.01 A list of terminology, with their definitions, utilized by this procedure and the laboratory staff is located in the Laboratory Quality Assurance Plan

### **5.0 Interferences**

- 5.01 None known

### **6.0 Safety**

- 6.01 Due to the microbiological nature of samples, the following safety equipment should be worn by technicians: lab coat; safety glasses; and protective gloves.

**7.0    Equipment and Supplies**

    7.01 Portable D.O. meter with probe

**8.0    Reagents and Standards**

    8.01 Not Applicable

**9.0    Sample Collection, Preservation and Storage**

    9.01 Sample is collected in a 500mL plastic container and brought directly to the laboratory for analysis.

**10.0    Quality Control, Acceptance Criteria, Corrective Actions & Contingencies**

    10.01 Analyze sample in duplicate, duplicate results should agree within 0.2 mg/L.

    10.02 Corrective Actions:

        10.02.1 If the above acceptance criteria is not met, the analysis must be stopped, trouble shoot the problem, and re-analyze.

        10.02.2 Refer to the instruments manufacturers instruction manual and look under trouble shooting.

    10.03 Contingencies:

        10.03.1 If the problem cannot be solved or re-analysis is not possible (sample out of holding or insufficient sample size), the data must be flagged on the bench sheet and on the report.

**11.0    Calibration and Standardization**

    11.01 Turn probe on and allow to stabilize. The probe will automatically calibrate.

**12.0    Procedure**

    12.01 Place probe in sample (plant effluent) and allow to stabilize.

    12.02 Once stable record results.

    12.03 Rinse probe well with DI water between samples, if particles stick to the stirrer gently remove with a kim wipe.

**13.0 Data Analysis and Calculations**

13.01 DO measurements are read directly in mg/L.

**14.0 Method Detection Limit and Method Performance**

14.01 Not Applicable

**15.0 Pollution Prevention**

15.01 Unused sample is disposed through the laboratory sink.

**16.0 Waste Management**

16.01 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management.

16.02 The laboratory only produces and maintains chemicals necessary to perform required analysis to minimize waste.

16.03 This particular procedure does not generate materials more hazardous than plant waste.

**17.0 References**

17.01 Standard Methods 22nd Edition, Method 4500-O G.

**18.0 Tables, Diagrams, Flowcharts**

18.01 Not Applicable

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**TIMPANOGOS SPECIAL SERVICE DISTRICT**

**STANDARD OPERATING PROCEDURE**

**pH**

**STANDARD METHODS 4500-H+ B**

**REVISION 05**

**Document Control # T-05**

**EFFECTIVE DATE 06-15**

Signature of Approval:

Quality Assurance Officer:

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Ryan J. Freeman

**pH  
Standard Methods 4500 B**

**1.0 Scope and Application**

- 1.01 This method is applicable to the determination of the overall acidity or alkalinity of waters and wastewaters.

**2.0 Summary of Method**

- 2.01 pH is determined electrometrically with an electrode and ion analyzer.
- 2.02 A pH electrode is simply placed in a sample and compared against standards buffers.

**3.0 Method Deviations**

- 3.01 The laboratory pH probe does not specifically state "low sodium". According to Orion all pH probes manufactured today are low sodium probes.
- 3.02 The method states the holding time of pH to be immediate. DEQ allows a holding limit of 15 minutes for transportation from the sampling point to the laboratory.

**4.0 Definition of Terms**

- 4.01 A list of terminology, with their definitions, utilized by this procedure and the laboratory staff is located in the Laboratory Quality Assurance Plan.

**5.0 Interferences**

- 5.01 Sodium is known interferent at pH levels greater than 10, a low sodium electrode must be used to compensate
- 5.02 Oily or particulate materials will impair the electrode response. If this occurs the electrode may need cleaning with dilute acid, refer to the probe operation manual for details.
- 5.03 pH is temperature dependent. The instrument must have an ATC probe to correct for temperature differences in samples and buffers.

**6.0 Safety**

6.01 Due to the microbiological nature of samples, the following safety equipment should be worn by technicians: lab coat; safety glasses; and protective gloves.

**7.0 Equipment and Supplies**

7.01 pH or Ion Analyzer  
7.02 pH electrode  
7.03 ATC; temperature compensator  
7.04 Stir plate  
7.05 50mL, 100mL or 250mL beakers

**8.0 Reagents and Standards**

8.01 Deionized water  
8.02 Electrode filling solution  
8.03 pH Buffers: 4, 7, and 10  
8.04 Deionized water

**9.0 Sample Collection, Preservation and Storage**

9.01 Samples are collected in an unpreserved plastic container (4L), and brought directly to the laboratory for analysis. Analysis must occur within 15 minutes of collection.  
9.01.1 Analysis within 15 minutes is a DEQ requirement.

**10.0 Quality Control, Acceptance Criteria, Corrective Actions & Contingencies**

10.01 All quality control is to be performed per batch of 20 samples or less.  
10.02 Each batch must contain:  
    10.02.1 A calibration verification standard (beginning and ending)  
    10.02.2 All samples performed in duplicate  
10.03 Acceptance criteria is:  
    10.03.1 The calibration verification must be within 0.1 units of its true value  
    10.03.2 Duplicate samples must be within 0.1 units  
    10.03.3 The instrument slope must be 92 to 102%  
10.04 Corrective Action:  
    10.04.01 If the above acceptance criteria is not met, the analysis must be stopped, trouble shoot the problem, and re-analyze.  
        10.04.01.1 The calibration verification standard may be analyzed twice in attempts for a passing result.

If the second attempt fails, analysis must be stopped, maintenance performed, and the instrument re-calibrated.

**10.05 Contingencies:**

- 10.05.01 If the problem cannot be solved or re-analysis is not possible (sample out of hold or insufficient sample size), the data must be flagged on the bench sheet and on the report. The flag must be followed by an explanation of the failure.

**11.0 Calibration and Standardization**

- 11.01 Calibrate as per manufacturer's instructions.

**12.0 Procedure**

- 12.01 Remove electrode and probe from buffer and rinse with DI water  
12.02 Place electrode and probe in sample and document results and temperature, when meter indicates stable reading.  
12.03 Read successive aliquots of sample until 2 readings are within 0.1 pH units.

**13.0 Data Analysis and Calculations**

- 13.01 The pH meter reads directly in pH units. Report pH to the nearest 0.1 unit and temperature to the nearest degree C.

**14.0 Method Detection Limit and Method Performance**

- 14.01 MDL Studies are not applicable to this method.  
14.02 The laboratory tracks method performance through analysis of calibration verification standards and duplicates.

**15.0 Pollution Prevention**

- 15.01 Unused sample is disposed through the laboratory sink.  
15.02 Waste created by this method is flushed down the drain with plenty of water.

**16.0 Waste Management**

- 16.01 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management.  
16.02 The laboratory only produces and maintains chemicals necessary to perform required analysis to minimize waste.

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**17.0 References**

17.01 Standard Methods 22nd Edition, Method 4500-H+ B.

**18.0 Tables, Diagrams, Flowcharts**

18.01 Not Applicable

**Nitrogen, Ammonia  
Method HACH 10205**

**1.0 Identification of the Test Method**

- 1.1 HACH 10205

**2.0 Applicable Matrix**

- 2.1 Water, wastewater and industrial effluents.

**3.0 Limits of Detection**

- 3.1 MDL studies are performed annually or whenever there is a major change to the procedure or the instrumentation. Current MDL studies can be found in Appendix D of TSSD Laboratory's Quality Manual.

**4.0 Scope and Application**

- 4.1 For municipal and industrial wastewaters, environmental waters and watershed protection monitoring.

**5.0 Summary of Method**

- 5.1 A groundwater or wastewater (industrial or municipal) sample is added to HACH nitrogen, ammonia vials, left to sit for 15 minutes, and measured on a spectrophotometer.

**6.0 Definitions**

- 6.1 Definitions of terms are located in: Standard Methods 22nd Edition pages 1-4 & 1-5.
- 6.2 General laboratory terminology may be found in Appendix of Timpanogos Special Service District's Laboratory Quality Assurance Manual.

**7.0 Interferences**

- 7.1 Primary amines are determined and cause high-bias results. All reducing agents interfere and cause low-bias results.

## **8.0 Safety**

- 8.1 The following safety equipment should be used when performing this method: a lab coat, and protective gloves.

## **9.0 Equipment and Supplies**

- 9.1 HACH DR3900 Spectrophotometer
- 9.2 Pipettors
- 9.3 pH Meter/pH Probe
- 9.4 250mL Beakers

## **10.0 Reagents and Standards**

- 10.1 Deionized water.
- 10.2 Prepare a high range standard in the laboratory, a low range standard from a commercially prepared HACH 1mg/L NH<sub>3</sub>N standard, and spikes from a commercially prepared HACH 1000mg/L NH<sub>3</sub>N standard.
- 10.3 High range stock solution: dissolve 3.819g NH<sub>4</sub>Cl in deionized water and dilute to volume in a 1000mL volumetric flask = 1000mg/L. Good for 6 months.
  - 10.3.1 High range standard: 20mL of 1000mg/L high range stock solution (see 10.3) into a 500mL flask, dilute to volume with deionized water = 40mg/L. Prepare fresh before use.
- 10.4 Low range standard: 5mL of 1mg/L HACH NH<sub>3</sub>N standard into a 100mL flask, dilute to volume with deionized water = 0.05mg/L. Prepare fresh before use.
- 10.5 High range spiking solution: 10mL of 100mg/L HACH NH<sub>3</sub>N standard into a 250mL flask, dilute to volume with deionized water = 4mg/L. Good for 3 months.
- 10.6 Low range spiking solution: 1mL of 100mg/L HACH NH<sub>3</sub>N standard into a 250mL flask, dilute to volume with deionized water = 0.4mg/L. Good for 3 months.
- 10.7 HACH TNT830 and TNT832 vials: Ultra low range = 0.015-2.00mg/L, High range = 2-47mg/L.
- 10.8 Alkali solution for neutralization of preserved samples: dissolve 50g of NaOH into a 250mL flask, dilute to volume with deionized water = 5N.

## **11.0 Sample Collection, Preservation and Storage**

- 11.1 Samples are to be collected in cleaned plastic containers and analyzed the day of collection. If they are not analyzed the day of collection (weekend samples), they are preserved with 2mL HCl and refrigerated at 4°C until the time of analysis. Samples are to be analyzed within 28 days of sampling, and brought to room temperature and neutralized to pH 7.0 before analysis.

## **12.0 Quality Control**

- 12.1 All quality control is to be performed per batch, a batch being 20 samples or less. Quality control samples to be analyzed per vial strength are: a high blank, a high standard, a low blank, a low standard, a high matrix spike, a high matrix spike duplicate, or a low matrix spike, and a low matrix spike duplicate (high and low spikes/matrix spikes are to be alternated daily).
- 12.2 Both standards, matrix spikes, and matrix spike duplicates are to be checked for precision and accuracy as per section 18.0 below.

## **13.0 Calibration and Standardization**

- 13.1 This procedure is standardized by the analysis of a known sample.
- 13.2 Calibration of equipment used in this procedure shall be in accordance with procedures in TSSD's Laboratory Quality Assurance Manual.

## **14.0 Procedure**

- 14.1 Carefully remove the protective foil lid from the cap of the TNT vial. Unscrew the cap from the vial.
- 14.2 Pipette 0.2mL (for high range vials) or 5mL (for ultra low range vials) of sample into the vial (depending on the strength of the sample).
- 14.3 Flip the cap over so that the reagent side faces the vial. Screw the cap tightly onto the vial.
- 14.4 Shake the capped vial 2-3 times to dissolve the reagent in the cap. Verify that the reagent has dissolved by looking down through the open end of the cap.
- 14.5 Wait 15 minutes.
- 14.6 After 15 minutes, invert the sample an additional 2-3 times to mix.
- 14.7 Thoroughly clean the outside of the vial.
- 14.8 Insert the prepared vial into the cell holder. The instrument reads the barcode and performs the correct test. Results are in mg/L NH<sub>3</sub>N.
- 14.9 A blank is run first. Place blank into the spectrophotometer. Turn on the instrument reagent blank feature. The measured value of the blank should be displayed in the highlighted box. Accept this value for the high range blank vial. Manually calculate the low range standard true value by subtracting the blank value from the standard value. Show all calculations on the bench sheet.
- 14.10 Continue to read the remaining samples.

Note: If analyzing preserved samples, bring sample pH back to neutral using 5N NaOH prior to analysis.

## **15.0 Data Analysis and Calculations**

- 15.1 Instrument readings are displayed in mg/L.
- 15.2 The laboratory 40mg/L and 0.05mg/L standards must recover within  $\pm 15\%$ .
- 15.3 The matrix spike/matrix spike duplicate made with the HACH prepared standard must also recovery within  $\pm 15\%$  for accuracy.
- 15.4 The precision of the duplicates must recover within laboratory control limits.
- 15.5 If any of the above standards, spikes, or duplicates do not recover within acceptance limits stop the analysis and troubleshoot the problem. If the problem cannot be solved inform the Laboratory Director for assistance.

## **16.0 Method Performance**

- 16.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the values is above zero. The MDL achieved by this laboratory will be generated whenever there is a major change to the procedure or the instrumentation, with the actual values maintained in the laboratory files.

## **17.0 Pollution and Prevention**

- 17.1 Unused sample for this analysis is equivalent to plant effluent and disposed of through the laboratory sink.
- 17.2 Vials are unscrewed and poured into the sink while water is running, then recapped and thrown into a trash receptacle.

## **18.0 Data Assessment and Acceptance Criteria for Quality Control Measures**

- 18.1 The acceptable range for standard recovery is  $\pm 15\%$ .
- 18.2 The acceptable range for duplicate precision is  $\pm 10\%$ .
- 18.3 Spike recovery must be  $\pm 15\%$  because the spike is also the second source standard for this analysis.

## **19.0 Corrective Actions for Out of Control Data**

- 19.1 If quality control measures are out of the acceptable ranges, the samples will be held until the problem is corrected at which time they will be re-analyzed or if unable to hold samples, the data will be flagged and the Quality Assurance Officer will review the data to determine if a CAR needs to be initiated. If the problem is obvious to the laboratory technician, they can also generate a CAR.

## **20.0 Contingencies for Handling Out of Control Data**

- 20.1 If any of the above standards, duplicates or blanks do not recover within acceptance limits stop the analysis and troubleshoot the problem. Once the problem has been corrected, re-analyze the batch.
- 20.2 Samples may be sub-contracted to an outside lab for analysis.

## **21.0 Waste Management**

- 21.1 It is the laboratory's responsibility to comply with all federal, state and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

## **22.0 References**

- 22.1 HACH 10205 Method Standard Procedure.
- 22.2 EPA Method 350.1.
- 22.3 HACH DR3900 Handbook.
- 22.4 HACH DRB200 Handbook.

## **23.0 Tables, Diagrams, Flowcharts and Validation Data**

- 23.1 See attached Figure 23.1 for Ammonia bench sheet.

## **TIMPANOGOS SPECIAL SERVICE DISTRICT STANDARD OPERATING PROCEDURE**

Figure 23.1

**Phosphorus, Total and Ortho  
Methods HACH 10210 & 10209**

**1.0 Identification of the Test Method**

1.1 HACH 10210 & 10209

**2.0 Applicable Matrix**

2.1 Water, wastewater and industrial effluents.

**3.0 Limits of Detection**

3.1 MDL (Method Detection Limit) studies are performed whenever there is a major change to the procedure or the instrumentation. Current MDL Studies can be found in Appendix D of TSSD Laboratory's Quality Manual.

**4.0 Scope and Application**

4.1 For wastewater, drinking water, boiler water, surface water and process water.

**5.0 Summary of Method**

5.1 A wastewater (industrial or municipal) sample is added to a HACH phosphorus vial, digested in a reactor for 1 hour (only method HACH 10210 requires digestion), then a reagent is added and the vial is left to sit for 10 minutes, and measured on a spectrophotometer.

**6.0 Definitions**

6.1 Definitions of terms are in: Standard Methods 22nd Edition pp 1-4 & 1-5.

- 6.2 General laboratory terminology may be found in Definition of Terms in Timpanogos Special Service District's Laboratory Quality Assurance Manual.

## 7.0 Interferences

- 7.1 The ions listed in the *Interfering Substances* table in the HACH 10210 & 10209 Method Standard Procedure have been individually tested up to the given concentrations and do not cause interference. The cumulative effects of these ions or the influence of other ions have not been determined.

## 8.0 Safety

- 8.1 The following safety equipment should be used when performing this method: a lab coat, and protective gloves.

## 9.0 Equipment and Supplies

- 9.1 HACH DR3900 Spectrophotometer
- 9.2 HACH DBR200 Reactor
- 9.3 Pipettor

## 10.0 Reagents and Standards

- 10.1 Deionized water.
- 10.2 High range standard: commercially prepared or in-lab prepared reagent 10mg/L PO<sub>4</sub> standard.
- 10.3 Low range standard: 10mL of a commercially prepared or lab prepared 3mg/L PO<sub>4</sub> standard into a 100mL flask, dilute to volume with deionized water = 0.3mg/L. Prepare fresh before use.
- 10.4 High and low range spiking solution: commercially prepared or lab prepared 2mg/L PO<sub>4</sub> standard.
- 10.5 5N NaOH.
- 10.6 HACH TNT843 low range vials.

10.7 HACH TNT844 high range vials.

## 11.0 Sample Collection, Preservation and Storage

11.1 Samples are to be collected in clean plastic containers, and analyzed the day of collection. Samples to be analyzed for total phosphorus: preserve with 2mL sulfuric acid, store at 4°C and analyze within 28 days. Samples to be analyzed for reactive phosphorus: do not preserve with acid, store at 4°C and analyze within 48 hours. Before analysis, neutralize preserved samples with 5N NaOH and warm to room temperature.

## 12.0 Quality Control

- 12.1 All quality control is to be performed per batch, a batch being 20 samples or less. Quality control samples to be analyzed per vial set are: a blank, a standard, a matrix spike, and a matrix spike duplicate. High and low range spikes/spike duplicates are to be alternated between the high and low range vial sets.
- 12.2 The standards, matrix spikes and matrix spike duplicates are to be plotted on quality control charts to access precision and accuracy.

## 13.0 Calibration and Standardization

- 13.1 This procedure is standardized by the analysis of a known sample.
- 13.2 Calibration of equipment used in this procedure shall be in accordance with procedures in TSSD's Laboratory Quality Assurance Manual.

## 14.0 Procedure

### Method 10210 – Total Phosphorus:

- 14.1 Turn on the reactor and heat to 100°C.

- 14.2 Carefully remove the protective foil lid from the vial. Unscrew the cap from the vial.
- 14.3 Pipette 0.5mL (for high range vials) or 2.0mL (for low range vials) of sample into the reagent vial (depending on the strength of the sample).
- 14.4 Flip the cap over so that the reagent side on the cap faces in to the vial. Screw the cap tightly onto the vial.
- 14.5 Shake the capped vial 2-3 times to dissolve the reagent in the cap. Verify the reagent has dissolved by looking down through the open end of the cap lid.
- 14.6 Insert the vial into the reactor. Close the protective cover and heat for one hour at 100°C.
- 14.7 After the timer expires, carefully remove the hot vial from the reactor. Insert it in the cooling rack and allow to cool to room temperature.
- 14.8 Pipet 0.2mL of reagent B into the cooled vial. Immediately close the reagent B container.
- 14.9 Screw the grey cap C onto the vial.
- 14.10 Invert the capped vial 2-3 times to dissolve the reagent in the grey cap.
- 14.11 Wait 10 minutes.
- 14.12 When the timer expires, invert the vial again 2-3 times.
- 14.13 Clean the outside of the vial and insert it into the cell holder. The instrument reads the barcode, then selects the correct test. Results are in mg/L PO<sub>4</sub>.
- 14.14 A blank is run first on the spectrophotometer. Record value on bench sheet.
- 14.15 Continue to read the remaining samples.

**Method 10209 – Reactive Phosphorus:**

- 14.1 Unscrew the cap from the vial.
- 14.2 Pipette 0.5mL (for high range vials) or 2.0mL (for low range vials) of sample into the reagent vial (depending on the strength of the sample).
- 14.3 Pipet 0.2mL of reagent B into the vial. Immediately close the reagent B container.
- 14.4 Screw the grey cap C onto the vial.
- 14.5 Invert the capped vial 2-3 times to dissolve the reagent in the grey cap.
- 14.6 Wait 10 minutes.

- 14.7 When the timer expires, invert the vial again 2-3 times.
- 14.8 Clean the outside of the vial and insert it into the cell holder. The instrument reads the barcode, then selects the correct test. Results are in mg/L PO<sub>4</sub>.
- 14.9 A blank is run first. Place blank into the spectrophotometer and record value.
- 14.10 Continue to read the remaining samples.

## 15.0 Data Analysis and Calculations

- 15.1 Instrument readings are displayed in mg/L PO<sub>4</sub>. Total PO<sub>4</sub>-P and Ortho PO<sub>4</sub>-P = PO<sub>4</sub> ÷ 3.
- 15.2 The laboratory blank must record lower than the laboratory MDL.
- 15.3 The laboratory standards and spikes must recover within ± 10%.
- 15.4 If the standards or spikes do not recover within acceptance limits, re-analyze the parameter in question. If the QC is still out of range, flag the data and troubleshoot the problem. If the problem cannot be solved inform the Laboratory Director for assistance.

## 16.0 Method Performance

- 16.1 The LOQ is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the values are above zero. The LOQ achieved by this laboratory will be generated whenever there is a major change to the procedure or the instrumentation, with the actual values maintained in the laboratory files. Once the MDL had been established, there will be an annual reporting limit check.

## 17.0 Pollution and Prevention

- 17.1 Unused sample for this analysis is equivalent to plant effluent and disposed of through the laboratory sink.
- 17.2 Vials are unscrewed and poured into the sink while water is running, then recapped and thrown into a trash receptacle.

**18.0 Data Assessment and Acceptance Criteria for Quality Control Measures**

18.1 The acceptable range for duplicate precision is  $\pm 10\%$ .

**19.0 Corrective Actions for Out of Control Data**

19.1 If quality control measures are out of the acceptable ranges, the samples will be held until the problem is corrected at which time they will be re-analyzed or if unable to hold samples, the data will be flagged and the Laboratory Director and/or Quality Assurance Officer will review the data to determine if a CAR needs to be initiated. If the problem is obvious to the laboratory technician, they may also generate a CAR.

**20.0 Contingencies for Handling Out of Control Data**

- 20.1 If either standards and/or spikes do not recover within acceptance limits stop the analysis and troubleshoot the problem. Once the problem has been corrected, re-analyze the parameter in question.
- 20.2 Samples may be sub-contracted to an outside lab for analysis.

**21.0 Waste Management**

21.1 It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

**22.0 References**

22.1 HACH 10210 & 10209 Method Standard Procedures.

22.2 HACH DR3900 Handbook.

## TIMPANOGOS SPECIAL SERVICE DISTRICT STANDARD OPERATING PROCEDURE

## 22.3 HACH DRB200 Handbook.

## 23.0 Tables, Diagrams, Flowcharts and Validation Data

23.1 See attached Figure 23.1 for Total and Ortho Phosphorus bench sheet.

**TSSD Laboratory**  
**Total-Phosphorus, ortho-Phosphorus**

**HACH TNT 843, 844**

Rev 1.0

Analysis Date: \_\_\_\_\_

Tech: \_\_\_\_\_

Sample ID# \_\_\_\_\_

Spectrophotometer ID:

DR3900

1.0 mg/L Std ID: \_\_\_\_\_

HR Vial ID: \_\_\_\_\_

LR Vial ID: \_\_\_\_\_

| Sample ID             | Total                               | Total                            | Ortho                         | Ortho                            |
|-----------------------|-------------------------------------|----------------------------------|-------------------------------|----------------------------------|
|                       | LR PO4-P mg/L<br>(0.005-1.50 range) | HR PO4-P mg/L<br>(0.5-5.0 range) | LR PO4-P mg/L<br>(0.005-1.50) | HR PO4-P mg/L<br>(0.5-5.0 range) |
| Blank                 |                                     |                                  |                               |                                  |
| HR Std 1.0            |                                     |                                  |                               |                                  |
| LR Std 1.0            |                                     |                                  |                               |                                  |
| INF Grab              |                                     |                                  |                               |                                  |
| INF Grab              | Spk                                 |                                  |                               |                                  |
| INF Grab              | Spk/Dup                             |                                  |                               |                                  |
| EFF Grab              |                                     |                                  |                               |                                  |
| EFF Grab              | Spk                                 |                                  |                               |                                  |
| EFF Grab              | Spk/Dup                             |                                  |                               |                                  |
| W#1 bioreactor        |                                     |                                  |                               |                                  |
| W#2 bioreactor        |                                     |                                  |                               |                                  |
| W#3 bioreactor        |                                     |                                  |                               |                                  |
| W#4 bioreactor        |                                     |                                  |                               |                                  |
| E#1 bioreactor        |                                     |                                  |                               |                                  |
| E#2 bioreactor        |                                     |                                  |                               |                                  |
| E#3 bioreactor        |                                     |                                  |                               |                                  |
| E#4 bioreactor        |                                     |                                  |                               |                                  |
| W#1 Clarifier         |                                     |                                  |                               |                                  |
| W#2 Clarifier         |                                     |                                  |                               |                                  |
| W#3 Clarifier         |                                     |                                  |                               |                                  |
| W#4 Clarifier         |                                     |                                  |                               |                                  |
| E#2 Clarifier         |                                     |                                  |                               |                                  |
| E#3 Clarifier         |                                     |                                  |                               |                                  |
| Effluent              |                                     |                                  |                               |                                  |
| LAKE (pond discharge) |                                     |                                  |                               |                                  |

Comments: \_\_\_\_\_  
 \_\_\_\_\_

**QC Data Review:**

Acceptable Std Range:  $\pm 15\%$       Reviewed By: \_\_\_\_\_

Acceptable Dup Range:  $\pm 10\%$       Date: \_\_\_\_\_

## TKN

### Method HACH 10242 (EPA 351.2)

#### 1.0 Identification of the Test Method

##### 1.1 HACH 10242 (EPA 351.2)

#### 2.0 Applicable Matrix

##### 2.1 Water, wastewater and industrial effluents.

#### 3.0 Limits of Detection

##### 3.1 MDL (Method Detection Limit) studies are performed whenever there is a major change to the procedure or the instrumentation. Current MDL study results can be found in Appendix D of TSSD Laboratory's Quality Manual.

#### 4.0 Scope and Application

##### 4.1 For wastewater, drinking water, surface water and mineral water.

#### 5.0 Summary of Method

##### 5.1 A wastewater (industrial or municipal) sample is added to an empty reaction vial along with two reagents, and digested in a reactor for 1 hour. Following digestion, a reagent is added to the reaction vial, mixed and transferred to a HACH s-TKN Test Vial #1 along with a fourth reagent. In a separate HACH s-TKN Test Vial #2, undigested sample is added along with a reagent. Both Test Vials are left to sit for 15 minutes, and measured in sequence (test Vial #1, then Test Vial #2) on a spectrophotometer.

#### 6.0 Definitions

## TIMPANOGOS SPECIAL SERVICE DISTRICT STANDARD OPERATING PROCEDURE

- 6.1 Definitions of terms are located in: Standard Methods 22nd Edition pages 1-4 & 1-5.
- 6.2 General laboratory terminology may be found in Definition of Terms in Timpanogos Special Service District's Laboratory Quality Assurance Manual.

### **7.0 Interferences**

- 7.1 High levels of oxidizable organic substances (COD) affect the reagent color and give high results. This test can only be used for wastewater when the COD level is less than 500mg/L COD.

### **8.0 Safety**

- 8.1 The following safety equipment should be used when performing this method: a lab coat, and protective gloves.

### **9.0 Equipment and Supplies**

- 9.1 HACH DR3900 Spectrophotometer
- 9.2 HACH DBR200 Reactor
- 9.3 Pipettor
- 9.4 Beakers
- 9.5 Volumetric flasks

### **10.0 Reagents and Standards**

- 10.1 Deionized water.
- 10.2 Standard solution: commercially prepared or in-lab prepared standard or PT sample of acceptable strength.
- 10.3 Spiking solution: commercially prepared or in-lab prepared 3.56mg/L TKN (second source) standard.

- 10.4 5N NaOH.
- 10.5 Empty reaction vials.
- 10.6 HACH TNT880 vials.

## 11.0 Sample Collection, Preservation and Storage

- 11.1 Analyzed within the day sampled, or preserve samples with 2mL sulfuric acid, store at 4°C and analyze within 28 days. Before analysis, neutralize preserved samples with 5N NaOH and warm to room temperature.

## 12.0 Quality Control

- 12.1 All quality control is to be performed per batch, a batch being 20 samples or less. Quality control samples to be analyzed are: a blank, a standard, a matrix spike, and a matrix spike duplicate.
- 12.2 The standard, matrix spike and matrix spike duplicate are to be plotted on quality control charts to access precision and accuracy.

## 13.0 Calibration and Standardization

- 13.1 This procedure is standardized by the analysis of a known sample.
- 13.2 Calibration of equipment used in this procedure shall be in accordance with procedures in TSSD's Laboratory Quality Assurance Manual.

## 14.0 Procedure

- 14.1 Before analysis, neutralize preserved samples with 5N NaOH and warm to room temperature.
- 14.2 Turn on the reactor and heat to 100°C.
- 14.3 Pipette 1.3mL of sample, 1.3 mL of Solution A and 1 reagent B tablet in quick succession to a clean, dry reaction vial. Close the reaction tube immediately, do not invert.

- 14.4 Insert the vial into the reactor. Close the protective cover and heat for one hour at 100°C.
- 14.5 After the timer expires, carefully remove the hot vial from the reactor. Insert it in the cooling rack and allow to cool to room temperature.
- 14.6 Once the vial has cooled, remove the cap and add 1 Micro Cap C to the vial.
- 14.7 Cap and invert the vial several times until no more streaks can be seen in the vial solution.
- 14.8 Pipette 0.5mL of the digested sample from the reaction vial into a Test Vial #1 (red label).
- 14.9 Pipette 0.2mL of solution D into the test vial and quickly cap and invert it until no more streaks can be seen in the solution. Proceed immediately to next step.
- 14.10 Pipette 1.0mL of undigested sample into a Test Vial #2 (green label).
- 14.11 Pipette 0.2mL of Solution D into the test vial and quickly cap and invert it until no more streaks can be seen in the solution.
- 14.12 Wait 15 minutes.
- 14.13 After the timer expires, wipe the Test Vial #1 and insert it into the cell holder. The instrument reads the barcode and displays E1. Proceed immediately to next step.
- 14.14 Wipe the Test Vial #2 and insert it into the cell holder. The instrument reads the barcode. Results are in mg/L Total N, mg/L NO<sub>3</sub>-N + NO<sub>2</sub>-N and mg/L TKN.
- 14.15 A blank is run first. Place blank into the spectrophotometer. Record value on bench sheet.
- 14.16 Continue to read the remaining samples and record results.

## 15.0 Data Analysis and Calculations

- 15.1 Instrument readings are displayed in mg/L.
- 15.2 The laboratory blank must record lower than the laboratory MDL.

- 15.3 The laboratory standard and spikes must recover within  $\pm$  10%.
- 15.4 If the standard or spikes do not recover within acceptance limits, re-analyze the parameter in question. If the QC is still out of range, flag the data and troubleshoot the problem. If the problem cannot be solved inform the Laboratory Director for assistance.

## 16.0 Method Performance

- 16.1 The LOQ is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the values is above zero. The LOQ achieved by this laboratory will be generated whenever there is a major change to the procedure or the instrumentation, with the actual values maintained in the laboratory files.

## 17.0 Pollution and Prevention

- 17.1 Unused sample for this analysis is equivalent to plant effluent and disposed of through the laboratory sink.
- 17.2 Vials are unscrewed and poured into the sink while water is running, then recapped and thrown into a trash receptacle.

## 18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

- 18.1 The acceptable range for standard and spike recovery is  $\pm$  10%.

## 19.0 Corrective Actions for Out of Control Data

- 19.1 If quality control measures are out of the acceptable ranges, the samples will be held until the problem is corrected at which time they will be re-analyzed or if unable to hold samples, the data will be flagged and the Laboratory Director and/or Quality Assurance Officer will review the data to determine if a CAR needs to be initiated. If the problem is obvious to the laboratory technician, they may also generate a CAR.

**20.0 Contingencies for Handling Out of Control Data**

- 20.1 If the standard and/or spikes do not recover within acceptance limits stop the analysis and troubleshoot the problem. Once the problem has been corrected, re-analyze the parameter in question.
- 20.2 Samples may be sub-contracted to an outside lab for analysis.

**21.0 Waste Management**

- 21.1 It is the laboratory's responsibility to comply with all federal, state and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

**22.0 References**

- 22.1 EPA 351.2
- 22.2 HACH 10242 Method Standard Procedure.
- 22.3 HACH DR3900 Handbook.
- 22.4 HACH DRB200 Handbook.

**23.0 Tables, Diagrams, Flowcharts and Validation Data**

- 23.1 See attached Figure 23.1 for s-TKN bench sheet.

TSSD Laboratory  
TKN  
**HACH 10242**  
Rev 1.0

Date: \_\_\_\_\_

Analyst:

\_\_\_\_\_ mg/L Std ID: \_\_\_\_\_

Spectrophotometer ID: DR3900

3.56mg/L HACH Spk ID: \_\_\_\_\_ (second source)

Reactor ID: DRB200

Vial Set ID:

Spk TV: \_\_\_\_\_

### Preserved Samples:

Final Inf pH:

Final Eff pH: \_\_\_\_\_

NaOH ID: \_\_\_\_\_

Did all samples have an initial pH < 2 ?

YES \_\_\_\_\_

NO

**Comments:** \_\_\_\_\_

## QC Data Review:

Acceptable Std Range:  $\pm 10\%$

Acceptable Dup Range:  $\pm 10\%$

Reviewed By:

Date:

**TIMPANOGOS SPECIAL SERVICE DISTRICT STANDARD OPERATING PROCEDURE**

Revision 06

Revised BY: RJF

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**TIMPANOGOS SPECIAL SERVICE DISTRICT  
STANDARD OPERATING PROCEDURE**

**TOTAL & VOLATILE SUSPENDED SOLIDS**

**STANDARD METHODS 2540 D & E**

**REVISION 06**

**Document Control # T-04**

**EFFECTIVE DATE 06-15**

Signature of Approval:

Quality Assurance Officer:

Ryan J. Freeman

## **Total Suspended Solids and Volatile Suspended Solids Method 2540 D & E**

### **1.0 Scope and Application**

- 1.01 This method is applicable to drinking, surface, and saline waters, domestic and industrial waste.<sup>2</sup>
- 1.02 The practical range of determination is 5mg/L to 20,000mg/L.<sup>2</sup>

### **2.0 Summary of Method**

- 2.01 A well mixed sample is filtered through a glass fiber filter, and the residue is dried at 103 - 105<sup>0</sup>C.<sup>2</sup> The weight of dried residue is reported as mg/L TSS.<sup>1</sup>
- 2.02 After the TSS has been calculated the filter is transferred to a furnace set at 550<sup>0</sup>C and ignited for a maximum of 30 minutes.

### **3.0 Method Deviations**

- 3.01 The laboratory has demonstrated that on routine Influent and Effluent TSS samples, 1 hour of drying time produces a constant weight and repeat cycles are not necessary. A study was conducted on plant samples in 2007 and is on file in the laboratory.
- 3.02 Graduated cylinders are used to measure sample aliquots.
- 3.03 Aliquots are taken after vigorous mixing of the sample. A magnetic stirrer is not used during the measurement of the analysis aliquot.

### **4.0 Definition of Terms**

- 4.01 A list of terminology, with their definitions, utilized by this procedure and the laboratory staff is located in the laboratory Quality Assurance Plan.

### **5.0 Interferences**

- 5.01 The temperature at which residues are dried and length of heating impact results.
- 5.02 Analysis of non-homogeneous samples will result in erroneous results. Thoroughly mix all samples before analysis, excluding large floating particles or submerged agglomerates of non-homogeneous materials from the sample.
- 5.03 Highly mineralized water, calcium, magnesium, chloride and sulfate, may be hygroscopic and require prolonged drying.

# **TIMPANOGOS SPECIAL SERVICE DISTRICT STANDARD OPERATING PROCEDURE**

Revision 06

Revised BY: RJF

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## **6.0 Safety**

- 6.01 Due to the microbiological nature of samples analyzed, the following safety equipment should be worn: lab coat; safety glasses; and protective gloves.

## **7.0 Equipment and Supplies**

- 7.01 Analytical balance, capable of weighing to 0.1mg
- 7.02 Drying oven, capable of maintaining 103 - 105<sup>0</sup>C
- 7.03 Muffle furnace, capable of maintaining 500 +/- 50<sup>0</sup>C
- 7.04 Membrane filter funnel with base
- 7.05 Suction assembly
- 7.06 Glass fiber filters
- 7.07 Graduated cylinders of various sizes
- 7.08 Aluminum dishes or watch glasses
- 7.09 Vacuum source

## **8.0 Reagents and Standards**

None

## **9.0 Sample Collection, Preservation and Storage**

- 9.01 Samples are to be collected in cleaned glass or plastic containers, and refrigerated at 4<sup>0</sup>C until time of analysis. Samples are to be analyzed within 7 days of sampling, and brought to room temperature before analysis.

## **10.0 Quality Control, Acceptance Criteria, Corrective Actions & Contingencies**

- 10.01 All quality control is performed per batch, 10 samples or less. A sample duplicate is analyzed for quality control.

- 10.02 Acceptance sample duplicates are based on laboratory derived criteria.

10.03 Corrective Actions:

- 10.03.1 If the above acceptance criteria is not met, the analysis must be stopped, trouble shoot the problem, and re-analyze.
- 10.03.2 Common problems may be caused from inadequate mixing of solutions or samples, old standard solutions, or inadequate drying or desiccation of filters.

10.04 Contingencies:

- 10.04.1 Re-analyze samples and duplicate. If there is not enough sample for re-analysis data must be flagged.

**11.0 Calibration and Standardization**

11.01 Standardization of this method is validated by the analysis of the control standard and the calibration of the laboratory balance.

**12.0 Procedure**

- 12.01 Connect a clean filter funnel with its base to the filter assembly.
- 12.02 Place glass fiber filter on filter funnel base (wrinkle side up).
- 12.03 Apply vacuum and wash filter with three successive 20mL portions of DI water.
- 12.04 Allow each washing to drain before adding another.
- 12.05 After washing apply vacuum for approximately three minutes.
- 12.06 Remove the filter from filtration apparatus and place in a labeled aluminum dish or watch glass.
- 12.07 Dry washed filters in oven for a minimum of 1 hour at 103 - 105<sup>0</sup>C.
- 12.08 Cool in desiccator and weigh on analytical balance.
- 12.10 Assemble filtering apparatus, and place tared filter on funnel base.
- 12.11 Wet filter with a minimum of deionized water
- 12.12 Shake sample to homogenize, and transfer to a graduated cylinder.
- 12.13 Transfer the measured volume of sample to the filtration funnel with vacuum source on.
- 12.14 Wash filter and cylinder with three successive 10mL portions of deionized water, allow each washing to drain before continuing.
- 12.15 After washing apply vacuum for approximately three minutes
- 12.16 Place filter with solids in a labeled dish and dry in oven for a minimum of 1 hour at 103 – 105 °C.
- 12.17 Remove dishes from the oven and place in a desiccator to cool. Weigh on the analytical balance.
- 12.19 Ignite residue in a muffle furnace at 500 +/- 50 ° C to a constant weight (15-30 minutes).
- 12.20 After ignition place dishes in desiccator to cool to room temperature.
- 12.21 Weigh ash on an analytical balance, and record results.

**13.0 Data Analysis and Calculations**

13.01 mg/L TSS = (wt of filter + dry residue in g – wt of filter in g) x 1,000,000 / mL sample volume

13.02 mg/L VSS = (wt. of filter + dry residue before ignition in mg – wt of filter + residue after ignition) x 1000 / mL sample volume

**14.0 Method Detection Limit and Method Performance**

14.01 MDL Studies are not performed for this method. An LOQ (Reporting Limit) of 5 mg/L has been established.

14.02 The laboratory tracks method performance through analysis of duplicate samples.

**15.0 Pollution Prevention**

15.01 Unused sample for this analysis is equivalent to plant influent, and should be disposed of through the laboratory sink.

15.02 Filters with residue may be disposed with laboratory trash.

**16.0 Waste Management**

16.01 The laboratory will comply with all rules governing waste and hazardous materials. This particular method does not produce material more hazardous than plant influent.

**17.0 References**

17.01 Standard Methods 22nd Edition, Method 2540 D & E.

**18.0 Tables, Diagrams, Flowcharts**

18.01 Not Applicable