

Department of Civil Engineering College of Engineering King Saud University

Water & Wastewater Laboratory CE 443

Laboratory Handout

Prepared by: Dr. Mohamed A. Othman Eng. Mohamed Misbah

> RABI' II 1437 January 2016

Table of Contents

Lab #	Experiment	Page
	Introduction	1
Lab 1	pH – Alkalinity – Turbidity – Conductivity	10
Lab 2	Hardness – Chlorides	16
Lab 3	Sulfates – Total Solids (TS) – Total Dissolved Solids (TDS) – Total Suspended Solids (TSS)	20
Lab 4	Coagulation & Flocculation	27
Lab 5	Total and Fecal Coliform	31
Lab 6	Chlorine Residual	36
Lab 7	Biochemical Oxygen Demand (BOD)	40
Lab 8	Chemical Oxygen Demand (COD)	43
Lab 9	Ammonia-Nitrogen (NH ₃ -N)	45
Lab 10	Total Phosphorus	48
	Appendices	52

Introduction

Preface

Agencies like waterworks, industrial organization, water pollution control boards are required to analysis water/wastewater samples. The purpose of the analysis is to know the exact composition of the sample at the particular point of time of sample collection. After the analysis is over, the results may be interpreted to suit a particular purpose of either surveillance of water quality, of effluent quality or to assess the performance of wastewater treatment plant.

Scope of the Course

- 1. Supply background information on the nature, source and environmental significance of analytes.
- 2. Provide step-by-step procedures for the most commonly performed water and wastewater tests.
- 3. Guide students in interpreting analytical data.
- 4. Present overview of quality control concepts and procedures as well as general reporting requirements.

Goals of the Course

At the end of the course, the student should:

- 1. Have a practical understanding of the technical aspects of environmental testing.
- 2. Be able to collect, preserve and analyze water and wastewater for the most common parameters in a manner acceptable to regulatory agencies.
- 3. Understand and be able to implement basic quality control and reporting procedures.

1. GLOSSARY

Analyte: Is a substance or chemical constituent that is of interest in an analytical procedure.

Colorimetric Measurement: A method for measuring unknown concentrations of analytes in a sample by measuring the sample's color intensity. The color of the sample after adding specific chemicals (reagents) is compared with colors of known concentrations.

<u>Composite Sample:</u> A collection of individual samples obtained at set intervals over a period of time.

<u>Dilution</u>: Lowering the concentration of a solution by adding more solvent (usually distilled water).

<u>Effluent</u>: The output or discharge from a water treatment process.

Grab Sample: A single sample of wastewater.

<u>Gravimetric Measurement</u>: A method for measuring unknown concentrations of analytes in a sample by weighing a precipitate or residue of the sample.

Holding time: The storage time allowed between sample collection and sample analysis when the required preservation and storage techniques are followed.

Influent: Wastewater or other liquid flowing into a reservoir, basin, treatment process or treatment plant.

Inorganic: Commonly referred to as mineral, it includes all matter that is not animal or vegetable. Inorganic substances normally dissociate in water to form ions.

<u>Ions</u>: An atom or group of atoms with an electrical charge that are positive (cation) or negative (anion).

<u>Micro:</u> Prefix meaning millionth, as in microgram (μ g) and microliter (μ L).

Milli: Prefix meaning thousandth, as in milligram (mg) and milliliter (mL).

<u>Organic</u>: Organic matter is a broad category that includes both natural and man-made molecules containing carbon and usually hydrogen. All living matter is made up of organic matter.

Pathogens: Disease-causing organisms.

Preservative: A chemical or reagent added to a sample to prevent or slow decomposition or degradation of the analyte to be tested.

<u>Representative Sample:</u> A portion of water identical in content to that in the larger body of water being sampled.

Solution: A liquid mixture of dissolved substances.

<u>Standard Solution</u>: A solution in which the exact concentration of a chemical or compound is known.

Standardize: To compare with a standard. In wet chemistry, the strength of an unknown can be determined by comparison with a standard. Furthermore, instruments are adjusted to read accurately by using standards.

Units of measure:

centimeter (cm) = 1/100 meter centigrade (°C) = 5/9 (°F - 32) Fahrenheit (°F) = $1.8 \times$ (°C) + 32 gram (g) = 0.035 ounces kilogram (Kg) = 2.2 pounds liter (L) = 0.26 U.S. gallons meter (m) = 39.37 inches microgram (µg) = 1/1,000,000 gram milligram (mg) = 1/1,000 gram milligram (mg) = 1/1,000 liter parts per billion (ppb) = $1 \mu g/L$ parts per million (ppm) = 1 mg/L

2. GENERAL LABORATORY SAFETY

Safety rules in the laboratory must be closely followed to provide a healthy work environment for laboratory staff members. In any water and wastewater laboratory, there are corrosive and toxic chemicals, toxic fumes, hot glassware and disease-causing organisms in samples posing threats of fire, chemical burns, toxicity and disease to laboratory workers.

A vast majority of laboratory accidents are the result of carelessness, inattention to established safety guidelines, or lack of good common sense. At a minimum, the following safety equipment should be provided and be readily available in the laboratory to ensure a safe workplace:

Eye washes device. Emergency deluge shower Fire blanket Safety glasses/goggles Laboratory apron/coat Surgical or rubber gloves Fire extinguisher (A, B, & C fire) Spill clean up kit

2.1. Material Safety Data Sheets

Laboratory workers should be aware of all relevant safety information on the chemicals and reagents used in their workplace. It is required that this safety information on chemical handling and usage be provided to all workers on documents called Material Safety Data Sheets (MSDS).

MSDS provides valuable information with regard to the safe handling of chemicals, their storage, hazards, first aid and disposal. All chemical manufacturers, even for laboratory reagents, are required to provide MSDS. If you do not have these sheets for all the reagents or chemicals you have purchased, they will be supplied on request from your chemical supply company. The MSDS should be kept in or near the laboratory for reference in case of an emergency.



2.2. Basic Laboratory Safety Tips

The following are some basic safety tips to follow when working in the laboratory:

- 1. Use proper safety goggles or a face shield when performing any test where there is a potential danger to the eyes. **CAUTION**: Never look into the open end of a container during a chemical reaction or when heating the container.
- 2. Use care in making rubber-to-glass connections. Lengths of glass tubing should be supported while they are being inserted into rubber stoppers or tubing. The ends of the glass should be flame polished to smooth them out, and a lubricant, such as water or glycerin, should be used. <u>Never</u> use grease or oil. Gloves should be worn when making such connections. The glass tubing should be held as close to the end being inserted as possible to prevent breaking. Never try to force rubber stoppers or tubing from the glass. If necessary, cut the rubber.
- 3. Always check labels on bottles carefully to make sure that the proper chemical is being selected. Keep storage areas clean and organized. <u>Carefully</u> dispose of old or excess chemicals in accordance with accepted procedures. Acids and bases may be <u>slowly and carefully</u> poured down a sink drain <u>only if accompanied by a significant amount of water</u>. Toxic chemicals may not be disposed of in a sink, but must be sent to an approved disposal company. Separate flammable, explosive or special hazard items for storage in

an approved manner. All chemical containers should be clearly labeled, indicating contents and the date that the bottle was opened or the chemical prepared. All poisons must be labeled as such and have antidotes clearly marked.

- 4. Never handle chemicals with bare hands. Always wear rubber or surgical gloves and use a spoon or spatula for transferring dry chemicals and some type of stirring device (glass rod, magnetic stirrer, etc.) for mixing chemical solutions.
- 5. Be sure that the laboratory has adequate ventilation. Always work in a fume hood when using chemicals or samples that have toxic fumes. Even mild concentrations of fumes or gasses can be dangerous.
- 6. Never use laboratory glassware as a food container or a drinking cup.
- 7. When handling hot equipment, use tongs, insulated gloves or other suitable tools. Avoid using paper towels, rags, etc.
- 8. When working in the laboratory, avoid smoking and eating except in designated areas at prescribed times. Always wash hands **thoroughly** before smoking or eating.
- 9. Do not, under **any** circumstances, pipette by mouth. Always use some type of approved suction bulb or automatic pipette.
- 10. Handle all chemicals, reagents, and samples with care. Read and become familiar with all precautions or warnings on labels. Know and have all antidotes to poisons and poisonous chemicals readily available in the laboratory.
- 11. A short section of rubber hose on each water outlet provides an excellent way to wash away harmful chemicals from the eyes and skin.
- 12. Always add acid to water **unless the procedure specifically** requires the reverse. If water must be added to an acid or base, do so very slowly, stirring the solution as the water is added. Note that significant heat may build up during this process.
- 13. Wear a protective smock, apron, or lab coat, and surgical or rubber gloves when working in the laboratory to protect clothes and skin.
- 14. In the case of chemical spills or contact with skin or eyes, rinse **thoroughly** with **large** amounts of clean water. Notify your supervisor immediately and contact a physician. Special attention should be given if a caustic base is accidentally spilled into the eye.

- 15. Prevent fires by maintaining good housekeeping and keeping storage areas organized. Know how to use fire extinguishers and keep the recommended type handy. Contact local fire officials for fire safety tips and suggestions.
- 16. Keep an approved, well-stocked first-aid kit on hand and in a convenient location in the laboratory.

3. LABORATORY EQUIPMENT

The test procedures used in a laboratory will determine what types of equipment are required. Since there is a wide variety of equipment types available, this section will deal with only those pieces of equipment that are common to nearly all facilities.

Water and wastewater laboratory must have all the necessary equipment in order for an operator to perform the required tests, and also to prepare the necessary stock solutions and reagents, as well as to perform any required quality control procedures. This equipment must be maintained in proper working order, and in many cases must be calibrated on a routine basis.

The following sections will present a few examples of basic laboratory equipment and a brief description of their uses.

3.1. Measurement Equipment

<u>Analytical Balance</u>: An analytical balance is a highly accurate piece of equipment used for determining weights in "gravimetric" analyses and in weighing chemicals to prepare standard solutions. An analytical balance used for the total suspended solids test generally has a capacity of 160 grams and must be capable of weighing to an accuracy of 0.0001 grams.

Spectrophotometer: A visible light spectrophotometer is an instrument capable of producing light at a specific wavelength, and then measuring the amount of that light which passes through a colored liquid in tests referred to as "colorimetric" analyses. Spectrophotometers are used whenever the intensity of the color of a solution is needed to determine the concentration of a chemical. The visible-light spectrophotometer should have the ability to work in the 400 to 700-nanometer wavelength range.

<u>pH Meter</u>: A pH meter is an instrument which measures the intensity of the alkaline or acid strength of a solution. A pH measuring system consists of a pH meter and an electrode system, including a measuring probe and a reference probe (although these two probes are now often combined in a "combination" probe).

Dissolved Oxygen Meter (DO Meter): A dissolved oxygen meter is used to determine the dissolved oxygen content in a liquid solution based on the method called "polarography." This approach is now the method of choice for rapid, accurate dissolved oxygen measurements on most water, wastewater, and industrial waste samples, and generally provides an accuracy of +/-0.2 mg/L dissolved oxygen. A dissolved oxygen measuring system consists of a DO meter and a measuring probe.

3.2. Preparation Equipment

BOD Incubator: A specific requirement of the Biochemical Oxygen Demand (BOD) test is that the prepared sample bottles be incubated at 20° C +/- 1.0° C at zero illumination and 100% humidity. There are kits available to convert conventional refrigerators into BOD incubators as well as special incubators for BOD testing.

<u>Bench Top Incubators for Bacteriological Testing</u>: Testing for the presence of microorganisms in a water sample generally requires that a warm environment of a constant temperature be provided. Several different types of benchtop incubators are available for this purpose, including circulating and non-circulating water baths, and air or dry-block incubators. In general, circulating water incubators provide the most consistent temperatures and are recommended for fecal coliform testing at $44.5^{\circ}C + -0.2^{\circ}C$.

Hot Plates and Stirring Hot Plates: Many tests and reagent preparations require constant stirring and/or heating. Portable stirrers with or without heating are available. These generally stir using magnetic stir bars or stars, and heat using electric resistance heat.

Drying Oven: A drying oven is used for drying glassware and crucibles, and can be either a gravity convection type or a forced circulation type.

Muffle Furnace: A muffle furnace is a kiln type oven capable of maintaining extremely high temperatures for extended periods of time. Muffle furnaces are used for igniting organic solids in the determination of volatile solids. The furnace must reach a minimum of 600°C and have enough space to handle three or four evaporating dishes.

<u>Autoclave:</u> Used for sterilization of equipment prior bacteriological testing, an autoclave must be capable of developing and maintaining 15 psi at 121°C for at least 20 minutes.

3.3. Glassware and Plastics

<u>Pipettes:</u> Pipettes are specially calibrated glass tubes used for accurately transferring small volumes of solution (usually 50 mL or less) from one container to another. Pipettes are available in a variety of types and sizes for many different uses. The most common types of pipettes include volumetric and measuring pipettes.

Burettes: Burettes are long, graduated glass tubes that have a valve (called a stopcock) for use in measuring precise volumes of liquid. Burettes are used for titration, which is the method for dispensing exact amounts of one chemical solution into another to produce the desired chemical reaction.

Erlenmeyer Flasks: The primary uses for Erlenmeyer flasks are mixing chemicals and heating solutions. Although Erlenmeyer flasks contain volume markings, these volumes are estimates only and these flasks should **not** be used to measure volume. Because the sides of Erlenmeyer flasks are slanted and the mouth is narrow, mixing reagent liquids can be accomplished by swirling without fear of spilling the contents. In addition, the contents can be heated on a hot plate with minimal evaporation due to the narrow mouth.

Beakers: Beakers are straight-walled containers used for mixing chemicals and holding samples during testing. The flat bottom and straight sides make boiling or heating easy. When used for mixing, the straight sides make the use of some type of stirring equipment (such as a magnetic stirrer) necessary. As with the Erlenmeyer flask, beaker volume markings are only approximate. Beakers should <u>not</u> be used for measuring when precise volumes are required.





Graduated Cylinders: Graduated cylinders are glass or plastic tubes that are calibrated for measuring large volumes of liquids. Although not nearly as accurate as pipettes, graduated cylinders have an advantage in that they require much less technique to use and can generally be used for larger volumes.

<u>Volumetric Flasks</u>: Volumetric flasks are specially designed containers to measure large volumes very accurately. Like volumetric pipettes, volumetric flasks are designed to measure one volume only and are calibrated "to deliver" (so that any remaining liquid residue after emptying is ignored).



References

- 1. Standard Methods for the Examination of Water and Wastewater, APHA-AWWA-WEF, 18th Edition, 1992
- 2. Methods for Chemical Analysis of Water and Wastes, U.S. EPA-600/4-79-020, March 1979
- 3. Jenkins, D., Snoeyink, V.L., Ferguson, J.F., Leckie, J.O. (1980) Water Chemistry Laboratory Manual. Third Edition. John Wiley & sons, Inc. New York, NY.
- 4. Reynolds, T.D., and Richards, P.A. (1996) Unit Operations and Processes in Environmental Engineering. Second Edition. PWS Publishing Company. Boston, MA.

pН

(Hydrogen Ion Concentration)

Water is made up of both hydrogen ions (H^+) and hydroxyl ions (OH^-) . The usual chemical symbol of water is H₂O. Because water is made up of these two ions, any solution that contains water (called an aqueous solution) always has both hydrogen ions and hydroxyl ions.

 $H_2O \leftrightarrow H^+ + OH^{\text{-}}$

pH is a mathematical expression of the intensity of the acid or alkaline condition of a sample. The term "pH" is defined as the negative logarithm of the hydrogen ion concentration and is generally represented with a scale from 0 to 14 pH units. The pH scale is a means of showing which ion has the greater intensity.

$$pH = -\log [H^+]$$

 $[H^+][OH^-]=10^{-14} mol/l$

At a pH of 7.0, the intensities of hydrogen and hydroxyl ions are equal, and the solution is said to be pH neutral. When the pH is less than 7.0, the intensity of the hydrogen ions is greater than that of the hydroxyl ions and the solution is said to be acidic. When the pH is greater than 7.0, the hydroxyl ions have the greater intensity, and the solution is referred to as alkaline or basic.

High pH values in nature may be caused by algae, which removes CO_2 from water through photosynthesis.

Apparatus

- 1. pH-Meter with combined electrode
- 2. 250 ml beakers
- 3. Wash bottles
- 4. Magnetic stirrer with magnetic bar
- 5. Thermometer

Reagents

- 1. pH 7 and pH 9.2 buffer solutions
- 2. Distilled water
- 3. Standard Potassium Chloride Solution

Procedure

- 1. <u>Part A: Calibration of the pH-Meter</u>
- 2. Turn on the meter with combined electrode, which is immersed in standard potassium chloride solution.
- 3. Set the "Temp" knob at the temperature of buffer solution.
- 4. Remove the electrode from standard potassium chloride solution, rinse thoroughly with distilled water, dry with tissue and immerse in pH 7 buffer solution. Allow the reading to stabilize.
- 5. Turn "CALIB" knob until the needle points to 7, allow the reading to stabilize.
- 6. Remove the electrode from pH 7 buffer solution. Rinse thoroughly with distilled water, dry with tissue and immerse into pH 9.2 buffer solution, allow the reading to stabilize.
- 7. Turn "Temp" knob until the needle points to 9.2, allow the reading to stabilize.
- 8. Remove the electrode from the buffer solution. Rinse thoroughly with distilled water and dry with tissue and immerse into the standard potassium chloride solution.

Part B: pH Measurement of sample:

- 1. Pour a 100ml sample into a 250 ml beaker and mix it on a stirrer.
- 2. Remove the electrode from standard potassium chloride solution and rinse it with distilled water, dry it with soft tissue and immerse it in the sample.
- 3. The needle will show pH value when reading is stable, record the reading.
- 4. Remove the electrode from sample rinse with distilled water and immerse again in standard potassium chloride solution.

Questions

- 1. A pH of 5.5 would indicate that the sample is ______ *Acidic*
- 2. What is the acceptable range of pH values for wastewater to be treated by biological methods?

6.8 - 7.2

- 3. What type of sample must be used for pH measurements? *Grab sample*
- 4. What is the recommended holding time for pH samples? Samples should be analyzed immediately after collection. In no case should the pH analysis be done after 30 minutes holding time.
- 5. What is the approved method for pH measurement? *Electrometric (meter) method.*

Alkalinity

Alkalinity is the quantitative capacity of an aqueous solution to neutralize an acid. It is the measure of the water ability to resist changes in pH when an acid is added. Water alkalinity results from the presence of bicarbonate, carbonate, and hydroxide of elements such as calcium, magnesium, and sodium.

Alkalinity compounds originate from chemical compounds dissolved from rocks and soil. Also, from CO_2 from the atmosphere and microbial decomposition of organic matter:

$$CO_2 + H_2O \leftrightarrow H_2CO_3 \leftrightarrow H^+ + HCO_3^-$$

Carbonate and bicarbonate alkalinity complex some heavy metals and thus reduces their toxicity. High alkalinity water often has high pH and contains high levels of dissolved solids. High or low alkalinity has no ill effects on humans. The monitoring of alkalinity is very important for proper chemical treatment in water and wastewater treatment plants (e.g. coagulation, softening and corrosion control)

Apparatus

- 1. Burette
- 2. Erlenmeyer flask
- 3. Pipettes

Reagents

- 1. Distilled water.
- 2. 0.02 N sulfuric acid.

Procedure

- 1. Calibrate pH meter.
- 2. Pour 100 ml sample into a clean and dry 15 ml beaker using 100 ml graduated cylinder; turn on a magnetic stirrer and record initial temperature.
- 3. Set temperature compensation on the pH meter, rinse and dry pH electrodes, immerse in sample and record initial pH.
- 4. If pH is greater than 8.3 add 0.02N H₂SO₄ until pH reaches 8.3 "endpoint" and record volume of titrate used; this is the phenolphthalein endpoint.
- 5. Titrate to pH 4.5 without undue delay; as the endpoint is approached make smaller additions of acid and be sure equilibrium is reached before adding more titrate.
- 6. Record volume of $0.02N H_2SO_4$ titrate used; this is the total or methyl orange endpoint.

Calculations

Total Alkalinity (mg/l as CaCO₃) = $\frac{A \times N \times 50,000}{ml \text{ sample}}$

Where:

A: ml of standard acid used. N: normality of standard acid.

Questions

- 1. Define Alkalinity and comment on the problems and/or benefits of Alkalinity in water treatment and use.
- 2. What are the chemical ions that contribute to Alkalinity?
- 3. Why does the pH change on aerating the water?

Turbidity

Turbidity is a characteristic of water that makes it appear cloudy. It is caused by colloidal materials (e.g. clay, silt, microorganisms, fibers, oil, soaps and metal oxides) cause it.

Principle

Turbidity is measured using a Turbidity meter that measures the intensity of light scattered at right angles to the incident of light. The unit of turbidity measurement is Nephelometer Turbidity Units (NTU).

Apparatus

1. meter with NTU formalized-standard (25mm cells)

Procedure

- 1. Warm up the Turbidity meter, select the range of 20 NTU.
- 2. Place a standard cell of 18.8 NTU in the cell holder with the index mark on the cell aligned with the raised mark on the spill ring. Cover it with the light shield.
- 3. If the display shows a reading in the range (17.86 to 19.74), the calibration is acceptable.
- 4. Select the range of 200 NTU.
- 5. Place a standard cell of 174 NTU in the cell holder and cover with the light shield.
- 6. Acceptable calibration will give a reading in the range (165.3 to 182.7).
- 7. Fill a clean, wiped sample cell to the ring mark with the sample.
- 8. Select meter range closer to the expected turbidity in sample and measure turbidity in a similar done to the standard.
- 9. Record turbidity reading in (NTU)

Conductivity

Conductivity is the ability of water to carry an electric current. It is a function of the concentration of ions. Increasing the ions level will increase the conductivity. It can be used as a rough measure of the concentration of the total dissolved salts and TDS. Conductivity unit of measurement is Siemens/m (S/m). High quality deionized water has a conductivity of about 5.5 μ S/m, typical drinking water in the range of 5-50 mS/m, while sea water about 5 S/m.

Apparatus

1. Conductivity meter

Reagents

- 1. Standard KCl solution (known conductivity)
- 2. Distilled water

Procedure

- 1. Switch on the conductivity meter.
- 2. Immerse the probe into the test solution, ensuring that the electrodes are entirely covered and that no air bubbles are trapped in the electrode compartment.
- 3. Switch the range selector to 0.1 µSiemens range.
- 4. Determine sample conductivity.

Data Sheet

Sample	pН	Total Alkalinity		Conductivity	Turbidity
		Volume of H ₂ SO ₄ (ml)	Alkalinity (mg/l as CaCO ₃)	(µS/m)	(NTU)

Questions

1. Discuss the importance of conductivity and its relationship to dissolved solids in water.

Hardness

(EDTA Titrimetric Method)

Hardness is the total concentration of calcium and magnesium ions expressed as calcium carbonate. Water hardness prevents the lathering of soap and produces scale in hot water pipes. Water is classified according to its hardness as follows:

Class	concentration
Soft	\leq 50 mg/l
Moderately soft	50-150 mg/l
Hard	150 - 300 mg/l
Very hard	\geq 300 mg/l

There are two types of hardness:

- 1. Carbonate hardness (temporary hardness), caused by the presence of carbonate and bicarbonates of calcium and magnesium.
- 2. Non- Carbonate hardness (Permanent hardness), caused by the presence of chlorides, sulfates, and nitrates of calcium and magnesium.

Principle

A small amount of Eriochrome Black T dye is added to a sample containing calcium and magnesium ions at a pH of 10.0 - 0.1, which turns the solution to wine red. Ethylenediamine tetra acidic acid (EDTA) is added as the titrant, forming a chelated soluble complex with the calcium and magnesium ions. When all these ions have reacted, the solution turns from wine red to blue.

Apparatus

- 1. Burette
- 2. Pipette
- 3. Erlenmeyer flask
- 4. Bottle

Reagents

- 1. Standard EDTA titrant (0.01 M)
- 2. Eriochrome black T indicator
- 3. Ammonia buffers solution

Procedure

- 1. Dilute 25 ml sample (V) to about 50 ml with distilled water in an Erlenmeyer flask.
- 2. Add two full eyedroppers of buffer solution
- 3. Add a spoonful of an indicator. The solution turns wine red in color.
- 4. Add standard EDTA titrant slowly with continuous stirring until the last reddish tinge disappears; do not extend the duration of titration beyond 5 minutes after adding the buffer solution. The color of the solution at the end point is blue under normal conditions.
- 5. Note down the volume of EDTA added (A).

Calculations

Hardnass mall as CoCOs -	$A \!\times\! B \!\times\! 1000$
Hardness, mg/l as $CaCO_3 =$	V

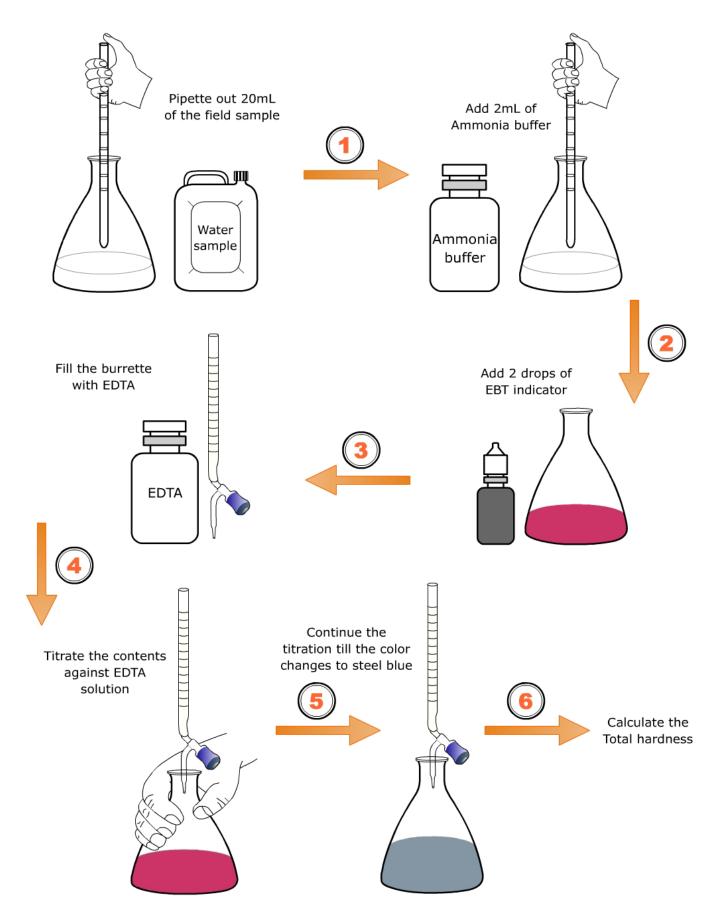
Where:	А	=	Volume of EDTA titrant used (ml)
	В	=	mg CaCO ₃ equivalent to 1.00 ml EDTA titrant
		=	1 mg CaCO ₃
	V	=	Volume of sample tested (ml)

Sampla	Volume of sample	Volume of EDTA	Hardness
Sample	(ml)	(ml)	(mg/l as CaCO ₃)

Questions

- 1. When determining total hardness, we measure the sum of hardness causing ions; what are the most important ions that produce harness in water?
- 2. How is hardness classified?
- 3. Explain the significance of determination of hardness of water in environmental engineering?
- 4. How can you remove permanent hardness from water?
- 5. Can you determine temporary hardness and permanent hardness separately? If yes, how?

Procedure Chart for Total Hardness



Chlorides

Chlorides occur widely in water and wastewater and are usually associated with Na ion. Although chlorides are not harmful, concentration beyond 250 mg/l imparts a peculiar taste to water rendering it unacceptable from the aesthetic point of view for drinking purpose.

Principle

If water containing chloride is titrated with silver nitrate solution, chlorides are precipitated as white silver chloride. Potassium chromate is used as an indicator, which supplies chromate ions. As the concentration of chloride ions approaches extinction, silver ion concentration increases to a level at which reddish-brown precipitate of silver chromate is formed indicating the end point.

Apparatus

- 1. Erlenmeyer flasks, 250 ml
- 2. Burette
- 3. Measuring cylinder

Reagents

- 1. Distilled water
- 2. Standard mercuric nitrate titrant (0.0141 N)
- 3. Diphenylcarbazone indicator.
- 4. Acid or alkali for adjusting pH.

Procedure

- 1. Measure 50 ml sample with a graduated cylinder and pour into 250 ml Erlenmeyer flask.
- 2. If the sample is colored, add 3mL of aluminum hydroxide, shake well; allow to settle, filter, wash and collect filtrate.
- 3. The sample is brought to pH 7–8 by adding acid or alkali as required.
- 4. Add 1 mL of an indicator; the color of the solution should be green-blue.
- 5. Titrate the solution against standard mercuric nitrate solution until a purple endpoint. Note down the volume (A).
- 6. Repeat the procedure for blank and note down the volume (B).

Calculations

Chloride concentration, Cl⁻ mg/l =
$$\frac{(A - B) \times N \times 35,450}{\text{sample volume (ml)}}$$

Where:

A = ml titrant for sample

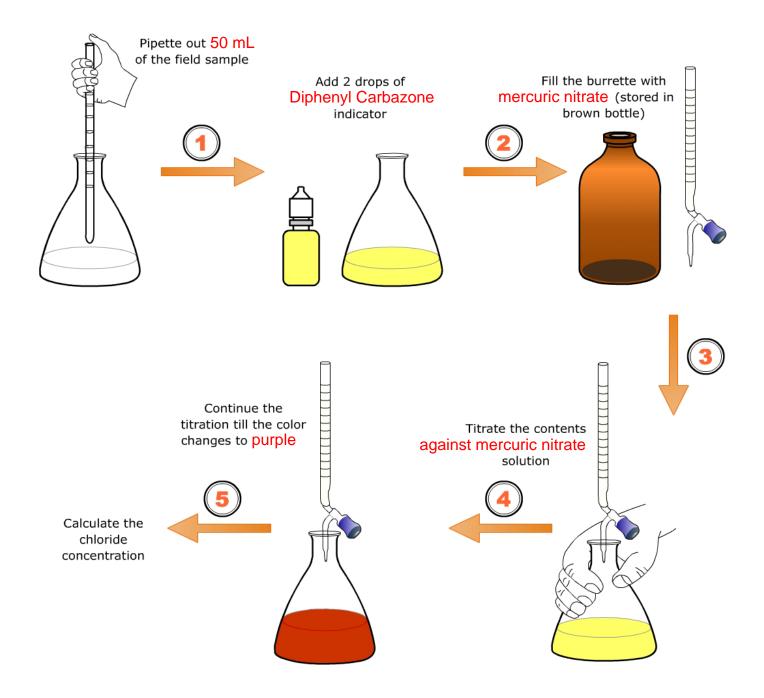
B = ml titrant for blank

 $N = Normality of AgNO_3$

Sample	Volume of sample (ml)	Volume of titrant (ml)	Chloride (mg/l)
Blank			
Blank			

Question

- 1. Explain the significance of high chloride in water.
- 2. Why has the water lower content of salt than sewage?



Sulfates (Turbidimetric Method)

Sulfate ions (SO₄²⁻) usually occur in natural waters. Sodium and magnesium sulfate exert cathartic action and hence its concentration above 250 mg/l in potable water is objectionable. Sulfate causes a problem of scaling in industrial water supplies, and the problem of odor and corrosion in wastewater treatment due to its reduction to H_2S .

Principle

Sulfate ions are precipitated in a hydrochloric acid (HCl) medium with barium chloride (BaCl₂), to form a uniform suspension of BaSO₄ crystals. The absorbance of the suspension is measured by a spectrophotometer.

Apparatus

- 1. Magnetic stirrer
- 2. Spectrophotometer for use at 420 nm wavelength
- 3. Erlenmeyer flasks, 250 ml
- 4. Stopwatch

Reagents

- 1. Conditioning reagent
- 2. Barium Chloride crystals, BaCl₂
- 3. Standard sulfate solutions

Procedure

- 1. Measure 100 ml sample or a suitable portion and then made up to 100 ml, pours into a 250 ml Erlenmeyer flask.
- 2. Add 5 ml conditioning reagent and mix in stirring apparatus.
- 3. While stirring, add a spoonful of BaCl₂ crystals and begin timing immediately. Stir for 1 min at a constant speed.
- 4. Measure the turbidity developed, as transmittance, after every 30 seconds for 4 minutes on a photometer at 420 nm. (After 2 minutes, reading will remain constant)
- 5. Prepare a standard curve by carrying sulfate standard solution through an entire procedure. Space standards at 10 mg/l increment in the 0 to 50 mg/l range.
- 6. Read mg SO_4^{2-} present in the sample from the standard curve

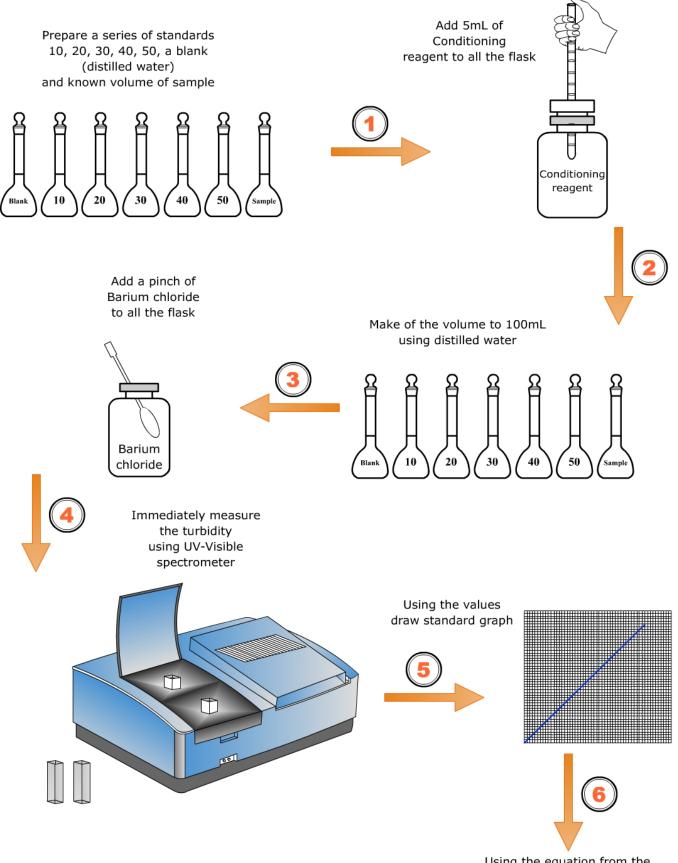
Department of Civil Engineering College of Engineering King Saud University

Calculations

Sulfate concentration,
$$SO_4^{2-}$$
 mg/l = $\frac{\text{mg } SO_4^{2-} \times 100}{\text{sample volume (ml)}}$

Sample	Sample Volume (ml)	Transmittance (T %)	mg SO_4^{2-} from standard curve	Sulfate (mg/l)

Procedure Chart for Sulfates



Using the equation from the graph calculate the concentration of sulphate

Total Solids (TS)

The term "solids" is generally used when referring to any material suspended or dissolved in water and wastewater that can be physically isolated either through filtration or through evaporation.

Solids can be classified as either filterable or non-filterable. Non-filterable solids may be either settleable or non-settleable. Solids can also be classified as organic or inorganic. "Filterable" solids are so small that they will pass through a standard laboratory filter, while "non-filterable" solids are large enough to be captured on a standard filter pad. The non-filterable solids are termed "settleable" if the solids settle out in a standard laboratory-settling container within a specified period. They are called "non-settleable" if they fail to settle out within that time limit. If solids are "organic," the material is carbon-based and will burn. "Inorganic" solids, on the other hand, are mineral based and generally will not burn. Any material that was at one time living (for example, body wastes, starches, sugars, wood, bacteria, and cotton) are all organic while limestone, iron, and calcium are inorganic.

Solids in wastewater are frequently used to describe the strength of the waste. The more solids present in a particular wastewater, the stronger that wastewater will be. If the solids in wastewater are mostly organic, the impact on a treatment plant is greater than if the solids are mostly inorganic.

Generally, large quantities of organic solids will create more pollution problems than will the same amount of inorganic solids. Therefore, not only is it important to know how much solids are present in the waste, but also the type of solids that are present. The test procedures for solids provide essential information about the level and type of solids coming into the treatment plant and whether the solids are actually being removed in the plant processes.

Principle

This experiment is done to determine the total amount of residual matter (suspended and dissolved) in a given water sample. Solids left in a porcelain dish after evaporation of a measured volume of a water sample at boiling temperature: 103 - 105 °C.

Apparatus

- 1. Porcelain evaporating dishes
- 2. Drying oven (103 105 °C)
- 3. Desiccator
- 4. Analytical balance capable of weighing to 0.1 mg

Procedure

- 1. Ignite clean evaporating dish at 550 ± 50 °C for 1 hour in a muffle furnace.
- 2. Cool dish in air for 2 min, desiccate until cool and weigh (B).
- 3. Transfer a measured volume of well-mixed sample to the pre-weighed dish.
- 4. Evaporate sample to dryness in an oven at 103-105 °C overnight.
- 5. Cool dish in desiccators and weight (A).

Data Analysis

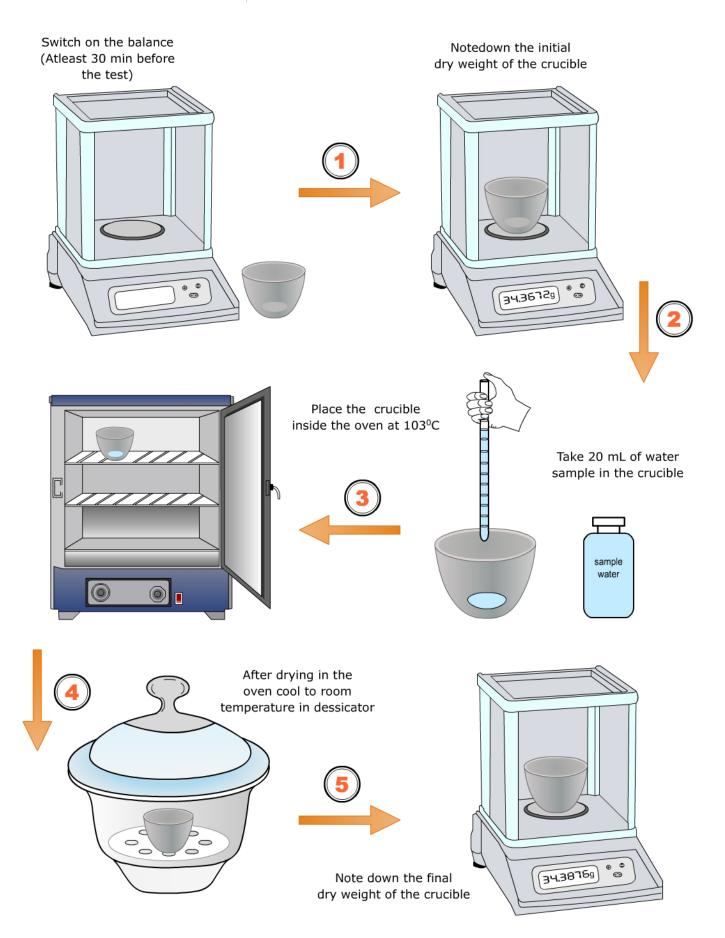
Total Solids (mg/l) =
$$\frac{(A - B) \times 10^6}{\text{sample volume (ml)}}$$

Where:

A = Weight of dried residue + dish (g)B = Weight of dish (g)

	Total Solids		
Sample	(B) Weight of dish (g)	(A) Weight of dried residue + dish (g)	TS (mg/l)

Procedure Chart for Total Solids



Total Dissolved Solids (TDS)

Principle

Total dissolved solids (TDS), also called filterable residue, is material that passes through a standard glass-fiber filter and remains after evaporation and drying to constant weight at 103 - 105 °C. TDS can also be measured in relation with conductivity.

Apparatus

- 1. Glass fiber filters (0.45 μ m)
- 2. Gooch crucible
- 3. Suction flask
- 4. Porcelain evaporating dishes
- 5. Drying oven (103 105 °C)
- 6. Desiccator
- 7. Analytical balance capable of weighing to 0.1 mg

Procedure

- 1. Place filter on Gooch crucible. Apply vacuum and wash the filter with three successive 20 ml volumes of distilled water. Continue suction to remove all traces of water and discard washings.
- 2. Ignite clean evaporation dish at 550 ± 50 °C for 1 hour in a muffle furnace. Cool and store in desiccators until needed. Weight immediately before use (B).
- 3. Filter a measured volume of well-mixed sample though the pre-washed filter, wash with three successive 10 ml volumes of distilled water, and continue suction for about 3 minutes after filtration is complete.
- 4. Transfer filtrate to the pre-weighed dish and evaporate to dryness at 103-105 °C overnight.
- 5. Cool dish in desiccators and weight (A).

Data Analysis

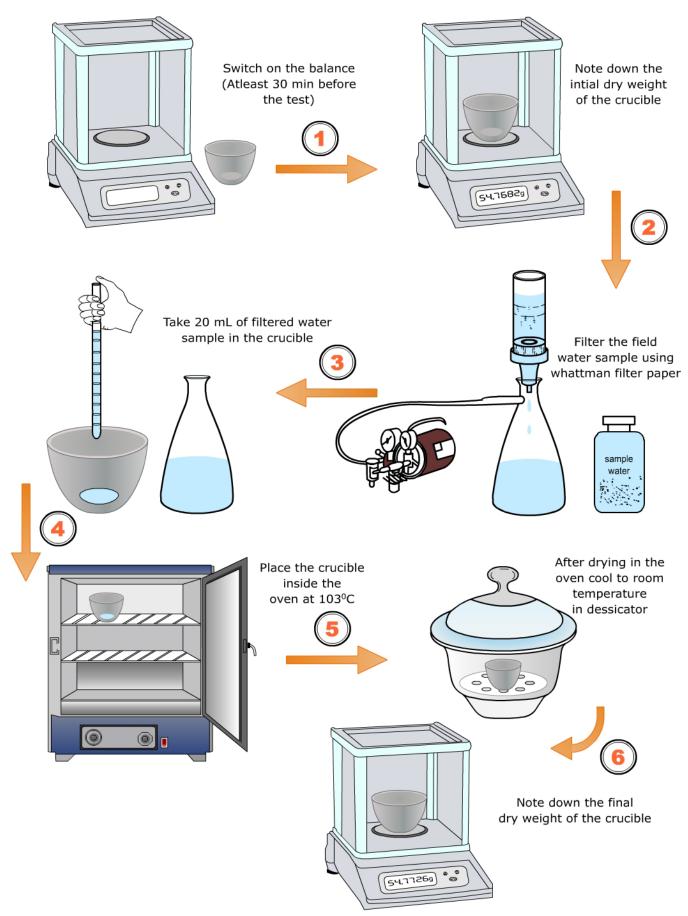
Total Dissolved Solids (mg/l) = $\frac{(A - B) \times 10^6}{\text{sample volume (ml)}}$

Where:

A = Weight of dried residue + dish (g) B = Weight of dish (g)

	Total Dissolved Solids			
Sample	(B) Weight of dish (g)	(A) Weight of dried residue + dish (g)	TDS (mg/l)	

Procedure Chart for TDS



Total Suspended Solids (TSS)

Suspended solids, also called a non-filterable residue, are classified depending on the size of particles that are larger than 0.001 micrometers (μ m). Colloidal (non-settleable) solids range from 0.001 to 100 μ m, solids are greater than 100 μ m (settleable solids). Suspended solids can be organic or non-organic.

Principle

Total Suspended Solids is determined by filtering a measured volume of well-mixed sample through a glass-fiber filter, after filtration, the filter pad is dried and weighted.

Apparatus

- 1. Glass fiber filters (0.45 μ m)
- 2. Gooch crucible
- 3. Suction flask
- 4. Porcelain evaporating dishes
- 5. Drying oven (103 105 °C)
- 6. Desiccator
- 7. Analytical balance capable of weighing to 0.1 mg

Procedure

- 1. Place filter on Gooch's crucible. Apply vacuum and wash the filter with three successive 20 ml volumes of distilled water. Continue suction to remove all traces of water and discard washings.
- 2. Remove crucible/filter combination and dry in an oven at 103 105 °C for 1 hour. Cool in desiccators and weight immediately before use (B).
- 3. Filter a measured volume of well-mixed sample through pre-weighed crucible/filter. Wash filter with three successive 10 ml volumes of distilled water.
- 4. Carefully remove crucible/filter combination, dry for 1 hour (to constant weight) at 103 105 ° C, cool in a desiccator, and weigh (A).

Data Analysis

Total Suspended Solids (mg/l) =
$$\frac{(A - B) \times 10^6}{\text{sample volume (ml)}}$$

Where:

- A = Weight of dried residue + crucible/filter combination (g)
- B = Weight of crucible/filter combination (g)

	-	Fotal Suspended Solids	
Sample	(B) Weight of crucible/filter (g)	(A) Weight of residue + crucible/filter (g)	TSS (mg/l)

Questions

- 1. What three types of solids make up the suspended solids in wastewater? *Floatable, Settleable and Colloidal.*
- 2. Approximately what percent of wastewater is made up of solids? 0.1% - one-tenth of one percent.
- 3. Why must solids samples be preserved by refrigeration if they cannot be analyzed immediately?

The biological activity could continue in the sample producing changes in solids characteristics and amounts.

4. What can cause losses or gains in weight of solids during the drying process of the various solids tests? Losses: volatilization of organic material, entrapped water, water of hydration and gasses formed by chemical reactions during heating;

Grains: oxidation during heating.

Coagulation & Flocculation

Coagulation and flocculation refer to the addition of chemicals (coagulants) to the water to promote the formation and growth of rapidly settling flocs. During coagulation – a period of relatively intense mixing – the added chemicals destabilize the colloidal particles. During flocculation, slow mixing promotes the aggregation of particulate matter and the formation of larger flocs that can be removed from the water by sedimentation or filtration. These processes are used in water treatment to enhance the removal of non-settleable colloidal solids and slow-settling particulates. Chemical precipitation is a closely related process that promotes the formation of settleable solids from unwanted excess ions.

Principle

The jar test is a laboratory technique widely used to evaluate the chemical dosages necessary for a proper coagulation/flocculation process. This test involves an examination of the effects of coagulant addition, mixing, and settling on water quality parameters such as turbidity, color, total organic carbon, pH and alkalinity.

The purpose of this lab is to use jar tests to:

- 1. Determine the optimum coagulant dosage (alum) for the removal of turbidity from a water sample.
- 2. Determine the dosage of lime required for the removal of hardness from a water sample.

Apparatus

- 1. Jar test apparatus.
- 2. Analytical Balance.
- 3. pH meter.
- 4. Turbidimeter and sample vials.
- 5. HACH hardness titration kits.
- 6. Filtration apparatus and paper filters
- 7. Small beakers (50 ml)

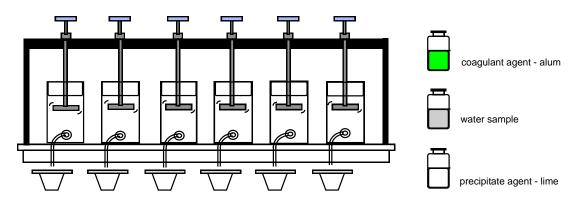
Reagents & Material

- 1. Alum [Al₂(SO₄)₃].
- 2. Slaked Lime, Ca(OH)₂ [Mix 50 g CaO in 1 liter].
- 3. $0.02 \text{ N} \text{ H}_2 \text{SO}_4$ solution and burette for alkalinity measurement
- 4. Soda Ash (Na₂CO₃) 0.5 M solution
- 5. Water samples (10 liters) collected from different sources (e.g., Well water, water treatment plant, lab), will be available at the beginning of the laboratory.

Procedure

Coagulation/Flocculation:

- 1. Measure the pH and turbidity of the original water sample. Make sure that the instruments are calibrated before using them (ask the instructor or the TA for help in the calibration).
- 2. Jar test setup: Add equal volumes (1000 ml) of the test water to each of the six square beakers and place them in the jar test apparatus.
- 3. Measure the initial pH and turbidity in each square beaker. Use the sampling ports on the side of each beaker to take 40-ml samples. For each measurement, pour the sample into a sampling vial and measure pH; then close the vial and measure turbidity.
- 4. Adjust the pH of the water by adding 10 mL of Na₂CO₃ solution to each beaker and thoroughly mixing. Measure pH and turbidity again after the pH adjustment step.
- Measure out five coagulant doses (alum) into small weighing boats as follows: <u>Group 1</u>: 25, 50, 75, 100, and 200 mg <u>Group 2</u>: 50, 100, 200, 400, and 700 mg
- 6. At the start of a 1-min rapid mix period (80 rpm), simultaneously add the coagulant dosages to five of the square beakers. The sixth beaker will be a control.
- 7. After the rapid mix, flocculate the solutions by mixing at 10 rpm for 20 minutes. Record any variations in the characteristics of the flocs during this time. Note the size and appearance of flocs.



Jar Test Apparatus

- 8. After flocculation, turn off the stirrers and allow the flocs to settle for 30 minutes. Observe differences in the type of settling among samples. Compare the settling characteristics of the different samples with the settling in the control sample.
- 9. At the end of the settling period, carefully take samples from the beakers, making sure not to disturb the sediment while sampling. Measure final turbidity and pH. Make sure to clean the sampling port before taking the final sample (withdraw and discard 20 ml before sampling).

- 10. Measure the depth of the sludge formed with each coagulant dose.
- 11. For comparison with the lime softening experiment, measure the alkalinity and the hardness of the original sample (see lime softening instructions).

Lime softening:

- 1. Measure the pH and turbidity of the initial water sample.
- 2. Measure initial alkalinity of the sample. Use the $0.02N H_2SO_4$ solution to titrate a 50 ml sample to a final pH of 4.5. Total alkalinity is equal to:

Total Alkalinity (mg/l as CaCO₃) = $\frac{\text{ml acid} \times \text{N} \times 50,000}{\text{ml sample}}$

- 3. Measure total and calcium hardness of the sample with the HACH hardness titration kits. Note, use only one type of titration kit.
- 4. Jar test setup: Add equal volumes (1000 ml) of water to each of the six square beakers and place them in the jar test apparatus.
- 5. At the start of a 1 min rapid mix period (150 rpm), add the following lime dosages [Ca(OH)₂ solution] to each of the square beakers:
 <u>Group 3</u>: 0 ml, 2 ml, 4 ml, 6 ml, 8 ml, and 10 ml
 <u>Group 4</u>: 0 ml, 1 ml, 3 ml, 5 ml, 7 ml, and 9 ml (the first jar is a control).
- 6. After the rapid mix, flocculate the solutions by mixing at 30 rpm for 10 minutes. Record the elapsed time before visible flocs are formed. Note the size and appearance of flocs.
- During the flocculation period, measure the pH of each sample. After flocculation, turn off the stirrers and allow the flocs to settle for 10 minutes. <u>Observe</u> the settling characteristics in the different jars.
- 8. At the end of the settling period, carefully take samples from the beakers, making sure not to disturb the sediment while sampling. Filter the samples and measure the final hardness of the filtered samples.

If time allows...

- 9. At the beginning of the second period of a rapid mix (1 min), add 2 ml of 0.5M Na₂CO₃ to each square beaker.
- 10. Reduce the mixing speed to 30 rpm and allow the samples to flocculate for 10 min. During this time, measure the new pH of the samples.
- 11. Turn off the mixers and allow 10 min of settling before measuring the final hardness of filtered samples.

Calculations

Coagulation/Flocculation:

- 1. Make a plot of final turbidity vs. Coagulant dosage.
- 2. Determine the best dosage of coagulant.
- 3. Determine the volume of sludge that would be produced by treating one million liters of this water with the coagulant dose selected.

Lime Softening:

- 1. Make plots of a) hardness removal vs. Lime dosage, with and without the addition of soda ash, and b) hardness removal vs. final pH.
- 2. Determine the best experimental dosage of lime for the removal of hardness.
- 3. Calculate the alkalinity of the original sample.
- 4. Calculate calcium concentration, magnesium concentration, carbonate hardness, noncarbonate hardness, calcium carbonate hardness, calcium non-carbonate hardness, magnesium carbonate hardness, and magnesium non-carbonate hardness (all in meq/liter).
- 5. Determine the theoretical dose of lime and soda ash required for the excess lime-soda ash process.
- 6. How do the theoretical doses compare with the best dose experimentally determined?

Questions

- 1. Compare initial pH, turbidity, alkalinity, and hardness measurements among the different water samples. Discuss similarities and differences.
- 2. In the coagulation/flocculation experiment, what was the relationship between pH variation and coagulant dose? Why?
- 3. Describe the coagulation/flocculation experiments in terms of the different types of settling observed for the various coagulant doses.
- 4. In the lime softening experiment, what is the relationship between pH and lime dose? Why? How does pH affect hardness removal?

Total and Fecal Coliform

(Membrane Filter Technique)

The test for coliform bacteria is used to measure the suitability of water for human use. The bacteria detected by this test are normally found in the intestinal tract of humans and other mammals. These bacteria are present in sewage, numbering as many as 1,000,000 per milliliter. Coliform bacteria are considered harmless. However, their presence does indicate the possible presence of other pathogenic organisms. As a consequence, when coliform bacteria are found, the water is suspected of being polluted by human waste discharge.

Depending on the use of the water, standards are established for the numbers of coliform bacteria permissible in a given volume of that water. For example, a safe bathing water standard would not be as strict as a safe drinking water standard. The test is not only useful in determining the bacterial quality of a finished water, but also can be used by the operator in the treatment plant as a guide in achieving a desired degree of treatment.

Principle

The membrane filter technique utilizes a specially designed filter pad with uniformly sized pores (openings). The pores of the membrane filter are small enough to prevent bacteria from passing through the filter. In fact, the bacteria cannot travel into the filter at all, but must remain on the filter's surface. Another unique characteristic of the filter allows liquids placed under the filter to pass upward through the filter. This feature lets media placed under the filter provide nourishment for bacterial growth.

Apparatus

- 1. Autoclave
- 2. Filtration units
- 3. Incubators (44.5 \pm 0.2 °C)

Reagents & Material

- 1. Sterile buffered distilled water
- 2. m-Endo agar medium, for total coliform
- 3. m-FC agar medium, for fecal coliform
- 4. Membrane filter paper (0.45 μm pore size)
- 5. Culture dishes (petri dish)
- 6. Absorbent pads.
- 7. Forceps, stainless steel.

Procedure

All items should be adequately sterilized by autoclave.

Sample Filtration

- 1. Using sterile forceps, place a sterile membrane filter on the filter support assembly.
- 2. Place the funnel portion of the assembly over the filter, making sure the filter is properly aligned during this step.
- 3. Clamp or lock the assembly in place.
- 4. Mix the sample (or sample dilution) thoroughly by shaking at least 3 times.
- 5. Pour the undiluted sample into the funnel assembly to the 100 mL mark <u>OR</u> pour 100 mL of a serially diluted sample into the funnel assembly.

NOTE: The sample size and/or the required serial dilution should be selected to grow 20-60 fecal coliform colonies after incubation.

- 6. Apply vacuum and filter the entire volume of sample or dilution through the membrane filter.
- 7. Rinse the funnel assembly and membrane filter with three 20-30 mL portions of sterile buffered dilution water. (Allow the entire volume of each portion to pass through the filter before adding the next portion.)
- 8. Carefully remove the funnel assembly and immediately remove the membrane filter, using sterile forceps.

NOTE: Filtration units should be sterile at the start of each filtration series and should be sterilized again if the series is interrupted for 30 minutes or more. A rapid interim sterilization can be accomplished by 2 minutes exposure to ultraviolet light, flowing steam or boiling water.

Incubation

- 1. Using sterile forceps, carefully place a sterile absorbent pad in the bottom portion of a sterile culture dish.
- 2. Transfer 2.0 mL of a medium with a sterile pipette onto the pad (use m-Endo agar medium for total coliform and m-FC broth for fecal coliform).
- 3. Drain off any medium not absorbed by the pad.
- 4. Using sterile forceps, carefully place the sample filter on the absorbent pad using a rolling motion to avoid catching air bubbles under the filter.
- 5. Cover the culture dish and mark the top of the cover to identify the sample.
- 6. Seal the culture dish in a plastic bag or by using electrical tape and place in a water bath incubator at 44.5° (+/-0.2°) C.

NOTE: All the prepared culture dishes should be placed in the water bath within 30 minutes after filtration. The plastic bags must be anchored or weighted to ensure the culture dishes are kept completely submerged during the entire incubation period.

7. Incubate the culture dishes for 24 (+/-2) hours.

- 8. At the end of the incubation period, remove the culture dishes from the water bath and count the colonies.
 - Pink to dark red colored with a metallic surface for total coliform.
 - Blue-colored colonies on the surface of the filter for fecal coliform (The fecal coliform colonies will appear blue, while non-fecal coliform colonies will appear gray or cream colored).

The desired range of colonies for the most accurate fecal coliform determination is 20 to 60 colonies per filter.

NOTE: Filters which show growth over the entire surface of the filter with no individually identifiable colonies should be recorded as TNTC (too numerous to count).

Calculations

Calculation of Colonies/100 mL

The fecal coliform density can be calculated using the following guidelines:

1. For samples with one or more volumes with colony counts in the range of 20 to 60 colonies, the correct average calculation is as follows:

Arithmetically averages only the samples with counts in the acceptable (20 to 60) range.

Example:	Volume	Colony count
	50 mL	59
	25 mL	30
	20 mL	18

Calculate the colony count per 100 mL for each sample in the acceptable range using the following formula:

colonies/100 mL = (100 mL x colony count)/volume used.

for 50 mL = $(100 \times 59)/50 = 118 \text{ colonies}/100 \text{ mL}$ for 25 mL = $(100 \times 30)/25 = 120 \text{ colonies}/100 \text{ mL}$ (Reject 20 mL sample since the count is less than 20.) Average the results arithmetically. $(118 + 120)/2 = \underline{119 \text{ colonies}/100 \text{ mL}}$ 2. For samples with colony counts for all volumes less than 20 and greater than zero, the correct average calculation is as follows:

Select the most acceptable count (usually the largest volume used) to avoid additional variability due to low counts. Calculate the colony count per 100 mL for that sample.

Example:	Volume	Colony count
	50 mL	19
	25 mL	10
	10 mL	4

For 50 mL = $(100 \times 19)/50 = \frac{38 \text{ colonies}}{100 \text{ mL estimated.}}$

3. For samples with all colonies counts greater than 60, but still countable (have not grown together into a mass of poorly defined colonies), the correct average calculation is as follows:

Select the count from the smallest volume filtered and calculate the colony count per 100 mL for that sample. (If the colonies have all grown together, report as TNTC.)

Example:	Volume	Colony count
	50 mL	199
	25 mL	110
	10 mL	65

for 10 mL = $(100 \times 65)/10 = \text{greater than } 650 \text{ colonies}/100 \text{ mL}$

The result is reported as "greater than" because all counts were greater than 60. Greater than values are to be avoided by analyzing multiple dilutions.



Total Coliform



Fecal Coliform

Questions

1. Why are fecal coliform bacteria referred to as indicator organisms? Fecal coliform bacteria are called indicator organisms because they originate in the same place as disease-causing bacteria. Their presence or absence is an indicator of the presence or absence of a pathogenic organism.

What type of water should be used for media and dilution water preparation for the fecal coliform tests?

Use distilled or deionized water.

- What is the acceptable method for sterilizing the dilution water utilized in the fecal coliform procedures? *Autoclave*.
- What reagent should be present in sample bottles used to collect chlorinated effluent samples? Why?
 0.1 mL of 10% sodium thiosulfate for dechlorination.
- 4. How can a fecal coliform samples be preserved? *Refrigeration to less than 10°C*.
- 5. What is the maximum allowable holding time for wastewater fecal coliform samples? *6 hours from the time of sample collection.*
- 6. How should the results in a colony count be recorded when the colonies have all grown together?
 TNTC = <u>Too Numerous To Count</u>

Chlorine Residual

(DPD Colorimetric Method)

The main purpose for the chlorination of water supplies and wastewater treatment plant effluents is the destruction of disease-causing microorganisms. Chlorine has a variety of other uses in the wastewater treatment plant operation that include: odor control and flies and ponding control (in trickling filters).

Unfortunately, chlorine can cause problems if its use is not controlled very carefully. When used as a control on the disease-causing bacteria, the idea is to disinfect and not sterilize the effluent. Disinfection is the process of killing disease-causing bacteria. Sterilization is the process of killing all living organisms. If an attempt were made to sterilize the effluent, the biological life in the receiving waters would likely be destroyed.

The chlorine residual test is used to determine the total amount of chlorine present as a residual (the amount of chlorine present after the demand has been satisfied). Because the residual determines how effective the disinfection process is, it is important to make sure that the residual remains within a specified range. Too little chlorine will not give adequate disinfection, and too much can kill the aquatic life in the receiving waters.

Principle

In this method, the chlorine residual is determined using a spectrophotometer. DPD is used as the indicator. In the presence of chlorine, the DPD indicator solution has a red color. The more chlorine that is present, the darker the red color will be. The intensity of the color is measured against known values from a standard curve. Potassium permanganate is used as the standard for establishing the standard curve because of the difficulty in getting accurate chlorine standards and the ease of handling potassium permanganate.

Apparatus

1. Spectrophotometer (with wavelength of 515 nm and a light path of 1 cm or longer)

Reagents & Material

- 1. Phosphate buffer solution
- 2. DPD indicator solution (caution: this chemical is poisonous)
- 3. Potassium iodide (KI), crystals
- 4. Standard potassium permanganate solutions
- 5. 250 mL Erlenmeyer flask

- 6. 15 mL test tubes
- 7. 10 mL measuring pipette
- 8. 1 mL pipettes, graduated to 0.1 mL

Procedure

- 1. Prepare a series of potassium permanganate standards covering the equivalent chlorine range of 0.1 to 4 mg/L (0.1, 0.2, 0.5, 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, and 4.0 mg/L).
- 2. For each standard develop color and measure %T at 515 nm as follows:
 - a. Set 100%T on a spectrophotometer using a distilled water blank, in accordance with manufacturer's instructions. (Prepare a distilled water blank in the same manner as the sample for testing).
 - b. Place 5 mL of phosphate buffer and 5 mL of DPD indicator reagent in each standard flask.
 - c. Add 100 mL of prepared standards and mix thoroughly.
 - d. Fill colorimeter cell from the flask and read each standard at 515 nm wavelength.
- 3. Plot a standard curve of mg/L equivalent chlorine versus %T.
- 4. Pipette 0.5 mL phosphate buffer solution into a test tube of appropriate volume (Test tubes should have a capacity of at least 15 mL, and have covered cap)
- 5. Add 0.5 mL DPD indicator solution by pipette.
- 6. Add a few crystals (approximately 0.1 g) of potassium iodide (KI).
- 7. Add 10 mL sample, stopper or cap, mix well and let stand for two minutes to allow the color to develop.
- 8. Set 100% T on spectrophotometer using a distilled water blank
- 9. Pour into colorimeter cell (also called a cuvette) and take a reading of % Transmittance.
- 10. Record the reading and calculate residual chlorine from the calibration curve.

The mg/L Cl₂ is read directly from the standard curve prepared by using potassium permanganate (KMnO₄) as the standard.

Optional: Development of chlorination breakpoint curve that demonstrates the relation between chlorine demand and residual chlorine.

- 11. Prepare chlorine standard stock solution.
- 12. Label chlorination bottles with numbers 1 to 14. Fill each bottle with 500 ml water sample.
- 13. Add increasing volumes of standard chlorine stock solution to give the following chlorine concentrations in the 14 bottles: 0.0, 0.5, 1.5, 3.0, 6.0, 8.0, 10.0, 12.0, 14.0, 15.0, 18.0, 20.0, 22.0 and 25.0 mg Cl₂/L, and mix thoroughly.
- 14. Shake flask well and wait for 20 minutes of chlorine contact in each flask.

15. Measure % transmittance as in step 2, with the addition of a few crystals (approximately 1.0 g) of potassium iodide (KI) for 2.b. step.

Calculations

For standard calibration curve

Chlorine Standards, mg/l	Transmittance, %

For breakpoint chlorination curve

Bottle #	Chlorine Dose, mg/l	Volume of Stock, ml	Transmittance, %	Chlorine Residual, mg/l

Questions

- 1. What is the main purpose for chlorination of water supplies and wastewater effluents? *The main purpose for chlorination of water supplies and wastewater effluents is the destruction of disease-causing organisms.*
- 2. What are the definitions of the following terms: Chlorine residual; combined chlorine residual, free available chlorine residual; and total chlorine residual?
 - *a.* chlorine residual: *The amount of available chlorine after a given contact time and under specific conditions.*
 - b. <u>combined chlorine residual</u>: chlorine residual from chlorine combined with ammonia or other nitrogen compounds.
 - *c.* <u>free available chlorine residual</u>: *is hypochlorite and/or hypochlorous acid-free existing in water.*
 - d. total chlorine residual: the total amount of chlorine available in a sample
- 3. What are the common minimum and maximum limitations for chlorinated effluents discharging to non-critical waters? To critical waters? (critical water defined as those waters which come into close human contact, such as lakes and rivers used for drinking water, shell fishing, and for water sports)

1.0 to 2.0 mg/L chlorine residual for non-critical water 1.5 to 2.5 mg/L chlorine residual for critical water

4. Discuss the importance of chlorination in water treatment and explain the different parts of the breakpoint chlorination curve.

Biochemical Oxygen Demand (BOD)

In the presence of free oxygen, aerobic bacteria use the organic matter found in wastewater as "food". The BOD test is an estimate of the "food" available in the sample. The more "food" present in the waste, the more Dissolved Oxygen (DO) will be required. The BOD test measures the strength of the wastewater by measuring the amount of oxygen used by the bacteria as they stabilize the organic matter under controlled conditions of time and temperature.

The BOD test is used to measure waste loads to treatment plants, determine plant efficiency (in terms of BOD removal), and control treatment plant processes. It is also used to determine the effects of discharges on receiving waters. A major disadvantage of the BOD test is the amount of time (5 days) required to obtain the results.

Principle

The BOD test relies on a measurable depletion of DO over a specified period. Experimentally BOD is determined by oxygen consumed by microorganisms in decomposing the organic matter present in the wastewater over a 5-d period of incubation. During the incubation period, mostly all sample of wastewater requires DO higher than the amount of oxygen available in the sample, so the sample must be diluted with aerated water at the beginning of the experiment. This dilution is done by adding dilution water to the sample in the BOD bottle

Apparatus

- 1. Dissolve Oxygen (DO) probe for measurement of DO in 300 mL BOD bottles.
- 2. Incubator, capable of maintaining $20 \pm 1^{\circ}C$

Reagents & Material

- 1. Dilution water (containing the necessary nutrients: phosphate buffer, magnesium sulfate, calcium chloride, ferric chloride).
- 2. 300 mL BOD bottles

Procedure

- 1. Completely fill one BOD bottles with dilution water.
- 2. Into new BOD bottles, partially filled with dilution water, carefully measure out the proper volume of sample. Add dilution water until the bottles are completely filled.

NOTE: If the modified Winkler procedure is to be used for DO measurements, two BOD bottles should be prepared for each dilution; one for determination of the initial DO and one for incubation and final DO measurement. If the meter method is used for DO measurements, the initial and final DO determinations can be performed on the same bottle.

- 3. Stopper each bottle taking care to avoid trapping air bubbles inside the bottles as the bottle stoppers are inserted.
- 4. Fill the top of each bottleneck around the stopper with dilution water.
- 5. Determine the initial DO content on one of each set of duplicate bottles, including the dilution water blank by one of the approved methods and record data on the lab sheet.
- 6. Place the bottles in the incubator at 20° C and incubate for five days.
- 7. At the end of exactly five days (± 3 hours), test the DO content of the incubated bottles.
- 8. Calculate the BOD for each dilution. The most accurate BOD will be obtained from those dilutions that have a depletion of at least 2 mg/L DO, and at least 1.0 mg/L DO residual. If there is more than one dilution that meets these criteria, the BOD results should be averaged to obtain a final BOD value.
- 9. The dilution water blanks are used <u>only</u> to check the quality of the dilution water. If the quality of the water is good and free from impurities, the depletion of DO should be less than 0.2 mg/L. In any event, do not use the depletion obtained as a blank correction.
- 10. Report the results of the nitrification inhibited samples as CBOD5 and uninhibited samples as BOD5.

Calculations

To determine the value of the BOD in mg/L, use the following formula:

Sample	Vol. (ml)	DO _o (mg/l)	DO ₅ (mg/l)	BOD ₅ (mg/l)
Blank	300			
Influent				
Effluent				

BOD, $mg/L =$	(Initial DO – Final DO) \times 300
DOD, IIIg / L = 1	mL Sample

Determination of Dissolved Oxygen by Winkler Titration

Dissolved oxygen represents the amount of oxygen dissolved in the liquid. While normal wastewater contains no dissolved oxygen unless the wastewater is very fresh or very weak wastewater. Natural unpolluted surface water contains dissolved oxygen (usually from 7 to 14 mg/1). This temperature-dependent oxygen-carrying capability is necessary for the life of fish and other aquatic organisms.

It is necessary to make dissolved oxygen determinations of samples at the time of collection.

Apparatus:

- 1. BOD bottles, 300 ml capacity
- 2. Incubator, thermostatically controlled at $20^{\circ}C$

Reagents:

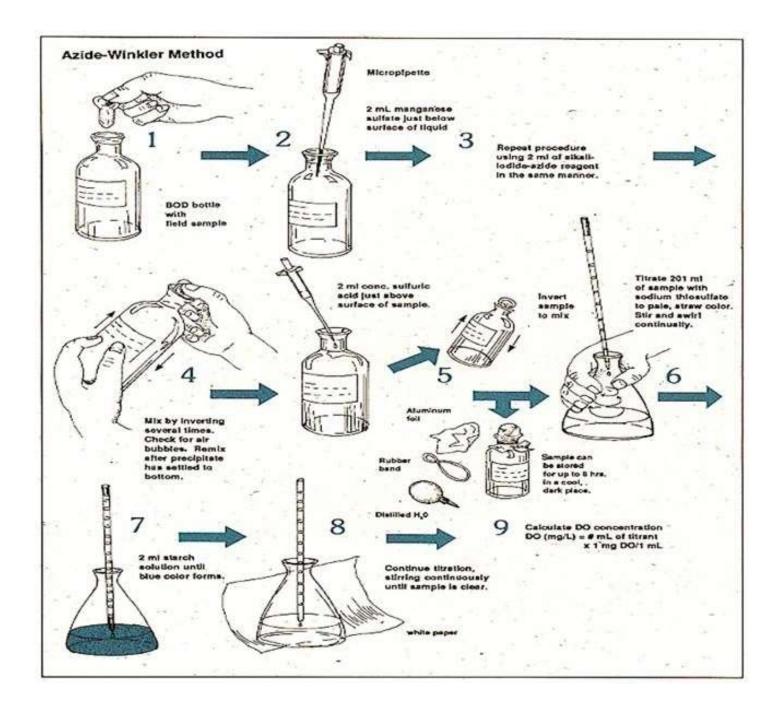
- 1. Manganous sulfate solution
- 2. Alkali iodide-azide solution
- 3. Sulfuric acid
- 4. Sodium thiosulfate solution (0.025N)
- 5. Starch solution

Procedure:

- 1. Completely fill a 300-ml BOD bottle with the sample to be analyzed.
- 2. By holding the tip of the pipet below the surface of the liquid, add <u>two ml Manganous</u> <u>sulfate</u> solution and <u>two ml alkali iodide-azide</u> solution.
- 3. Replace stopper, avoiding trapping air bubbles, and shake very well by gentle inversion. Repeat shaking after floc has settled halfway. Allow floc to settle again.
- 4. Remove stopper and add <u>two ml of concentrated sulfuric acid</u>. Hold pipet above the surface of the liquid. Mix until no floc is visible but not in direct sunlight.
- 5. Withdraw 203 ml of the solution into the Erlenmeyer flask and <u>titrate with sodium</u> thiosulfate solution until the yellow color almost disappears.
- 6. Add <u>one ml starch solution</u> and <u>continue titration</u> until the blue color just disappears.
- 7. Record the ml of thiosulfate used. Disregard any return to the blue color.

Calculations:

The dissolved oxygen present is expressed in mg/l and is equal to the number of ml of sodium thiosulfate solution used in the titration (step 7).



Questions

- 1. What does a BOD test measure? A BOD test measures the strength of the wastewater based on the amount of oxygen needed to stabilize the organic material in the wastewater.
- What can the BOD test be used for?
 A BOD test can be used to measure waste loadings to treatment plants, plant efficiency and the effects of discharge into a receiving stream, and to control the plant process.
- What are the types of samples and maximum sample holding time used for the BOD test?
 Grab or composite samples can be used. The maximum holding time is 48 hours at 4°C.
- Why must samples contain residual chlorine be dechlorinated before preparation of BOD dilutions?
 The presence of chlorine in a sample will inhibit the growth of bacteria during the BOD test.
- 5. Why must a dilution water blank be included with each series of BOD tests? *To check the quality of the dilution water.*
- 6. What are the acceptable temperature range and time of incubation for the BOD test? $20 \pm 1^{\circ}C$ and 5 days
- 7. What is being measured when nitrification inhibition is used in the BOD test? *Carbonaceous BOD (CBOD)*
- 8. What are the criteria for most valid results of the BOD test? Use dilutions which deplete at least 2.0 mg/L after 5 days and have at least 1.0 mg/L DO remaining in the dilution.

Chemical Oxygen Demand (COD)

The chemical oxygen demand (COD) test is commonly used to measure organic compounds in water sample indirectly. It is expressed in milligrams per liter (mg/L), which indicates the mass of oxygen consumed per liter of solution. COD is the measurement of the amount of oxygen in water consumed for chemical oxidation of pollutants.

This method covers the determination of COD in ground and surface waters, domestic and industrial wastewaters. COD value is always greater than BOD value. For domestic and some industrial wastewater, COD value is about 2.5 times BOD value.

Principle

Organic substances in the sample are oxidized by potassium dichromate in 50% sulfuric acid solution at reflux temperature. Silver sulfate is used as a catalyst, and mercuric sulfate is added to remove chloride interference. The excess dichromate is titrated with standard ferrous ammonium sulfate, using the ortho-phenanthroline ferrous complex as an indicator.

Apparatus

1. COD digester

Reagents & Material

- 1. Standard potassium dichromate solution (0.250 N)
- 2. Sulfuric acid reagent ($H_2SO_4 + Ag_2SO_4$).
- 3. Standard ferrous ammonium sulfate (FAS) (about 0.1 N)
- 4. Mercuric sulfate (Powdered HgSO₄).
- 5. Phenanthroline ferrous sulfate (ferroin) indicator solution.
- 6. COD Vials with stand
- 7. 250 mL conical flask

Procedure

- 1. Take 2 refluxing flask (one for the sample and one for the blank).
- 2. Add 20 mL of the sample to one flask; and the other flask for blank, add distilled water.
- 3. Add 10 mL of potassium dichromate reagent digestion solution to each flask.
- 4. Add 30 mL of sulfuric acid reagent
- 5. Attach flask to the condenser, start cooling water, switch on the COD Digester and fix the temperature at 150° C and heat for two hours.

- 6. After 2 hours, allow it to cool to the room temperature.
- 7. Transfer the contents of the sample to a conical flask.
- 8. Add few drops of ferroin indicators. The solution becomes green for sample and bluish green for blank.
- 9. Titrate it with the ferrous ammonium sulfate (FAS).
- 10. The end point of the titration is the appearance of the reddish-brown color.
- 11. Repeat step 8 to 11 for blank.

Calculations

To determine the value of the COD in mg/L, use the following formula:

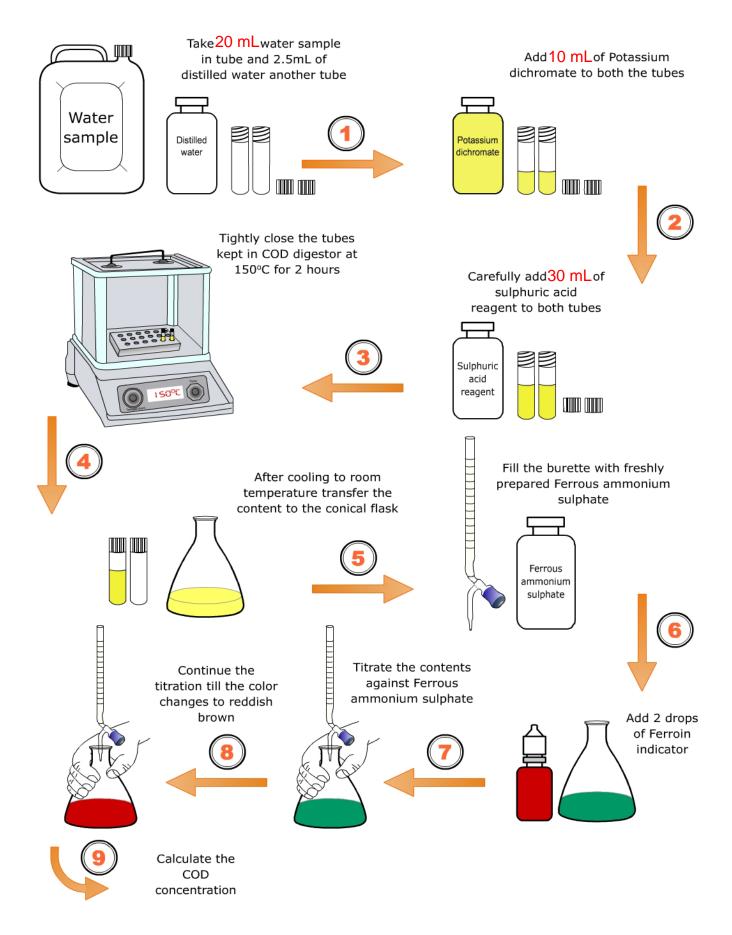
COD, mg/L =
$$\frac{(A - B) \times N \times 8000}{mL \text{ Sample}}$$

Where:

A = mL of FAS solution required for titration of the blank. B = mL of FAS solution required for of the sample. N = normality of the FAS solution.

Sample	Volume (mL)	Volume of FAS (mL)	COD (mg/L)
Blank			
Sample 1			
Sample 2			

Procedure Chart for COD



Ammonia- Nitrogen (NH₃-N) Distillation Titrimetric Method

Nitrogen compounds are of interest to wastewater treatment plant operators because of the importance of nitrogen in the life cycles of plants and animals. Nitrogen is a nutrient and occurs in many forms, including ammonia, organic, nitrate, and nitrite each of which may be tested for in a variety of ways. Raw wastewater nitrogen is usually present in the organic nitrogen and ammonia forms, with small quantities of the nitrite and nitrate forms. Depending on the amount of nitrifications that occurs within the plant, the effluent may contain either ammonia or nitrate nitrogen. Under normal circumstances, the nitrite form of nitrogen will not be present in large quantities due to its rapid oxidation or conversion to nitrate.

The presence of large concentrations of ammonia in a stream or lake can create a significant oxygen demand. This demand is caused by the conversion of ammonia to nitrate. High levels of nitrate in wastewater treatment plant effluent can cause algae to grow in large quantities. Dead and decaying algae can cause oxygen depletion problems that in turn can kill fish and other aquatic organisms in streams. For this reason, testing for nitrogen in the plant effluent is critical.

Principle

When ammonia gas is dissolved in water, it will react with the water to form some ammonium ions. Depending upon the pH of the solution, the ratio of ammonia to ammonium will vary. At a higher pH, there is more ammonia. At a lower pH, there is more ammonium. In the distillation procedure, the sample pH is raised to 9.5 and the ammonia gas formed will be removed by distillation. The ammonia gas is then absorbed in an acid solution where it is converted back to ammonium. The distillation eliminates the ammonia from the sample and leaves substances, which may interfere with the analysis behind.

Apparatus

- 1. Distillation apparatus consisting of:
 - 500 or 800 mL Kjeldahl flasks
 - Connecting bulbs
 - Vertical condensers
 - Hot plates
 - 300 mL receiving beakers or flasks
- 2. pH meter
- 3. Titration apparatus

Reagents & Material

- 1. Ammonia-free distilled water
- 2. Borate buffers solution
- 3. Sodium hydroxide solution, 1 N
- 4. Mixed indicator solution
- 5. Boric acid absorbing solution
- 6. Sulfuric acid titrant , 0.02 N

Procedure

- 1. Measure 200 ml of distilled water for blank, and 100 ml sample + 100 ml distilled water for sample into 500 ml flask.
- 2. Add 25 ml borate buffer to blank and samples.
- 3. Adjust pH to 9.5 with 1 N NaOH, using the pH meter.
- 4. Measure 25 ml of boric acid solution into 250 ml Erlenmeyer receiving flasks marked with sample designation.
- 5. Connect flasks to a distillation apparatus.
- 6. Check that cooling water is on; turn on the heating element to "High", and distill blank and samples; if the sample boils up the neck of the flask, reduce heat.
- 7. After 175 ml mark on 250 ml, Erlenmeyer receiving flask is reached, discontinue distillation.
- 8. Add 5 drops mixed indicator to blank and sample distillations; if sample color is similar to blank color so that less than 1.00 ml of titrant would be used, then transfer to stopper sample flask and put aside for selective ion determination.
- 9. Titrate blank with $0.02N H_2SO_4$ to pale lavender endpoint and record volume of titrant used.
- 10. Titrate sample with $0.02N H_2SO_4$ to the same color as blank.

Calculations

To determine the concentration of the NH₃-N in mg/L, use the following formula:

$$NH_3 - N, mg/L = \frac{(A - B) \times 280}{mL Sample}$$

Where:

 $A = mL \text{ of } 0.02N \text{ H}_2\text{SO}_4 \text{ titrant used for sample}$ $B = mL \text{ of } 0.02N \text{ H}_2\text{SO}_4 \text{ titrant used for blank}$

Sample ID	Volume (mL)	Volume of H2SO4 (mL)	NH3-N (mg/L)
Blank	200		
Sample 1	100		
Sample 2	100		

Questions

- 1. What are the four forms of nitrogen found in wastewater? *The four forms of nitrogen found in wastewater are organic nitrogen, Ammonia nitrogen, Nitrate nitrogen and Nitrite nitrogen.*
- 2. What occurs when excessive effluent ammonia is discharged into receiving waters? *The conversion of ammonia to nitrate creates a significant oxygen demand. This reduces the amount of oxygen available to the aquatic organism in the receiving stream.*
- If nitrogen testing is not going to be run immediately, how should samples be preserved?
 Acidify the sample to less than pH 2 with sulfuric acid and cool to 4°C.

Total Phosphorus

Wastewater is relatively rich in phosphorus compounds. Phosphorus is a nutrient used by organisms for growth. It occurs in natural water and wastewater bound to oxygen to form phosphates. Phosphates come from a variety of sources including agricultural fertilizers, domestic sewage, detergents, industrial process wastes and geological formations.

The discharge of wastewater containing phosphorus may cause algae growth in quantities sufficient to cause taste and odor problems in drinking water supplies. Dead and decaying algae can cause oxygen depletion problems which in turn can kill fish and other aquatic organisms in streams. For this reason, phosphorus removal is an essential role of wastewater treatment plants and testing for phosphorus in the plant effluent is critical.

Phosphates are classified as orthophosphates, polyphosphates, and organic phosphates. In this procedure, orthophosphates can be determined directly by colorimetric analysis. Other types require a digestion step to convert the "combined" phosphate to the ortho form for analysis. This gives the Total Phosphorus result.

Principle

Since phosphorus exists in several distinct forms in wastewater samples and the approved test method measures only the orthophosphate form. The sample is digested to convert both the polyphosphate and the organic phosphate to the ortho form.

An accepted method for determining phosphate-phosphorus is the Ascorbic Acid Procedure. The procedure is suitable for concentrations of 0.01 to 6 mg/L PO₄-P. Ammonium molybdate and antimony potassium tartrate react in acidic solution with orthophosphate to form a heteropoly acid (phosphomolybdic acid) which can be reduced by ascorbic acid to form an intense blue color.

Apparatus

- 1. Hotplate
- 2. Spectrophotometer with an infrared phototube for use at 880 nm with a light path of at least 2.5 cm.
- 3. An analytical balance capable of weighing to 0.1 mg accuracy.

Reagents & Material

- 1. Phenolphthalein indicator
- 2. Sulfuric acid solution, 5.0 N

- 3. Ammonium persulfate, crystal
- 4. Sodium hydroxide, 1.0 N
- 5. Antimony potassium tartrate solution
- 6. Ammonium molybdate Solution
- 7. Ascorbic acid solution
- 8. Combined reagent
- 9. Standard phosphate solution
- 10. 125 mL Erlenmeyer flasks; acid washed
- 11. 50 mL graduated cylinders; acid washed

Procedure

Digestion:

- 1. Measure an appropriate amount of sample diluted to 50 mL with distilled water.
- 2. Add one drop phenolphthalein indicator. If a red color develops, add sulfuric acid solution until color just disappears.
- 3. Add 1 mL of sulfuric acid solution and 0.4 g of ammonium persulfate.
- 4. Boil gently for 30 to 40 minutes or until the total volume is 10 mL.
- 5. Cool, add 1 drop of phenolphthalein and neutralize to a faint pink color with 1.0 N sodium hydroxide.
- 6. Make up to 50 mL with distilled water. The digested sample is then tested for total phosphate as outlined in next step.

Color Development:

1. Pipette 50.0 mL or an appropriate amount diluted to 50 mL of digested sample into an acid cleaned, dry 125 mL Erlenmeyer flask.

NOTE: If the only orthophosphate is to be determined, an undigested sample is used.

- 2. Add 1 drop of the phenolphthalein indicators. If a red color develops, add 5 N sulfuric acid until the color disappears.
- 3. Add 8.0 mL of combined reagent and mix thoroughly.
- 4. Allow at least 10 minutes (but not more than 30 minutes) for color development.
- 5. Measure absorbance at 880 nm using a reagent blank to zero the spectrophotometer.

NOTE: The reagent blank is made using 50 mL of distilled water carried through the digestion step and ascorbic acid procedure.

In highly colored or turbid samples, prepare a sample blank by adding all the reagents to the sample except the ascorbic acid and antimony potassium tartrate. Subtract the absorbance of this blank from the absorbance of the sample.

6. Check the sample's absorbance against the calibration curve and determine the concentration. Correct for dilution.

Calibration Curve:

Since the phosphate concentration is measured as a function of absorbance, a standard curve of absorbance versus known phosphate concentrations must be prepared. Six standard phosphorus concentrations and a distilled water blank are treated with the same digestion procedures as the samples. These 6 values are used to plot absorbance versus phosphate concentration to give a straight line passing through the origin.

Prepare 6 dilutions of the 5.0 mg/L Phosphate standard to result in the following final concentrations:

Final conc.	Volume of std.	Final vol. in a flask
0.1 mg/L	1.0 mL	50.0 mL
0.2 mg/L	2.0 mL	50.0 mL
0.4 mg/L	4.0 mL	50.0 mL
0.6 mg/L	6.0 mL	50.0 mL
0.8 mg/L	8.0 mL	50.0 mL
1.0 mg/L	10.0 mL	50.0 mL

Perform the Total Phosphorus procedure, including the digestion step and color development. Record the absorbances and plot the curve. At least one standard phosphate concentration must be included with each batch of samples as a check on the calibration curve. Prepare a new curve every six months.

Calculations

To determine the concentration of the phosphate-phosphorus in mg/L, use the following formula:

P, mg/L =
$$\frac{\text{mg/L from the curve} \times 50}{\text{volume of sample in mL}}$$

For example, if 5 mL of Influent gives an absorbance of 0.35, which represents 0.558 mg/L on the calibration curve, the corrected concentration of phosphate-phosphorus is:

P, mg/L =
$$0.558 \text{ x} 50 \text{ mL} \div 5 \text{ mL} = 5.58 \text{ mg/L}$$

Sample ID	Volume (mL)	Absorbance	Concentration	Corrected Concentration

Questions

- 1. Why is phosphorus removed from wastewater? Phosphorus is removed from wastewater because it provides a nutrient or food source for algae. Dead algae can cause serious oxygen depletion problems in receiving streams which in turn can kill fish and other aquatic life. Furthermore, algae can cause taste and odor problems in drinking water supplies.
- 2. What are the three basic forms of phosphorus found in wastewaters? The three basic forms of phosphorus found in wastewaters are Orthophosphates Polyphosphates and Organic phosphates.
- 3. What must be done before polyphosphates can be measured? *The samples must be acid hydrolyzed (or digested) to convert the polyphosphates to the orthophosphate form for measurement.*



How to Prepare a Laboratory Report

Laboratory reports are assignments that take the time to prepare. You will be given a one week to complete each lab report. All labs are graded on a 10-Point system. All reports are due on time; any late reports will lose grade points! There will be NO EXCUSES!

Cover Page

This page is the first page of your report and should have the title of the lab, your name, your computer number, date due and group.

Introduction

This is the second page of the lab report and should have the word INTRODUCTION on it! Include in this section some background information, the purpose of the lab, procedures or techniques (names) used in the lab "all information supposed to be related to environmental engineering."

Materials

Head this section MATERIALS. This section will include the materials your group used in the lab. Simply list all supplies you used in the lab. Include chemicals, reagents, apparatus, measuring devices, containers, processing equipment and so on.

Procedure

This section will include HOW you did your lab. In most cases you will be following a set of instructions, writes a short version of it. DO NOT include the handout procedures and instructions with your report.

Data and Calculations

In case there is any calculation states the equations used and give a sample calculation for only one of the tested samples.

Results and Discussion

This is where all your data goes. ALL GRAPHS and CHARTS go here. All chart graphs need headings and labels. Discuss the results in relation to the water standards, and answer the questions that are always included in your lab handout.

Conclusion

This is a two-paragraph section. The first paragraph will relate the findings that you have found in the experiment. The second paragraph will be a short paragraph on sources of error or confirmation of final findings. Here you will either explain incorrect, unexpected or unexplained results or confirm correct findings and proper techniques.

References

This section should be labeled REFERENCES. Given credit for any information you may have gotten from the textbooks, references books, the Internet or other sources.

Parameter	المواصفة	نوع التحليل	النوع
Color	15 وحدة	اللون	j
Turbidity	5 حدات	العكارة	الخصائص الطبيعية
Taste	مقبول	الطعم	2
Odor	عديم الرائحة	الرائحة	لطبيع
рН	6.5 - 8.5	الرقم الهيدروجيني	بم عاي
TDS	700	الاملاح الكلية الذائبة	
Total hardness ⁽²⁾	300	العسر الكلي	
Magnesium	30	المغنسيوم	
Calcium	75	الكالسيوم	
Sodium	200	الصوديوم	
Sulfate	250	الكبريتات	
Chloride	250	الكلوريدات	
Aluminum	0.2	الالمنيوم	
Iron	0.3	الحديد	1
Copper	1	النحاس	صأند
Zinc	5	الزنك (الخارصين)	5
Manganese	0.05	المنجنيز	لخصائص الكيميائية
Arsenic	0.05	الزرنيخ	أئية
Cadmium	0.01	الكادميوم	
Mercury	0.001	الزئبق	
Selenium	0.01	السلينيوم	
Chrome	0.05	الكروم	
Nitrate	10	النترات	
Nitrite	0.005	النتريت	
Fluoride	0.8	الفلورايد	
Lead	0.05	الرصاص	
Alpha Radiation	0.1 pCi/L	أشعة ألفا	الخصائص
Beta Radiation	1 pCi/L	أشعة بيتا	الإشعاعية
Fecal Coliform	Zero	بكتريا القولون	
Bacteria (for 24 hr)	50 cell/mL	بكتريا (خلال ٢٤ ساعة)	الخصائص الميكروبية
Bacteria (for 72 hr)	100 cell/mL	بكتريا (خلال ٧٢ ساعة)	الميدروبيه

مواصفات وزارة الشئون البلدية والقروية لمياه الشرب المعبأة (١٤٢٦ه - ٢٠٠٥م)

Note:

All units are in mg/L, except for stated one.
 Calculated as calcium carbonate

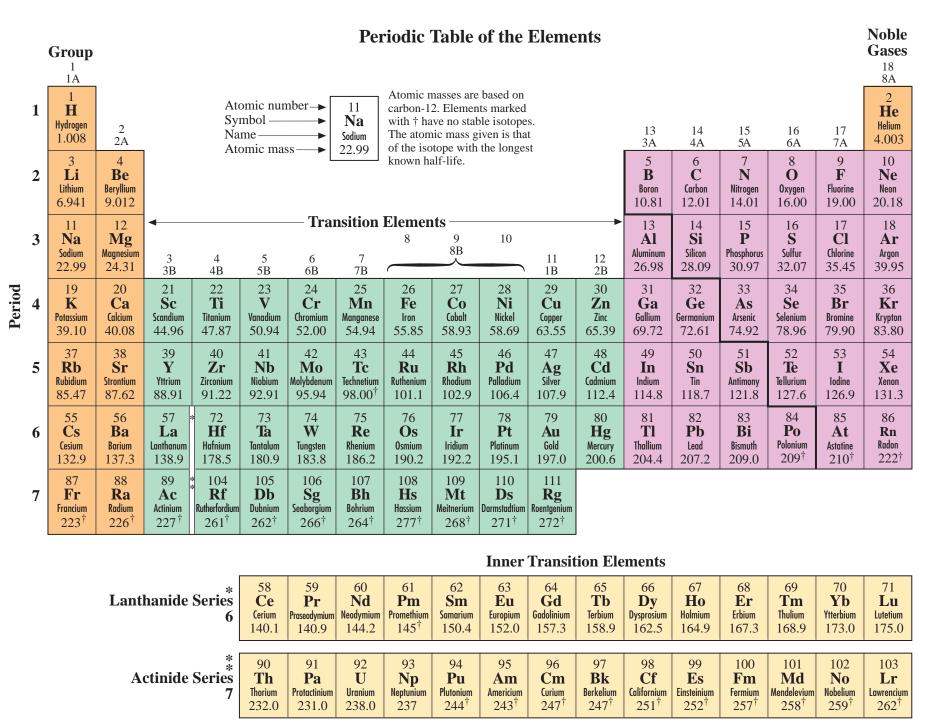


Figure A.1 The periodic table of the elements.