UNIVERSITY OF ANBAR
COLLEGE OF ENGINEERING
CIVIL ENGINEERING DEPARTMENT

SANITARY AND ENVIRONMENTAL ENGINEERING LAB

LECTURES
FOR
UNDERGRADUATE STUDENTS
4th GRADE

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Contents

Preface

Continued leadership in environmental protection requires efficient transfer of innovative environmental technologies to the next generation of engineers.

Responding to this challenge, the Civil Engineering department in our university redesigned the undergraduate civil engineering curriculum and created a new senior-level laboratory course. This laboratory manual is one of the products of the course development. Our goal is to disseminate this information to help expose undergraduates at Civil Engineering department and at other related departments to current environmental engineering problems and innovative solutions.

A major goal of the undergraduate laboratory course is to develop an atmosphere where student understanding will emerge for the physical, chemical, and biological processes that control material fate and transport in environmental and engineered systems. Student interest is piqued by laboratory exercises that present modern environmental problems to investigate and solve.

The experiments were designed to encourage the process of "learning around the edges." The manifest purpose of an experiment may be a current environmental problem, but it is expected that students will become familiar with analytical methods in the course of the laboratory experiment (without transforming the laboratory into an exercise in analytical techniques). It is our goal that students employ the theoretical principles that underpin the environmental field in analysis of their observations without transforming the laboratories into exercises in process theory. As a result, students can experience the excitement of addressing a current problem while coincidentally becoming cognizant of relevant physical, chemical, and biological principles.

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CHAPTER ONE

LABORATOR SAFETY

1. Introduction

Safety is a collective responsibility that requires the full cooperation of everyone in the laboratory. However, the ultimate responsibility for safety rests with the person carrying out a given procedure. In the case of an academic laboratory, that person is usually the student. Accidents often result from an indifferent attitude, failure to use common sense, or failure to follow instructions. Each student should be aware of what the other students are doing because all can be victims of one individual's mistake. Do not hesitate to point out to other students that they are engaging in unsafe practices or operations. If necessary, report it to the instructor. In the final assessment, students have the greatest responsibility to ensure their own personal safety.

This guide provides a list of do's and don'ts to minimize safety and health problems associated with experimental laboratory work. It also provides, where possible, the ideas and concepts that underlie the practical suggestions. However, the reader is expected to become involved and to contribute to the overall solutions. The following are general guidelines for all laboratory workers:

1) Follow all safety instructions carefully.

2) Become thoroughly acquainted with the location and use of safety facilities such as safety showers, exits and eyewash fountains.

3) Become familiar with the hazards of the chemicals being used, and know the safety precautions and emergency procedures before undertaking any work.

4) Become familiar with the chemical operations and the hazards involved before beginning an operation.

2. Personal Protection

1.2.1 Eye Protection

All people in the laboratory including visitors must wear eye protection at all times, even when not performing a chemical operation. Wearing of contact lenses in the laboratory is normally forbidden because contact lenses can hold foreign materials against the cornea. Furthermore, they may be difficult to remove in the case of a splash. Soft contact lenses present a particular

hazard because they can absorb and retain chemical vapors. If the use of contact lenses is required for therapeutic reasons fitted goggles must also be worn.

1.2.2 Clothing

Clothing worn in the laboratory should offer protection from splashes and spills, should be easily removable in case of accident, and should be at least fire resistant.

Nonflammable, nonporous aprons offer the most satisfactory and the least expensive protection.

High-heeled or open-toed shoes, sandals, or shoes made of woven material should not be worn in the laboratory.

1.2.3 Gloves

Gloves can serve as an important part of personal protection when they are used correctly. Check to ensure the absence of cracks or small holes in the gloves before each use. In order to prevent the unintentional spread of chemicals, gloves should be removed before leaving the work area and before handling such things as telephones, doorknobs, writing instruments, computers, and laboratory notebooks. Gloves may be reused, cleaned, or discarded, consistent with their use and contamination. A wide variety of gloves is available to protect against chemical exposure. Because the permeability of gloves of the same or similar material varies from manufacturer to manufacturer, no specific recommendations are given here. Be aware that if a chemical diffuses through a glove, that chemical is held against the worker's hand and the individual may then be more exposed to the chemical than if the glove had not been worn.

1.2.4Personal Hygiene

Everyone working in an environmental engineering laboratory should be aware of the dangers of ingesting chemicals. These common sense precautions will minimize the possibility of such exposure:

1) Do not prepare, store (even temporarily), or consume food or beverages in any chemical laboratory.

2) Do not smoke in any chemical laboratory. Additionally, be aware that tobacco products in opened packages can absorb chemical vapors.

4) Wash hands and arms thoroughly before leaving the laboratory, even if gloves have been worn.

5) Wash separately from personal laundry, lab coats or jackets on which chemicals have been spilled.

6) Never wear or bring lab coats or jackets into areas where food is consumed.

7) Never pipette by mouth. Always use a pipette aid or suction bulb.

3. Laboratory Protocol

The chemistry laboratory is a place for serious learning and working. Horseplay cannot be tolerated. Variations in procedures including changes in quantities or reagents may be dangerous. Such alterations may only be made with the knowledge and approval of the instructor.

1.3.1 Housekeeping

In the laboratory and elsewhere, keeping things clean and neat generally leads to a safer environment. Avoid unnecessary hazards by keeping drawers and cabinets closed while working. Never store materials, especially chemicals, on the floor, even temporarily. Work spaces and storage areas should be kept clear of broken glassware, leftover chemicals and scraps of paper. Keep aisles free of obstructions such as chairs, boxes and waste receptacles. Avoid slipping hazards by keeping the floor clear of ice, stoppers, glass beads or rods, other small items, and spilled liquids. Use the required procedure for the proper disposal of chemical wastes and solvents.

1.3.2 Cleaning Glassware

Clean glassware at the laboratory sink or in laboratory dishwashers. Use hot water, if available, and soap or other detergent. If necessary, use a mild scouring powder. Wear appropriate gloves that have been checked to ensure that no holes are present. Use brushes of suitable stiffness and size. Avoid accumulating too many articles in the cleanup area. Usually work space around a sink is limited and piling up dirty or cleaned glassware leads to breakage. Remember that the turbid water in a sink may hide a jagged edge on a piece of broken glassware that was intact when put into the water. A pair of heavy gloves may be useful for removing broken glass, but care must be exercised to prevent glove contamination. To minimize breakage of glassware, sink bottoms should have rubber or plastic mats that do not block the drains.

Avoid the use of strong cleaning agents such as nitric acid, chromic acid, sulfuric acid, strong oxidizers, or any chemical with a "per" in its name (such as perchloric acid, ammonium persulfate, etc.) unless specifically instructed to use them, and then only when wearing proper protective equipment. A number of explosions involving strong oxidizing cleaning solutions, such as chromic sulfuric acid mixtures, have been reported. The use of flammable solvents should be minimized and, when they are used, appropriate precautions must be observed.

1.3.3 Unattended Operation of Equipment

Reactions that are left to run unattended overnight or at other times are prime sources for fires, floods and explosions. Do not let equipment such as power stirrers, hot plates, heating mantles, and water condensers run overnight without fail-safe provisions and the instructor's consent. Check unattended reactions periodically. Always leave a note plainly posted with a phone number where you and the instructor can be reached in case of emergency. Remember that in the middle of the night, emergency personnel are entirely dependent on accurate instructions and information.

1.3.4 Fume Hoods and Ventilation

A large number of common substances present acute respiratory hazards and should not be used in a confined area in large amounts. They should be dispensed and handled only where there is adequate ventilation, such as in a hood. Adequate ventilation is defined as ventilation that is sufficient to keep the concentration of a chemical below the threshold limit value or permissible exposure limit. If you smell a chemical, it is obvious that you are inhaling it. However, odor does not necessarily indicate that a dangerous concentration has been reached. By contrast, many chemicals can be present at hazardous concentrations without any noticeable odor.

1.3.5 Refrigerators

Chemicals stored in refrigerators should be sealed, double packaged if possible, and labeled with the name of the material, the date placed in the refrigerator, and the name of the person who stored the material A current inventory should be maintained. Old chemicals should be disposed of after a specified storage period. Household refrigerators should not be used for chemical storage. If used for storage of radioactive materials, a refrigerator should be plainly marked with the standard radioactivity symbol and lettering, and routine surveys should be made to ensure that the radioactive material has not contaminated the refrigerator. Food should never be stored in a refrigerator used for chemical storage.

1.3.6 Radioactive Materials

Radioactive materials are used in the Environmental Engineering laboratories. Doors of rooms containing radioactive materials are clearly labeled. Areas where radioactive materials are used are clearly delineated with labeling tape and signs. All equipment within areas labeled radioactive are potentially contaminated and should not be touched or removed. Do not place anything into or take anything from an area labeled radioactive.

1.3.7 Working Alone

Avoid working alone in a building or in a laboratory.

4. Use of Chemicals

Before using any chemical you need to know how to safely handle it. The safety precautions taken are dependent on the exposure routes and the potential harmful effects.

1.4.1 Routes of Exposure

1) Ingestion

2) Inhalation

3) Absorbed through skin

4) Eye contact

Each potential exposure route requires different precautions. Chemical exposure may have acute (immediate, short term) or chronic (long term potentially cumulative) affects. Information on health hazards can be found on chemical labels and in Material Safety Data Sheets.

1.4.2 Chemical Labels

All chemicals must be labeled. Unlabeled containers of mystery chemicals or chemical solutions are a nightmare for disposal as well as a potential safety hazard.

In a laboratory covered under the Lab Standard, if a chemical is designated as a hazardous material, that is having the characteristics of corrosivity, ignitability, toxicity , reactivity, etc. All other chemicals must have at minimum a label with the full chemical name (not just the chemical formula), concentration, and date prepared.

1.4.3 Chemical Storage

There has been much concern, and some confusion, about the proper storage of laboratory chemicals. Here "proper" means the storage of chemicals in such a manner as to prevent incompatible materials from being accidentally mixed together in the event of the breakage of one or more containers in the storage area or to prevent the formation of reactive vapors that may require vented chemical storage areas. Below is a concise guide to the storage of common laboratory chemicals.

1) Perchloric acid is separated from all other materials.

2) Hydrofluoric acid is separated from all other materials.

3) Concentrated nitric acid is separated from all other materials.

4) Highly toxic materials (LD50 of 50 mg/kg or less) are stored separately.

5) Carcinogenic chemicals are stored separately.

6) Inorganic acids (except for 1, 2, 3 above) are stored separately.

7) Bases are stored separately.

8) Strong oxidizing agents are stored separately.

9) Strong reducing agents are stored separately.

10) Water reactive, pyrophoric and explosive materials are stored separately.

11) Flammable organic materials (solvents, organic acids, organic reagents) are stored separately.

5. Laboratory Measurements and Procedures

Measurements of masses, volumes, and preparation of chemical solutions of known composition are essential laboratory skills. The goal of this exercise is to gain familiarity with these laboratory procedures. You will use these skills repeatedly throughout the course.

1.5.1 Theory

Many laboratory procedures require preparation of chemical solutions. Most chemical solutions are prepared on the basis of mass of solute per volume of solution (grams per liter or Moles per liter). Preparation of these chemical solutions requires the ability to accurately measure both mass and volume. Preparation of dilutions is also frequently required. Many analytical techniques require the preparation of known standards. Standards are generally prepared with concentrations similar to that of the samples being analyzed. In environmental work many of the analyses are for hazardous substances at very low concentrations (mg/L or μg/L levels). It is difficult to weigh accurately a few milligrams of a chemical with an analytical balance. Often dry chemicals are in crystalline or granular form with each crystal weighing several milligrams making it difficult to get close to the desired weight. Thus it is often easier to prepare a low concentration standard by diluting a higher concentration stock solution. For example, 100 mL of a 10 mg/L solution of NaCl could be obtained by first preparing a 1 g/L NaCl solution (100 mg in 100 mL). One mL of the 1 g/L stock solution would then be diluted to 100 mL to obtain a 10 mg/L solution.

1.5.2 Experimental Methods.

Mass can be accurately measured with an electronic analytical balance. Perhaps because balances are so easy to use, they are easy to forget that they should be calibrated on a regular basis. It is recommended that balances be calibrated once a week, after the balance has been moved, or if excessive temperature variations have occurred. In order for balances to operate correctly they also need to be level. Most balances come with a bubble level and adjustable feet.

Before calibrating a balance verify that the balance is level. The environmental laboratory is equipped with balances manufactured by Denver Instruments. To calibrate the Denver Instrument balances:

1) Zero the balance by pressing the tare button.

2) Press the MENU key until "MENU #1" is displayed.

3) Press the 1 key to select Calibrate.

4) Note the preset calibration masses that can be used for calibration on the bottom of the display.

5) Place a calibration mass on the pan (handle the calibration mass using a cotton glove or tissue paper).

6) The balance will automatically calibrate. A short beep will occur and the display will read CALIBRATED for three seconds, and then return to the measurement screen.

Dry chemicals can be weighed in disposable plastic "weighing boats" or other suitable containers. It is often desirable to subtract the weight of the container in which the chemical is being weighed. The weight of the chemical can be obtained either by weighing the container first and then subtracting, or by "zeroing" the balance with the container on the balance.

6. Sampling

The significance of a chemical analysis depends to a large extent on the sampling programme. An ideal sample should be done which is both valid and representative. These conditions are met by collection of samples through a process of random selection. This ensures that the composition of the sample is identical to that of the water body from which it is collected and the sample shares the same physico-chemical characteristics with the sampled water at the time and site of sampling. The relevant factors for any sampling programme are (a) frequency of sample collection, (b) total number of samples, (c) size of each sample, (d)sites of sample collection, (e) method of sample collection, (f) data to be collected with each sample, and (g) transportation and care of samples.

It may be stated in general, that it is more meaningful to analyse a large number of separate samples taken at different times and different locations than to compile and analyse a single representative sample. Separate samples must be collected for chemical and biological analysis. , since the sampling and preservation techniques are quite different. For accurate analysis, it is desirable to allow a short-time interval between sampling and analysis. As matter of fact

temperature, pH and dissolved oxygen (D.O.) must be determined in the field and as quickly as possible after sampling.

1.6.1 Preservation

It is essential to protect samples from changes in composition and deterioration with aging due to various interactions. The optimum sample holding time ranges from zero for parameters .such as pH , temperature and D.O to one week for metals. The preservation techniques for various parameters are summarized in the following table. As mentioned above, these are essential for retarding biological action , hydrolysis of chemical compounds and complexes and reduction of volatility of constituents. It is desirable for accurate results, that analysis must be undertaken within 4 hours for some parameters and 24 hours for others, from the time of collection and it must be concluded within a week.

1.6.2 Water Sample Preservation

Parameter	Min. Sample Size (mL)	Container	Preservation
1	2	3	4
pH	100	Polythene	Measure within 0~4 hours
DO	100	Polythene	
COD	500	Polythene	Add H_2SO_4; refrigerate
Nitrogen Ammonia Nitrate+ Nitrite	 500 500	 Polythene Polythene	 Analyze as soon as possible,add0.8mL conc. H_2SO_4 / L Add 40 mg $HgCl_2$ / L and refrigerate
Cyanide	500	Polythene	Add NaOH to pH 12and 25mL of 2% ascorbic acid and refrigerate
Sulphide	500	Polythene	Add 1 mL of 2N $Zn(CH_3COO)_2$ and 2 mL of 1M NaOH Stir and refrigerate
Phosphate	500	Polythene/ Glass	Add 40 mg $HgCl_2$ / L and refrigerate
Phenol	500	Polythene/ Glass	Acidify with H_3PO_4 to pH 4.0 and add 1g Cu_2SO_4 .$5H_2O$ per L to inhibit biodegradation

CHAPTER TWO

EXPERIMENTS

DOs and DON'Ts in the Laboratory:

1. Do thoroughly clean the glassware before and after use.
2. Do handle the glassware carefully.
3. Do not handle chemicals with bare hands.
4. Do not blow out the last drop from the pipette. When the liquid has drained out completely, touch the tip of the pipette to the inner surface of the vessel.
5. Do not add water to acids. Do always add acid to water.
6. Do use large volumes of water, when a person is splashed with acid to prevent serious burns.
7. Do weigh the articles in a balance only at room temperature.
8. Do use different pipette for different reagents.
9. Do not pipette out acids and other toxic reagents by mouth.
10. Do read the level of the curve (meniscus), in all volumetric glassware, with the eye at approximately the same level as the curve of solution.

Experiment No.1 Determination of Solids in Water

1. Purpose (Aim)

The aim of the experiments is to determine the following types of solids in the given sample(s):

(a) Total solids

(b) Total dissolved solids

(c) Total suspended solids

(d) Settleable solids

2. Theory

'Total solids' is the term applied to the material left in the vessel after evaporation of a sample of water/waste water and its subsequent drying in an oven at a definite temperature. Total solids include "total suspended solids" the portion of total solids retained by a filter and "total dissolved solids" the portion that passes through the filter. Fixed solids is the residue remaining after ignition for 1 hour at 550°C. The solid portion that is volatilised during ignition is called volatile solids. It will be mostly organic matter. Waters that are low in organic matter and total mineral content and are intended for human consumption may be examined under 103–105°C or 179–

181°C. But water containing considerable organic matter or those with pH over 9.0 should be dried at 179–181°C. In any case, the report should indicate the drying temperature.

The sample is filtered and the filtrate evaporate in a weighed dish on a steam bath, the residue left after evaporation is dried to constant weight in an oven at either 103–105°C or 179–181°C. The increase in weight over that of the empty dish represents total dissolved solids and includes all materials, liquid or solid, in solution or otherwise, which passes through the filter and not volatilised during the drying process.

The difference between the total solids and the total dissolved solids will give the total suspended solids. The dishes with the residue retained after completion of the tests for total solids and total dissolved solids are subjected to heat for 1 hour in a muffle furnace held at 550°C. The increase in weight over that of the ignited empty vessel represents fixed solids in each instance.

The difference between the total dissolved/total suspended solids and the corresponding fixed solids will give volatile solids in each instance. All the quantities should be expressed in mg/L. Settleable matter in surface and saline waters as well as domestic and industrial wastes may be determined and reported on a volume basis as millilitre per litre.

3. Apparatus

1. Porcelain evaporating dishes of 150–200 mL capacity
2. Steam bath
3. Drying oven
4. Desiccators
5. Weigh balance
6. Filter paper (preferably of glass fibre)
7. Electric muffle furnace
8. Imhoff cone

4. Procedure

(a) Total solids

1. Ignite the clean evaporating dishes in the muffle furnace for 30 minutes at 550°C and cool in a desiccator.
2. Note down the empty weight of the dish (W_1).

3. Pour a measured portion (50 to 100 mL) of the well-mixed sample into the dish and evaporate the contents by placing the dish on a steam bath.

4. Transfer the dish to an oven maintained at either 103–105°C or 179–181°C and dry it for 1 hour.

5. Allow the dish to cool briefly in air before placing it, while still warm in a desiccator to complete cooling in a dry atmosphere.

6. Weigh the dish as soon as it has completely cooled (W_2).

7. Weight of residue = ($W_2 – W_1$) mg. W_2 and W_1 should be expressed in mg.

(b) Total dissolved solids

1. Filter a measured portion of the mixed sample (50 or 100 mL) through a filter paper and collect the filtrate in a previously prepared and weighed evaporating dish.

2. Repeat the steps 3 to 6 outlined in total solids procedure.

3. Weight of dissolved solids = ($W_5 – W_4$) mg. W_4 = Weight of empty evaporating dish in mg.

W_5 = Weight of empty evaporating dish in mg + Residue left after evaporating the filtrate in mg.

(c) Total suspended solids = Total solids – Total dissolved solids.

(d) Settleable solids by volume

1. Fill an imhoff cone to the litre mark with a thoroughly mixed sample.

2. Settle for 45 minutes.

3. Gently stir the sides of the cone with a rod or by spinning.

4. Settle 15 minutes longer.

5. Record the volume of Settleable matter in the cone as mL/L.

5. Observation

NO.	Item	Sample I	Sample II	Sample III
1	Volume of sample taken			
2	Wt. of empty evaporating dish = W_1 mg (For total solids)			
3	Wt. of dish + total solids = W_2 mg			
4	Total solids S_1 = ($W_2 – W_1$) mg			
5	Wt. of empty evaporating dish = W_4 mg (For total dissolved solids)			
6	Wt. of dish + total dissolved solids = W_5 mg			

7	Total dissolved solids $S_2 = (W_5 - W_4)$ mg			
8	Total suspended solids in mg/L $S_3 = (S_1 - S_2)$			
9	ml/L of Settleable solids			

6. Calculation

1. $^{mg}/_{L}$ Total Solids $= \frac{\text{mg Total Solids} \times 1000}{\text{ml of Sample}}$

2. $^{mg}/_{L}$ Total Dissolved Solids $= \frac{\text{mg Total Dissolved Solids} \times 1000}{\text{ml of Sample}}$

3. mg/L Total Suspended Solids = mg/L of Total Solids – mg/L of Total Dissolved Solids

Note: These calculations need to be shown only for one sample.

7. Result

NO.	Item	Sample I	Sample II	Sample III
1	mg/L of Total Solids			
2	mg/L of Total Dissolved Solids			
3	mg/L of Total Suspended Solids			
4	ml/L of Settleable Solids			

Experiment No.2 Determination of Turbidity of Water

1. Purpose (Aim)

To determine the turbidity of the given sample using Nephelometer in NTU.

2. Principle

The method presented below is based on a comparison of the intensity of light scattered by the sample in specific conditions with the intensity of light scattered by standard reference suspension under the same condition. The higher the intensity of scattered lights, higher the turbidity. Formazine polymer, which has gained acceptance as the turbidity standard reference suspension is used as a reference turbidity standard suspension for water. It is easy to prepare and is more reproducible in its lights scattering properties than the clay or turbid natural water standards previously used. The turbidity of a given concentration of Formazine has an approximate turbidity of 100 NTU, when measured on candle turbidity meter. Nephelometric turbidity units based on Formazine preparation will have approximate units derived from Jackson candle turbidimeter but will not be identical to them.

3. Apparatus

Nephelometer with accessories

4. Reagents

(i) Turbidity free distilled water (for setting zero).

(ii) Formazine turbidity concentrate (hydrazine sulphate + hexamine).

(iii) Formazine standard (for setting 100 of the instrument).

5. Preparation of Turbidity Free Distilled Water

Pass distilled water through a membrane filter having a precision pore size of less than 10 microns (Whatman filter No. 42). Rinse collecting flask at least twice with such filtered water and discard the next 200 mL. Use this filtered water for setting zero of the instrument.

6. Preparation of Formazine Turbidity Concentrate

(a) Solution I

Weigh accurately 5 g of 'Anal–R' quality hydrazine sulphate $(NH_2)2H_2SO_4$ into a 500 mL volumetric flask and add distilled water to make up to the mark. Leave the mixture to stand for 4 hours.

(b) Solution II

Weigh accurately 50g of 'Anal–R' quality hexamethylene tetramine $(CH_2)6N_4$ (hexamine) into a 500 mL volumetric flask and add distilled water to make up to the mark.

Mix equal volume of solution I and II to form Formazine turbidity concentrate. Allow it to stand in a closed container at 25°C to 30°C for 48 hours to produce insoluble white turbidity corresponding to 4000 NTU.

Note: Once prepared, Formazine turbidity concentrate (which corresponds to 10000 ppm SiO_2) is stable for 2 to 3 months.

Preparation of Formazine Standard

Dilute 25mL of the Formazine turbidity concentrate to 1 litre with turbidity free distilled water to obtain 250 ppm or 100 NTU for setting '100' of the instrument.

Note: Formazine standard 100 NTU should be prepared weekly.

6. Procedure

(1) Switch the instrument on.

(2) Open the lid of the sample compartment.

(3) Insert a test tube filled with distilled water into the sample compartment. Close the lid.

(4) Adjust 'SET 0' control to get '0' displayed on the read out.

(5) Open the lid. Replace the test tube filled with distilled water with a test tube filled with Formazine standard. Close the lid.

(6) Adjust the 'SET 100' control to get '100' displayed on the read out.

(7) Repeat the above operation to get consistent values of 0 to 100 within 1% to 2%.

7. Measurement of turbidity less than 100 NTU

1. Thoroughly shake the sample.
2. Wait until air bubbles disappear and pour the sample into the nephelometer tube.
3. Read the turbidity directly from the instrument.

8. Measurement of turbidity above 100 NTU

Dilute the sample with one or more volume of turbidity free distilled water until the turbidity fall below 100 NTU.

$$NTU\ of\ sample = \frac{A\ (B+C)}{C}$$ Where,

A = NTU found in diluted sample

B = volume of dilution water in mL C = sample volume taken for dilution in mL.

9. Observation

0 – 100 NTU		**> 100 NTU**			
Sample No.	NTU	A NTU	B ml	C ml	$NTU = A\,(B + C)/C$

10. Results

Description of Sample	**Turbidity in NTU**

10. Discussion:

QUESTIONS:

1. Where do you find the adverse effects of turbidity in environmental engineering? Mention two instances.
2. Discuss the significance of determination of turbidity in sanitary engineering.
3. Discuss the nature of materials causing turbidity in

(a) River water during flash flood

(b) Polluted river water

(c) Domestic wastewater

4. What is the standard unit of turbidity?
5. What are NTU and JTU?

Experiment No.3 JAR Test for Optimum Coagulant Dose

1. Purpose (Aim)

To determine the optimum dosage of given coagulant.

2. General

Chemical coagulation, flocculation and sedimentation together reduce suspended and colloidal solids and organic matter. Alum, ferrous and ferric salts, when used for clarification, result in producing better effluent than by the plain sedimentation. The exact doses of these coagulants cannot be theoretically calculated and therefore, laboratory tests have to be carried out using the jar test procedure.

3. Principle

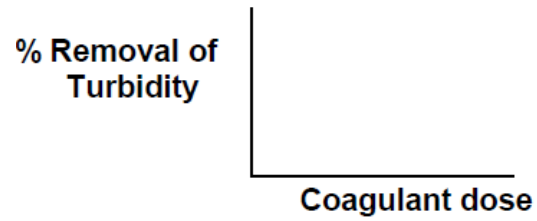
Metal salts hydrolyse in presence of the natural alkalinity to form metal hydroxides. The multivalent actions can reduce the zeta-potential. The metal hydroxides are good adsorbents and hence remove the suspended particles by enmeshing them.

4. Reagents

Standard Alum / Ferrous / Ferric salt solution: Prepare standard chemical solution such that 1 ml of solution contains 10 mg of salt in the solution.

5. Procedure

1. Measure initial turbidity of the sample.
2. Measure 1 liter quantities of the water to be tested into a series of glass jars.
3. Attach to stirring device.(Jar test apparatus).
4. Add progressive volumes of the chemical solution to each of the jars covering the range of chemical dosage expected.
5. Mix rapidly each sample for 1 minute.
6. Reduce the speed to about 10 rpm and mix for 15 minutes, (Flocculation).
7. Allow the floes to settle for 15 minutes.
8. Measure turbidity of each settled sample.
9. Plot graph % removal of turbidity Vs. Coagulant dose and select the optimum dosage.



6. Precautions

1. Add coagulant doses simultaneously to all glass jars while stirring.

2. It is advisable to siphon out the settled sample from the jars so as not to disturb the settled floc.

9. Observation

Initial Turbidity of the sample - ---- NTU

Sample NO.	Coagulate dose added	Final Turbidity	% of Removal	Remarks

10. Results

Optimum dose of coagulant = ---------- mg/l

Experiment No.4 Chemical Oxygen Demand (COD)

1. Purpose (Aim)

To determine the Chemical Oxygen Demand (C.O.D.) for given sample.

2. Theory

Chemical oxygen demand (COD) test determines the oxygen required for chemical oxidation of organic matter with the help of strong chemical oxidant. The limitation of COD test is that it can not differentiate between the biologically oxidizable and biologically inert material. COD determination has an advantage over BOD determination in that the result can be obtained in about 3-4 hours as compared to 5 days required for BOD test.

3. Principle

The organic matter gets oxidized completely by (Potassium dichromate) $K_2Cr_2O_7$ in the presence of H_2SO_4 to produce CO_2 + H_2O. The excess $K_2Cr_2O_7$ remaining after the reaction is titrated with Fe (NH_4) 2 $(SO_4)_2$.The dichromate consumed gives the O_2 required for oxidation of organic matter. Blanks are used also treated and titrated to get the correct value of COD.

4. Apparatus

1. Reflux apparatus consisting of a flat bottom 250 to 500 m1 capacity flask
2. Burner or hot plate with temperature regulator.

5. Reagents

1. Standard potassium dichromate 0.250 N

Dissolve 12.259g by $K_2Cr_2O_7$ dried at 103°C for 24 hours in distilled water and dilute to 1000 ml. Add about 120 mg sulphuric acid to take care of 6 mg/L NO_2-N.

2. Sulphuric Acid reagent

Add 10 g of Ag_2SO_4 to 1000 ml cone. H_2SO_4 and keep over night for dissolution.

3. Standard ferrous ammonium sulphate 0.1 N

Dissolve 39 g $Fe(NH_4)_2(SO_4)_2 .6H_2O$ in about 400 ml distilled water. Add 20 ml H_2SO_4 and dilute to 1000 ml.

4. Ferroin indicator

Dissolve 1.485 g of 1, 10 phenanthroline monohyderate and 695 mg $FeSO_4.7H_2O$ and dilute to 100 ml with distilled water.

5. Mercuric Sulphate

$HgSO_4$ crystals analytical grade.

6. Procedure

1. Place 50.0 mL of sample in a 500 mL refluxing flask.
2. Add 1g mercuric sulphate and a few glass beads.
3. Add sulphuric acid to dissolve the mercuric sulphate and cool.
4. Add 25.0 ml 0.25 N potassium dichromate solution and mix well.
5. Attach the flask to the condenser and start the cooling water.
6. Add the remaining acid reagent (70 mL) through the open end of condenser and mix well.
7. Apply heat and reflux for 5 hours.
8. Cool and wash down the condenser with distilled water.
9. Dilute the mixture to about twice its volume and cool to room temperature.
10. Titrate the excess dichromate with standard ferrous ammonium sulphate using ferroin indicator (2 to 3 drops).
11. The colour change from blue green to reddish indicates the end point.
12. Reflux in the same manner a blank consisting of distilled water of equal volume as that of the sample.

7. Observation

	Burette reading		**Volume of ferrous ammonium sulphate**
	Initial	**Final**	
Sample			
Blank			

8. Calculation

mg/L COD = $= \frac{(V_1 - V_2) \times C \times 8 \times 1000}{V}$ Where,

V_1 = mL ferrous ammonium sulphate used for blank

V_2 = mL ferrous ammonium sulphate used for sample

N = normality of ferrous ammonium sulphate V = volume of sample used.

9. Results

Sample NO.	COD, mg/L

10. Discussion

QUESTIONS:

1. Differentiate between B.O.D. and C.O.D.
2. Discuss the application of C.O.D. analysis in environmental engineering practice.
3. What are the interferences during C.O.D. test? How this can be eliminated?
4. Why ferroin is used as indicator in the C.O.D. test?

Another Procedure

(COD) was tested using Analyzer Microwave Digestion method that developed by (Gu Guozhen and Xue Xuejuan). A 5 ml of water was placed in a digestion vessel, then adding 5ml of ($K_2Cr_2O_7$ standard solution) and 5mL of (H_2SO_4–Ag_2SO_4 solution), shake well and put into the microwave for digestion at a specified time. After cooling, the solution was titrated with ferrous sulfate ammonium standard solution. The formula for computing COD is:

$$\text{COD } (O_2,\ \text{mg / L}) = \frac{(V_0 - V_1) \times C \times 8 \times 1000}{V_2}$$

Where

V_0 Amount of ammonium ferrous sulfate consumption (ml) in titration with distilled water

V_1 Amount of ammonium ferrous sulfate consumption (ml) in titration with sample of wastewater.

V_2 Sample volume (ml)

C Concentration of ferrous ammonium sulfate solution (mol / l)

8 1/2 of the oxygen molar mass (g / mol)

Experiment No.5 Determination of Dissolved Oxygen in Water

1. Purpose (Aim)

The aim of the experiment is to determine the quantity of dissolved oxygen present in the given sample(s) by using modified Winkler's (Azide modification) method.

2. Theory

The solubility of atmospheric oxygen in fresh water ranges from 14.4 mg/l at 0º C to about 7.0 mg/L at 35ºC at one atmospheric pressure. Since it is poorly soluble gas, its solubility directly varies with the atmospheric pressure at any given temperature. Analysis of Dissolved Oxygen (D.O.) is important in sanitary engineering practice. It is necessary to know D.O. levels to keep a check on stream pollution, and also to assess raw water quality. D.O. is necessary for all aerobic biological treatment processes. D.O. is the basis for BOD test which is an important parameter to evaluate pollution potential of wastes.

3. Principle

D.O. levels in natural and wastewaters are dependent on the physical, chemical and biochemical activities prevailing in the water body. The analysis of D.O. is a key test in water pollution control activities and waste treatment process control.

Improved by various techniques and equipment and aided by instrumentation, the Winkler (or iodometric) test remains the most precise and reliable titrimetric procedure for D.O. analysis. The test is based on the addition of divalent manganese solution, followed by strong alkali to the water sample in a glass-stoppered bottle. D.O. present in the sample rapidly oxidises in equivalent amount of the dispersed divalent manganous hydroxide precipitate to hydroxides of higher valency states. In the presence of iodide ions and upon acidification, the oxidised manganese reverts to the divalent state, with the liberation of iodine equivalent to the original D.O. content in the sample. The iodine is then titrated with a standard solution of thiosulphate.

In another meaning, Oxygen present in a sample rapidly oxidizes the dispersed divalent manganese hydroxide to its higher valency which precipitates as a brown hydrated oxide after addition of NaOH and KI. Upon acidification, manganese reverts to divalent state and liberates iodine from KI equivalent to the original DO content. The liberated iodine is titrated against $Na_2S_2O_3$ using starch as an indicator.

4. Apparatus

1. 300 mL capacity bottle with stopper
2. Burette
3. Pipettes, etc.

5. Reagents

1. Manganous sulphate solution ($MnSO_4.4H_2O$)
2. Alkali-iodide azide reagent
3. Conc. sulphuric acid (36 N)
4. Starch indicator
5. Standard sodium thiosulphate solution (0.025N)
6. Standard potassium dichromate solution (0.025N)

6. Procedure

1. Add 2 mL of manganous sulphate solution and 2 mL of alkali-iodide azide reagent to the 300 mL sample taken in the bottle, well below the surface of the liquid.

(The pipette should be dipped inside the sample while adding the above two reagents.)

2. Stopper with care to exclude air bubbles and mix by inverting the bottle at least 15 times.
3. When the precipitate settles, leaving a clear supernatant above the manganese hydroxide floc, shake again.
4. After 2 minutes of settling, carefully remove the stopper, immediately add 3 mL concentrated sulphuric acid by allowing the acid to run down the neck of the bottle.
5. Re stop and mix by gentle inversion until dissolution is complete.
6. Measure out 200 mL of the solution from the bottle to an Erlenmeyer flask.
7. Titrate with 0.025 N sodium thiosulphate solution to a pale straw colour.
8. Add 1–2 mL starch solution and continue the titration to the first disappearance of the blue colour and note down the volume of sodium thiosulphate solution added (V), which gives directly the D.O. in mg/L.

7. Observation:

Sample (2ml) × Standard sodium thiosulphate solution (0.025N) (Starch indicator)

Description of Sample	Trial NO.	Volume of Sample, ml	Burette Reading		Volume Titrant, ml	D.O. , mg/L
			Initial	Final		
Sample I						
Sample II						
Sample III						

8. Results

Description of Sample	D.O. , mg/L
Sample I	
Sample II	
Sample III	

9. Discussion:

QUESTIONS:

1. Discuss the environmental significance of dissolved oxygen.

2. Most of the critical conditions related to dissolved oxygen deficiency occur during summer months. Why?

4. The turbulence of water should be encouraged. Why?

5. Draw the oxygen saturation curve.

Experiment No.6 Determination of BOD of Waste Water Sample

1. Purpose (Aim)

To determine the amount of B.O.D. exerted by the given sample(s).

2. Principle

The Biochemical Oxygen Demand (B.O.D.) of sewage or of polluted water is the amount of oxygen required for the biological decomposition of dissolved organic matter to occur under aerobic condition and at the standardized time and temperature. Usually, the time is taken as 5 days and the temperature 20°C as per the global standard.

The B.O.D. test is among the most important method in sanitary analysis to determine the polluting power, or strength of sewage, industrial wastes or polluted water. It serves as a measure of the amount of clean diluting water required for the successful disposal of sewage by dilution. The test has its widest application in measuring waste loading to treatment plants and in evaluating the efficiency of such treatment systems.

The test consists in taking the given sample in suitable concentrations in dilute water in B.O.D. bottles. Two bottles are taken for each concentration and three concentrations are used for each sample. One set of bottles is incubated in a B.O.D. incubator for 5 days at 20°C; the dissolved oxygen (initial) content (D_1) in the other set of bottles will be determined immediately. At the end of 5 days, the dissolved oxygen content (D_2) in the incubated set of bottles is determined.

Then,

$$\text{B.O.D. , mg/L} = \frac{D_1 - D_2}{P}$$

Where,

P = decimal fraction of sample used.

D_1 = dissolved oxygen of diluted sample (mg/L), immediately after preparation.

D_2 = dissolved oxygen of diluted sample (mg/L), at the end of 5 days incubation.

Among the three values of B.O.D. obtained for a sample select that dilution showing the residual dissolved oxygen of at least 1 mg/L and a depletion of at least 2 mg/L. If two or more dilutions are showing the same condition then select the B.O.D. value obtained by that dilution in which the maximum dissolved oxygen depletion is obtained.

3. Apparatus

1. B.O.D. bottles 300mL capacity
2. B.O.D. incubator
3. Burette
4. Pipette
5. Air compressor
6. Measuring cylinder etc.

4. Reagents

1. Distilled water
2. Phosphate buffer solution
3. Magnesium sulphate solution
4. Calcium chloride solution
5. Ferric chloride solution
6. Acid and alkali solution
7. Seeding
8. Sodium sulphite solution
9. Reagents required for the determination of D.O.

5. Procedure

1. Place the desired volume of distilled water in a 5 litre flask (usually about 3 litres of distilled water will be needed for each sample).

2. Add 1mL each of phosphate buffer, magnesium sulphate solution, calcium chloride solution and ferric chloride solution for every litre of distilled water.

3. Seed the sample with 1–2 mL of settled domestic sewage.

4. Saturate the dilution water in the flask by aerating with a supply of clean compressed air for at least 30 minutes.

5. Highly alkaline or acidic samples should be neutralized to pH 7.

6. Destroy the chlorine residual in the sample by keeping the sample exposed to air for 1 to 2 hours or by adding a few mL of sodium sulphite solution.

7. Take the sample in the required concentrations. The following concentrations are suggested:

Strong industrial waste: 0.1, 0.5 and 1 per cent

Raw and settled sewage: 1.0, 2.5 and 5 per cent

Oxidized effluents: 5, 12.5 and 25 per cent

Polluted river water: 25, 50 and 100 per cent

8. Add the required quantity of sample (calculate for 650 mL dilution water the required quantity of sample for a particular concentration) into a 1000 mL measuring cylinder. Add the dilution water up to the 650mL mark.

9. Mix the contents in the measuring cylinder.

10. Add this solution into two B.O.D. bottles, one for incubation and the other for determination of initial dissolved oxygen in the mixture.

11. Prepare in the same manner for other concentrations and for all the other samples.

12. Lastly fill the dilution water alone into two B.O.D. bottles. Keep one for incubation and the other for determination of initial dissolved oxygen.

13. Place the set of bottles to be incubated in a B.O.D. incubator for 5 days at 20°C. Care should be taken to maintain the water seal over the bottles throughout the period of incubation.

14. Determine the initial dissolved oxygen contents in the other set of bottles and note down the results.

15. Determine the dissolved oxygen content in the incubated bottles at the end of 5 days and note down the results.

16. Calculate the B.O.D. of the given sample.

Note: The procedure for determining the dissolved oxygen content is same as described in the experiment under "Determination of dissolved oxygen".

6. Observation:

Sample NO. or Description	**Concentration**	**Dissolved Oxygen Content , mg/L**				**B.O.D. mg/L description (5 days 20°C)**
		Initial (D_1)		**Final (D_2)**		
		Bottle NO.	**B.O.D. value**	**Bottle NO.**	**B.O.D. value**	

Note: $\text{B.O.D. , mg/L} = \frac{D_1 - D_2}{P}$

If concentration is 0.1 per cent, then P = (0.1/100 = 0.001) and so on

7. Sample calculation:

D_1 = Initial Dissolved Oxygen = ... mg/L

D_2 = Dissolved Oxygen at the end of 5 days = ... mg/L

P = Decimal fraction of sample used =

Therefore, $\text{B.O.D. , mg/L} = \frac{D_1 - D_2}{P}$

8. Results:

Description of Sample	**$(B.O.D.)_5$, mg/L @ 20°C**
Sample I	
Sample II	
Sample III	

Experiment No.7 Determination of Hardness of Water by EDTA Titrimetric Method

1. Purpose (Aim)

To determine the total hardness of the given samples by EDTA titrimetric method.

2. General

Hardness is the capacity of water to react with soap, hard water requiring more amount of soap to produce lather. Scaling of hot water pipes, boilers and other household appliances is due to hard water. It is caused by dissolved ions of calcium and magnesium. The degree of hardness of drinking water has been classified in terms of the equivalent CaCO3 concentration as follows:

Classifications	Concentrations, mg/L.
Soft	**0 – 60**
Medium	**60 – 120**
Hard	**120 –180**
Very hard	**> 180**

3. Principle

Generally, In alkaline condition, EDT A reacts with Ca and Mg to form a soluble chelated complex. Ca and Mg ions develop wine red colour with eriochrome black T under alkaline condition. When EDT A is added as a titrant, Ca and Mg divalent ions get complexed resulting in a sharp change from wine red to blue which indicates end-point of the titration.

Originally, the hardness of water was understood to be a measure of the capacity of water for precipitating soap. Soap is precipitated chiefly by the calcium and magnesium ions commonly present in water, but may also be precipitated by ions of other polyvalent metals, such as aluminium, iron, manganese, strontium and zinc, and by hydrogen ions. Because, all but the first two are usually present in insignificant concentrations in natural waters, hardness is defined as a characteristic of water, which represents the total concentration of just the calcium and the magnesium ions expressed as calcium carbonate. However, if present in significant amounts, other hardness producing metallic ions should be included.

When the hardness is numerically greater than the sum of the carbonate alkalinity and the bicarbonate alkalinity, the amount of hardness, which is equivalent to the total alkalinity, is called carbonate hardness; the amount of hardness in excess of this is called non-carbonate hardness. When the hardness is numerically equal to or less than the sum of carbonate and

bicarbonate alkalinity all of the hardness is carbonate hardness and there is no non-carbonate hardness. The hardness may range from zero to hundreds of milligrams per litre in terms of calcium carbonate, depending on the source and treatment to which the water has been subjected.

4. Apparatus

1. Burette 2. Pipette 3. Erlenmeyer flask 4. Bottle, etc.

5. Reagents

1. Standard EDTA titrant (0.01 N): Dissolve 3.723 g EDTA sodium salt and dilute to 1000 ml
2. Eriochrome black T indicator: Mix O.5g dye with 100g NaCI to prepare dry powder.
3. Ammonia buffer solution: Dissolve 16.9g NH_4CI in 143 ml Of NH_4OH.

6. Procedure

1. Dilute 25 mL of sample (V) to about 50 mL with distilled water in an Erlenmeyer flask.
2. Add 1 mL of buffer solution.
3. Add two drops of indicator solution. The solution turns wine red in colour.
4. Add the standard EDTA titrant slowly with continuous stirring until the last reddish tinge disappears from the solution. The colour of the solution at the end point is blue under normal conditions.
5. Note down the volume of EDTA added (V_1).

7. Observation

Sample NO.	Volume of Sample (mL)	Burette Reading		Volume of EDTA (mL)
		Initial	Final	

8. Calculation

Hardness as $CaCO_3 = \frac{V_1 \times 1000}{V}$, mg/L

9. Results

Description of Sample	Total Hardness in mg/L as $CaCO_3$
Sample I	
Sample II	
Sample III	

10. Discussion

QUESTIONS:

1. Explain the significance of determination of hardness of water in environmental engineering.
2. What are the principal cations causing hardness in water and the major anions associated with them?
3. How is hardness classified?
4. Why is softening of water necessary? What are the advantages of soft water?

Experiment No.8 Free Chlorine in Water

1. Purpose (Aim)

To determine the concentration of free chlorine available in water.

2. General

The chlorination of water supplies and polluted water serves primarily to destroy or deactivate disease producing microorganisms. A secondary benefit, particularly in treating drinking water, is the overall improvement in water quality resulting from the reaction of chlorine with ammonia, iron, manganese, sulfide and some organic substances. Chlorine is widely used for disinfection of water, for deodourization since it is powerful oxidizing agent and is cheaply available.

Chlorination may produce adverse effects, taste and odour characteristic of phenols and other organic compounds present in a water supply may be intensified. Combined chlorine formed on chlorination of ammonia and amine-bearing water adversely affect some aquatic life. To fulfill the primary purpose of chlorination and to minimize any adverse effects, it is essential that proper testing procedure be used with a foreknowledge of the limitations of the analytical determination.

3. Principle

Chlorine combines with water to form hypochlorous and hypochloric acid. Hypochlorous acid dissociates to give the OCl^- ion .Quantities of ace and HOCI depend on pH of the solution. Chlorine is a strong oxidizing agent and liberates iodine from potassium iodide at pH = 8 or less. The liberatediodine is equivalent to the amount of chlorine present and can be titrated against sodium thiosulphate using starch as an indicator at pH 3 to 4 because the reaction is not stoichimetric at neutral pH due to partial oxidation of thiosulphate to sulphate.

4. Reagents

(1) Acetic acid (glacial). (2) Potassium iodide crystals.

(3) 0.1 N standard sodium thiosulphate solution (4) Starch indicator

5. Apparatus

Pipette (10 ml) – burette – beaker – volumetric flask – conical flask – analytical balance, etc.

6. Procedure

1. Pipette out 25 ml of water sample in conical flask.
2. Add a pinch of KI and sufficient distilled water (l00 mI).
3. Add 10 ml acetic acid and allow the reaction to complete.

4. Titrate free iodine liberated with 0.1 N thiosulphate solution. Note the volume required (A).

5. Prepare a reagent blank using distilled water. Note the volume of 0.1 N thiosulphate solution required for blank (B).

7. Formula

$$\text{Available Chlorine (mg/l)} = \frac{(A - B)\ N \times 35450}{\text{Volume of Sample}}$$

[1 g equiv. of chlorine = 35.45×10^3 mg of chlorine]

Where,

A = Volume of thiosulphate required by sample in ml.

B = Volume of thiosulphate required by blank in ml.

N = Normality of thiosulphate solution.

8. Observations

Sample NO.	Volume of Solution , mL	Burette Reading		Volume of thiosulphate used, mL
		Initial	Final	
1	10			
2	10			
3	10			
4	10			
Blank without sample	10			

Why do we prefer chlorination over other methods of disinfection?

Experiment No.9 Determination of pH of Water

1. Purpose (Aim)

To determine the pH of given samples using (1) universal indicator (2) pH paper, and (3) digital pH meter.

2. Principle

pH value of water indicates the hydrogen ion concentration in water and concept of pH was put forward by Sorenson (1909). pH is expressed as the logarithm of the reciprocal of the hydrogen ion concentration in moles/ litre at a given temperature. The pH scale extends from 0 (very acidic) to 14 (very alkaline) with 7 corresponding to exact neutrality at 25°C. pH is used in the calculation of carbonate, bicarbonate and CO2, corrosion and stability index etc. While the alkalinity or acidity measures the total resistance to the pH change or buffering capacity, the pH gives the hydrogen ion activity. pH can be measured calorimetrically or electrometrically. Colorimetric method is used only for rough estimation. It can be done either by using universal indicator or by using pH paper. The hydrogen electrode is the absolute standard for the measurement of pH. They range from portable battery operated units to highly precise instruments. But glass electrode is less subjected to interferences and is used in combination with a calomel reference electrode. This system is based on the fact that a change of 1 pH unit produces an electric charge of 59.1 mV at 25°C.

3. Apparatus

1. pH meter with electrode 2. Beaker 3. Thermometer

4. Colour comparator with discs 5. Cuvettes

4. Reagents

1. Buffer solutions 2. pH paper 3. Universal indicator

5. Procedure:

(a) Using Universal Indicator

1. 10 mL of sample is taken in a cuvette.

2. Another 10 mL sample is taken in another cuvette and 0.2 mL of universal indicator is added and placed in the hole provided for.

3. A colour disc corresponding to this indicator is inserted into the comparator and the disc rotated such that the 2 circles indicate identical colours.

4. The reading is noted.

5. The procedure can be repeated using an indicator whose range is near the value obtained.

6. The exact pH is obtained.

(b) Using pH Papers

1. Dip the pH paper in the sample.

2. Compare the colour with that of the colour given on the wrapper of the pH paper book.

3. Note down the pH of the sample along with its temperature.

(c) Using pH Meter

1. Follow the manufacturer's operating instructions.

2. Dip the electrode in the buffer solution of known pH.

3. Switch on the power supply and take the reading. Standardize the instrument using the calibrating knob.

4. After cleaning, again dip the electrodes in the buffer solution of pH 7. Note the reading. If it is 7, the instrument is calibrated. If not, correct the value and is manipulated so that the reading in the dial comes to 7.0.

5. A solution whose pH is to be found is taken in a beaker and the temperature knob is adjusted such that the temperature of solution is same as that in dial.

6. The electrode is washed with distilled water and reused with the solution and then it is dipped in the solution.

7. The reading on the dial indicates the pH of the solution.

6. Results

Sample NO.	**pH**		
	pH Paper	**pH meter**	**Universal Indicator**
1			
2			
3			

7. Discussion:

QUESTIONS:

1. Discuss the relationship between (a) pH and hydrogen ion concentration (b) pH and hydroxide ion Concentration?

2. Why is it necessary to maintain the pH of water nearly 7?

Experiment No.10 Determination of Chloride in Water

1. Purpose (Aim)

To determine the amount of chloride (in the form of Cl^-) present in the given water sample by Mohr's method.

2. Principle

If water containing chlorides is titrated with silver nitrate solution, chlorides are precipitated as white silver chloride. Potassium chromate is used as 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.

3. Apparatus

1. Burette 2. Pipettes 3. Erlenmeyer flasks 4. Measuring cylinder

4. Reagents

1. Chloride free distilled water.
2. Standard silver nitrate solution (0.0141N)
3. Potassium chromate indicator.
4. Acid or alkali for adjusting pH.

5. Procedure

1. Take 50mL of sample (V) and dilute to 100mL.
2. If the sample is coloured add 3mL of aluminium hydroxide, shake well; allow to settle, filter, wash and collect filtrate.
3. Sample is brought to pH 7–8 by adding acid or alkali as required.
4. Add 1mL of indicator (Potassium chromate).
5. Titrate the solution against standard silver nitrate solution until a reddish brown precipitate is obtained. Note down the volume (V_1).
6. Repeat the procedure for blank and note down the volume (V_2).

Water Sample vs. Silver Nitrate (N 0.0141) (Potassium Chromate Indicator)						
Sample NO.	**Trial NO.**	**Volume of Sample, (mL.)**	**Burette Reading**		**Volume of Silver Nitrate, (mL.)**	**Chloride, (mg/L.)**
			Initial	**Final**		
1	**1**					
	2					
	3					
2	**1**					
	2					
	3					
3	**1**					
	2					
	3					
Distilled Water	**1**					
	2					
	3					

6. Calculation:

For one sample (Sample No. …...)

V = V_1 = V_2 = N =

Chloride in mg/L $= \frac{(V_1 - V_2) \times N \times 35.46 \times 1000}{V} = \frac{(V_1 - V_2) \times N \times 500}{V} =$ mg/L

Description of Sample	Chloride, (mg/L.)
1	
2	
3	

What are the sources of chloride in water?

Experiment No.11 Determination of Ammonia in Wastewater Sample

1. Purpose (Aim)

To determine the ammonia nitrogen of the given sample of water.

2. Principle

Colorimetric method, using Nessler's reagent is sensitive to 20mg/L of ammonia N and may be used up to 5mg/L of ammonia N. Turbidity, colour and substances precipitated by hydroxyl ion interfere with the determination. The sample containing ammonia must be analyzed immediately after collection; if not 0.8 M conc. H_2SO_4/L should be added to the sample stored at 4°C.

3. Apparatus

Spectrophotometer, and Nessler tube tall form (50 mL or 100 mL capacity)

4. Reagents

1. Standard stock solution of ammonia, ρN = 1 000 μg / ml.

Weigh 3.819 g of ammonium chloride NH_4Cl which dried at 100 ~ 105 4°C 2 hours before), was dissolved in water and transferred to 1000 ml volumetric flask then diluted to the mark.

** Ammonia standard solution, ρN = 10 μg / ml.

Taking 5.00 ml of ammonia standard stock solution (prepared in 1) to 500 ml flask and diluted to the mark.

2. Potassium sodium tartrate solution, ρ = 500 g / L.

Weigh 50g of potassium sodium tartrate ($KNaC_4H_6O_6 \cdot 4H_2O$) and then dissolved in 100 ml of water, heated to boiling to exclude ammonia, then cooled sufficiently and diluted to 100 ml.

3. Mercuric iodide - Potassium iodide - Sodium hydroxide (HgI_2-KI-NaOH) solution

Weigh 16.0 g of sodium hydroxide (NaOH)and dissolved into 50 ml of water then later cooled to room temperature. Weigh 7.0 g of potassium iodide (KI) and 10.0 g mercuric iodide (HgI_2), dissolved in water, and then the solution was under stirring slowly added to the 50 ml sodium hydroxide solution, diluted with water to 100 ml. Stored in polyethylene bottle with a rubber or polyethylene lid tightly closed, in a dark storage.

5. Procedure

a. Calibration curve

1. In 10 ml colorimetric tube, the (ammonia standard solution ρN = 10 μg / ml) was added as follow to build the curve (0, 0.1, 0.2, 0.4, 0.8, 1.6, and 2) ml.

2. Adding 0.2 ml of (Potassium sodium tartrate solution, ρ = 500 g / L)

3. Adding 0.2 ml of Mercuric iodide - Potassium iodide - Sodium hydroxide (HgI_2-KI-NaOH) solution

4. Add water to the mark of the 10 ml tube and later all the solutions were mixed well and placed for settling for 10 min then analyzed by UV at 420nm.

b. Sample analysis

1 ml of the filtered sample was added to 10ml tube then 0.2 ml of (Potassium sodium tartrate solution, ρ = 500 g / L) and 0.2 ml of Mercuric iodide - Potassium iodide - Sodium hydroxide (HgI_2-KI-NaOH) solution were added respectively, and finally add water to the mark of the 10 ml tube and later all the solutions were mixed well and placed for settling for 10 min then analyzed by UV at 420nm.