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Lab Manual:

Environmental Engineering Laboratory(ACEB24)

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INTRODUCTION

1.1 Introduction

This course is intended to enhance the learning experience of the student in topics encountered in STM. In this lab, students are expected to gain experience in using the testing tools used in computer science and engineering and in interpreting the results of testing results in terms of the concepts introduced in the software testing methodology course. The student, lab teaching assistant, and faculty coordinator all have certain responsibilities toward successful completion of the lab's goals and objectives.

1.1.1 Student Responsibilities

The student is expected tocome prepared for each lab.Lab preparation includes understanding the labexperiment from the lab manual and reading the related textbook material.

Students have to write the allotted experiment for that particular week in the work sheets given and carry them to the Lab. In case of any questions or problems with the preparation, students can contact the Faculty Teaching the Lab course, but in a timely manner.

Students have to be in formal dress code, wear shoes and lab coat for the Laboratory Class.

After the demonstration of experiment by the faculty, student has to perform the experiment individually. They have to note down the observations in the observation Tables drawn in work sheets, do the calculations and analyze the results.

Active participation by each student in lab activities is expected. The student is expected to ask the Faculty any questions they may have related to the experiment.

The student should remain alert and use commonsense while performing the lab experiment.They are also responsible for keeping a professional and accurate record of the labexperiments in the files provided.

1.1.2 Responsibilities of Faculty Teaching the Lab Course

The Faculty shall be completely familiar with each labprior to the laboratory. He/She shall provide the students with details regarding the syllabus and safety review during the first week.Lab experiments should be checked in advance to make sure that everything is in working order.The Faculty should demonstrate and explain the experiment and answer any questions posed by the students.Faculty have to supervise the students while they perform the lab experiments. The Faculty is expected to evaluate the lab worksheets and grade them based on their practical skills and understanding of the experiment by taking Viva Voce. Evaluation of work sheets has to be done in a fair and timely manner to enable the students, for uploading them online through their CMS login within the stipulated time.

1.1.3 Laboratory In-charge Responsibilities

The Laboratory In-charge should ensure that the laboratory is properly equipped, i.e., the Faculty teaching the lab receive any equipment/components necessary to perform the experi-

ments.He/She is responsible for ensuring that all the necessary equipment for the lab is available and in working condition. The Laboratory In-charge is responsible for resolving any problems that are identified by the teaching Faculty or the students.

1.1.4 Course Coordinator Responsibilities

The course coordinator is responsible fo rmaking any necessary corrections in Course Description and lab manual. He/She has to ensure that it is continually updated and available to the students in the CMS learning Portal.

1.2 Lab Policy and Grading

The student should understand the following policy:

ATTENDANCE: Attendance is mandatory as per the academic regulations.

LAB RECORD's: The student must:

1. Write the work sheets for the allotted experiment and keep them ready before the beginning of eachlab.
2. Keep all work in preparation of and obtained during lab.
3. Perform the experiment and record the observations in the worksheets.
4. Analyze the resultsand get the work sheets evaluated by the Faculty.
5. Upload the evaluated reports online from CMS LOGIN within the stipulated time.

Grading Policy:

The final grade of this course is awarded using the criterion detailed in the academic regulations. A large portion of the student's grade is determined in the comprehensive final exam of the Laboratory course (SEE PRACTICALS),resulting in a requirement of understanding the concepts and procedure of each lab experiment for successful completion of the lab course.

INSTRUCTIONS TO STUDENTS

• Before entering the lab the student should carry the following things (MANDATORY)
  o Identity card issued by the college.
  o Work Sheets
• Student must sign in and sign out in the register provided when attending the lab session without fail.
• Come to the laboratory in time. Students, who are late more than 15 min., will not be allowed to attend the lab.
• Students need to maintain 100% attendance in lab if not a strict action will be taken.
• All students must follow a Dress Code while in the laboratory
• Foods, drinks are NOT allowed.
• All bags must be left at the indicated place.
• Refer to the lab staff if you need any help in using the lab.
• Respect the laboratory and its other users.
• Workspace must be kept clean and tidy after experiment is completed.
• Read the Manual carefully before coming to the laboratory and be sure about what you are supposed to do.

• Do the experiments as per the instructions given in the manual.
• Copy all the programs to observation which are taught in class before attending the lab session.
• Students are not supposed to use floppy disks, pen drives without permission of lab- incharge.
• Lab records need to be submitted on or before the date of submission.
• Computer labs are established with sophisticated and high end branded systems, which should be utilized properly.
• Students / Faculty must keep their mobile phones in SWITCHED OFF mode during the lab sessions. Misuse of the equipment, misbehaviors with the staff and systems etc., will attract severe punishment.
• Students must take the permission of the faculty in case of any urgency to go out; if anybody found loitering outside the lab / class without permission during working hours will be treated seriously and punished appropriately.
• Students should LOG OFF/ SHUT DOWN the computer system before he/she leaves the lab after completing the task (experiment) in all aspects. He/she must ensure the system / seat is kept properly.

1.3 Course Goals and Objectives

The engineering geologist's main objective is to protect life and property from damage caused by different geological conditions. The practice of engineering geology is also very closely linked to the practice of geological engineering and geotechnical engineering.

Students will try to learn:
The course should enable the students to: I. Investigate the different characteristics of water & wastewater Understand the shift from 2D representation to 3Dsimulation.
II. Outline the procedure for preparations of stock and standard solutions, their handling, storage,etc.
III.Assess the suitability of water for drinking, irrigation purpose and concretingworks.
IV.Determine the BOD, COD and bacterial density of portablewater.

1.4 Use of Laboratory Instruments

One of the major goals of this lab is to familiarize the student with the proper equipment andtechniques for conducting experiments. Some understanding of the lab instruments is necessaryto avoid personal or equipment damage.By understanding the device's purpose and following a fewsimple rules, costly mistakes can be avoided.

The following rules provide a guideline for instrument protection.

1.4.1 Instrument Protection Rules

1. New students must receive an orientation on lab operating procedures before working in a lab.
2. Students shall publish a safety checklist for equipment for which they are responsible.
3. Students must read the safety checklist for each piece of equipment before operating it.
4. Ensure you know the location of the emergency stop button before starting equipment.
5. Always depressurize accumulators or pneumatic reservoirs before working on fluid power apparatus.

6. Check the application pressure, system pressure, and component pressure before connecting a system to a pump or pressure source. The maximum operating pressures are listed on equipment labels or published on manufacturer websites.
7. Periodically check hoses for leakage, cracks, kinks, or breaks.
8. Test your equipment for leaks at low pressure before raising the pressure to the operating pressure.
9. All components shall operate within manufacturer's specifications.
10. Equipment shall incorporate an emergency stop or emergency return control, whichever provides maximum safety.
11. Emergency stops shall be readily accessible under all conditions of working and shall operate immediately.
12. Equipment shall be designed so that loss of electrical, pneumatic and/or hydraulic power shall not cause a hazard.
13. Pump inlet temperatures should not exceed 600C when maximum ambient temperatures exist.
14. Rotating parts shall be guarded to provide adequate protection against hazard.
15. Flexible hoses shall only be used where necessary. Their length shall be minimized and they shall be protected from abrasion. If failure causes a hazard, the hose shall be restrained or shielded.

1.5 Data Recording and Reports

1.5.1 The Laboratory Notebook:

Students must record their experimental values in the provided tables in this laboratory manual and reproduce them in the lab reports. Reports are integral to recording the methodology and results of an experiment. In engineering practice, the laboratory notebook serves as an invaluable reference to the technique used in the lab and is essential when trying to duplicate a result or write a report. Therefore, it is important to learn to keep accurate data. Make plots of data and sketches when these are appropriate in the recording and analysis of observations. Note that the data collected will be an accurate and permanent record of the data obtained during the experiment and the analysis of the results. You will need this record when you are ready to prepare a lab report.

1.5.2 The Laboratory Worksheets:

Reports are the primary means of communicating your experience and conclusions to other professionals. In this course you will use the lab report to inform your LTA about what you did and what you have learned from the experience. Engineering results are meaningless unless they can be communicated to others. You will be directed by your LTA to prepare a lab report on a few selected lab experiments during the semester. Your assignment might be different from your lab partner's assignment.
Your laboratory report should be clear and concise. The lab report shall be typed on a word processor. As a guide, use the format on the next page. Use tables, diagrams, sketches, and plots, as necessary to show what you did, what was observed, and what conclusions you can draw

from this. Even though you will work with one or more lab partners, your report will be the result of your individual effort in order to provide you with practice in technical communication.

CONCLUSIONS - The conclusion section should provide a take-home message summing up what has been learned from the experiment:

1. Briefly restate the purpose of the experiment (the question it was seeking to answer)
2. Identify the main findings (answer to the research question)
3. Note the main limitations that are relevant to the interpretation of the results
4. Summarise what the experiment has contributed to your understanding of the problem.

LAB-1 pH

2.1 Objective

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

2.2 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 colorimetrically 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.

2.3 Apparatus

1. pH meter with electrode 2. Beaker 3. Thermometer 4. Colour comparator with discs 5. Cuvettes

2.4 Reagents

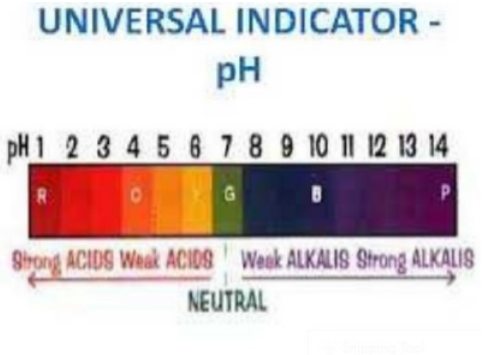
1. Buffer solutions 2. pH paper 3. Universal indicator

2.5 Procedure

(a) Using Universal Indicator (If comparators are not available, compare the colour with colours given in the chart.) 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.



2.6 Observations

Sample No.	pH		
	pH paper	pH meter	Universal indicator

2.7 Result

The pH of given water sample is . . . . . .

2.8 Further Probing Questions

1. What is the pH of pure water at 25°C? 2. What is the H+ ion concentration in pure water?

LAB-2 Turbidity

3.1 Objective

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

3.2 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.

3.3 Apparatus

Nephelometer with accessories

3.4 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).

3.5 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 atleast twice with such filtered water and discard the next 200 mL. Use this filtered water for setting zero of the instrument.

3.6 Preparation of Formazine Turbidity Concentrate

3.6.1 Solution I

Weigh accurately 5 g of 'Anal–R' quality hydrazine sulphate (NH2)2H2SO4 into a 500 mL volumetric flask and add distilled water to make up to the mark. Leave the mixture to stand

for 4 hours

3.6.2 Solution II

Weigh accurately 50g of 'Anal–R' quality hexamethylenetetramine (CH2)6N4 (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 to10000 ppm SiO2) is stable for 2 to 3 months.

3.7 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.

3.8 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%.

3.8.1 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.

3.8.2 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 =A(B+C) C

A = NTU found in diluted sample

B = volume of dilution water in mL

C = sample volume taken for dilution in mL

3.9 Observation:

0-100 NTU		>100 NTU			
Sample No.	NTU	A (ml)	B(ml)	C(ml)	NTU = A(B+C)/C

3.10 Result

The turbidity of the given sample using Nephelometer is ……..

3.11 Probing Further Questions

1. The good quality of water should have dissolved solids of?
2. In a Nephelometer the light detectors are at

LAB-3 Conductivity

4.1 Objective

To determine conductivity of given water sample

4.2 Principle

Conductivity is a numerical expression of the ability of an aqueous solution to carry the electric current. This ability depends on the presence of ions, their mobility, valence, relative concentrations and on the temperature of measurement. The inorganic acids, bases, and salt solutions are relatively good conductors. On the contrary, molecules of organic compounds that do not dissociate in aqueous solution have a poor conductivity. The conductivity is measured in the laboratory in term of resistance measured in ohms. The electric resistance of a conductor is inversely proportional to its cross sectional area and directly proportional to its length. The magnitude of the resistance measured in an aqueous solution therefore depends on the characteristics of the conductivity cell used. Specific resistance is the resistance of a cube of 1cm. In aqueous solutions such a measurement is seldom made because of the difficulties in fabrication of electrode. Actually the electrodes measure a given fraction of the specific resistance known as the cell constant C

C Measured resistance, Rm Specific resistance, Rs

The reciprocal of resistance is conductance. It measures the ability to conduct a current and is expressed in reciprocal of ohms i.e mhos. In water analysis generally micromhos is used. Knowing the cell constant the measured conductance is converted to the specific conductance or conductivity, Ks, as the reciprocal of the specific resistance. Ks= 1/Rs = C/R m The term conductivity is preferred and usually reported in micromhos per centimetre (µ mhos/cm)Freshly made distilled water has a conductivity of 0.5 to 2 .0 µ mhos/cm that increases after some days due to the absorption of CO2 from atmosphere.The conductivity of potable waters varies generally from 50 to 1500 µ mhos/cm. The conductivity of municipal waste waters may be near to that of the potable water. However 10000 µ mhos/cm.Measurement of conductivity with lesser accuracy than laboratory analysis is done continuously by the field recorders. These automatic recorders give idea about any sudden drastic change in the quality of raw water or the waste water, so that required precautions may be taken.Actually the total dissolved solids in water can be estimated by measuring itsconductivity and multiplying it by an empirical factor. This factor varies from 0.55 to 0.9 depending upon the soluble components of water and the temperature. This factor can be obtained for a system by observing the conductivity and the dissolved solids and then it can be used for continuous monitoring.

4.3 Apparatus

4.3.1 Conductivity meter:

This is an instrument consisting of a source of alternating current, a Wheatstone bridge, a null indicator and a conductivity cell. Generally an instrument capable of measuring conductivity with an accuracy of 1 % or 1 mhos/cm is used.

4.3.2 Conductivity Cell:

Platinum-electrode type conductivity cells containing platinized electrodes are used depending upon the expected range of conductivity. Non platinum-electrode type conductivity cells containing electrodes constructed from durable metals like stainless steel are used for continuous monitoring systems.

4.4 Reagents

Conductivity water: Pass distilled water through a mixed bed deionizer and discard first liter. Conductivity should be less than 1 µ mhos/cm mg.
(b) Standard Potassium Chloride Solution (KCl, 0.01M), Dissolve 745.6 of anhydrous KCl in conductivity water and dilute to 1000 ml at 25oC. This the tandard reference solution having a conductivity of 1413 µmhos/cm at 25oC,useful for the cell constants between 1 and 2.

4.5 Procedure

(i) Determination of Cell Constant Wash the conductivity cell with 0.01 M KCl solution. Adjust the temperature of the standar d KCl at 25± 0.1oC. Measure resistance of the KCL and note the temperature. The Cell Constant, C = (0.001413) (RKCL) [1+0.0191(t-25)]
(ii) Conductivity Measurement Rinse cell with the sample. Adjust temperature of the sample to 25± 0.1oC. Measure sample resistance or conductivity and the temperature If the temperature deviates from 25oC the corrected conductivity shall be as follows

K = (Km) C (1+0.019(t-25) Km is the measured conductivity at toC.

4.6 OBSERVATIONS AND CALCULATION

Water sample no.	Temperature	Electrical conductivity μ mhos / cm	Total dissolved solids

4.7 Result

The electrical conductivity of the given water sample is .... μ mhos/ cm

4.8 Probing Further Questions

1. How are conductivity and TDS related?
2. What causes conductivity in water?

LAB-4 Total Dissolved Solids

5.1 Objective

To determine the total dissolved solids of given water sample

5.2 Theory

Sewage contains 99.9% water and only 0.1% solids but the nuisance caused by them is considerable, as they are highly putrescible (readily degradable) and therefore require proper treatment before disposal. The solids present in sewage may be classified as suspended and dissolved solids which may further be subdivided into volatile and non volatile solids. The volatile matter is organic matter. Quantification of volatile or organic fraction of solid which is putrescible is necessary as this constitutes the load on biological treatment units or oxygen resources of a stream when sewage is disposed of in a river. The dissolved solid may be inorganic also and the inorganic fraction is considered when sewage is used for land irrigation or when reuse of sewage is done for any other purpose. The measurement of total dissolved solids in water can be done in similar way, by taking the sample of water, in place of sewage.

5.3 Apparatus

(i) Evaporating dishes
(ii) Drying oven
(iii) Standard filter paper
(iv) Digital weighing balance (microgram)
(v) Conical flask
(vi) Measuring cylinder

5.4 Procedure

1. Take 50 ml of well mixed sewage sample in a measuring cylinder. Have four folds of the standard filter paper and fix it on the funnel placed over a conical flask.
2. Pour the sewage gently on the funnel and allow it to slowly filter down through the funnel shaped filter paper.
3. Pour it intermittently so that the filtrate is only sewage containing dissolved solids and the suspended impurities are filtered out.
4. Transfer filtrate to a weighed evaporating dish (weight say A mg) and evaporate to dryness in the drying oven.
5. Dry evaporated sample for 1 hr in an oven at 180°C and cool it. Weight it say as B mg, and calculate the dissolved.

5.5 Calculations

Total Dissolved Solids in mg/litre = (A–B) × 1000 50 (volume of sample in ml)

The total dissolved solids give an idea about the organic and inorganic matter present in the 18

sewage in dissolved form. Organic matter is volatile and can be determined by igniting the Drying oven residue at higher temperature at 550 °C. Even the total dissolved solids give a fair idea about the organic matter and the anticipated treatment of the wastewater. Treatment means to satisfy the BOD. BOD can be satisfied aerobically or anaerobically. Aerobic treatment is better as it produces less harmful end products but it is generally costly. So depending upon the foulness (organic solid matter) and the funds available the selection of process is done.

5.6 Result

The total dissolved solids in the given sewage sample are .....mg/L which shows that.....

5.7 Probing Further Questions

1. Fine suspended solid in water is removed by which method?
2. As per IS Code what is the acceptable TDS value?

LAB-5a Alkalinity

6.1 Objective

To determine the amount of the following types of alkalinity present in the given samples: a. Hydroxide alkalinity
b. Carbonate alkalinity
c. Bicarbonate alkalinity
d. Hydroxide–Carbonate alkalinity
e. Carbonate–Bicarbonate alkalinity

6.2 Principle

The alkalinity of water is a measure of its capacity to neutralize acids. It is primarily due to salts of weak acids, although weak or strong bases may also contribute. Alkalinity is usually imparted by bicarbonate, carbonate and hydroxide. It is measured volumetrically by titration with 0.02 N sulphuric acid and is reported in terms of CaCO3 equivalent. For samples whose initial pH is above 8.3, the titration is conducted in two steps. In the first step, the titration is conducted until the pH is lowered to 8.2, the point at which phenolphthalein indicator turns from pink to colourless. This value corresponds to the points for conversion of carbonate to bicarbonate ion. The second phase of titration is conducted until the pH is lowered to 4.5, corresponds to methyl orange end point, which corresponds to the equivalence points for the conversion of bicarbonate ion to carbonic acid.

6.3 Apparatus

1. Burette
2. Erlenmeyer flask
3. Pipettes

6.4 Reagents

1. Carbon dioxide free distilled water.
2. Phenolphthalein indicator.
3. Methyl orange indicator.
4. 0.1 N sodium thiosulphate solution
5. 0.02 N sulphuric acid.

6.5 Reagents preparation

1. 0.02 N standard sulphuric acid: Prepare stock solution approximately0.1 N by diluting 2.5 mL concentrated sulphuric acid to 1 litre. Dilute 200 mL of the 0.1 N stock solution to 1 litre CO2 free distilled water. Standardise the 0.02 N acid against a 0.02 N sodium carbonate solution which has been prepared by dissolving 1.06 g anhydrous Na2CO3 and diluting to the mark of a 1 litre volumetric flask.
2. Methyl orange indicator: Dissolve 500 mg methyl orange powder indistilled water and dilute it to 1 litre. Keep the solution in dark or in an amber coloured bottle.
3. Phenolphthalein indicator: Dissolve 5 g phenolphthalein in 500mL ethylalcohol and add 500 mL distilled water. Then add 0.02 N sodium hydroxide drop-wise until a faint-pink colour appears.
4. Sodium thiosulphate 0.1 N: Dissolve 25 g Na2S2O3.5H2O and dilute to 1litre.

6.6 Procedure

1. Pipette 50 mL of sample into a clean Erlenmeyer flask (V).
2. Add one drop of sodium thiosulphate solution, if residual chlorine is present.
3. Add two drops of phenolphthalein indicator; if the pH is above 8.3, colour of solution becomes pink.
4. Titrate against standard sulphuric acid in the burette, till the colour just disappears. Note down the volume (V1).
5. Then add two drops of methyl orange indicator, the colour turns yellow.
6. Again titrate against acid, until the colour turns to orange yellow. Note down the total volume (V2).

6.7 Observation

0.02 N H2SO4 x sample (Methylorange/phenolphthalein indicator)

Description of sample	Trial no.	Burette reading (phenolphthalein indicator)		Volume of acid used V_1	Burette reading (methyl orange indicator)		Volume of acid used V_1
		Initial	Final		Initial	Final	

6.8 Calculation

1. Phenolphthalein alkalinity (P) as mg/L CaCO3 = V1 x 1000 mL of sample
   2. Total alkalinity (T) as mg/L CaCO3 =
   V2 x 1000 mL of sample The types of alkalinities present in the samples are calculated using

Result of titration	Hydroxide alkalinity as $CaCO_3$	Carbonate alkalinity as $CaCO_3$	Bicarbonate alkalinity as $CaCO_3$
P = O	O	O	T
P < ½T	O	2P	T – 2P
P = ½T	O	2P	O
P > ½T	2P – T	2 (T – P)	O
P = T	T	O	O

the equations given in the following table and the results are tabulated.

6.9 Result

The alkalinity of given water sample is. . . . . .

6.10 Probing Further Questions

1. What is meant by alkalinity?
2. An alkalinity test measures the level of?

Lab 5b - Acidity

7.1 Objective

To determine the acidity of the given sample of water.

7.2 Principle

Acidity of water is its quantitative capacity to neutralise a strong baseto a designated pH. Strong minerals acids, weak acids such as carbonic and acetic and hydrolysing salts such as ferric and aluminium sulphates may contribute to the measured acidity. According to the method of determination, acidity is important because acid contributes to corrosiveness and influences certain chemical and biological processes. It is the measure of the amount of base required to neutralise a given sample to the specific pH. Hydrogen ions present in a sample as a result of dissociation or hydrolysis of solutes are neutralised by titration with standard alkali. The acidity thus depends upon the end point pH or indicator used. Dissolved CO2 is usually the major acidity component of unpolluted surface water. In the sample, containing only carbon dioxide-bicarbonatecarbonate, titration to pH 8.3 at 25°C corresponds to stoichiometric neutralisation of carbonic acid to carbonate. Since the colour change of phenolphthalein indicator is close to pH 8.3, this value is accepted as a standard end point for the titration of total acidity. For more complex mixture or buffered solution fixed end point of pH 3.7 and pH 8.3 are used. Thus, for standard determination of acidity of wastewater and natural water, methyl orange acidity (pH 3.7) and phenolphthalein acidity (pH 8.3) are used. Thus, in determining the acidity of the sample the volumes of standard alkali required to bring about colour change at pH 8.3 and at pH 3.7 are determined.

7.3 Apparatus

1. Burette
2. Pipette
3. Erlenmeyer flasks
4. Indicator solutions

7.4 Reagents

1. CO2 free water
2. Standard NaOH solution 0.02N
3. Methyl orange indicator solution
4. Phenolphthalein indicator solution
5. Sodium thiosulphate 0.1 N.
6. NaOH solution 0.02 N: Dissolve 4 g NaOH in 1 litre water. This gives 0.1N NaOHsolution. Take 200 ml of this 0.1 N solution and make it up to 1 litre to obtain 0.02 N NaOHsolution.

7. Methyl orange indicator: Dissolve 500 mg methyl orange powder indistilled water and dilute it to 1 litre.

8. Phenolphthalein indicator: Dissolve 5 g phenolphthalein disodium saltin distilled water and dilute to 1 litre. 9. Sodium thiosulphate 0.1 N: Dissolve 25 g Na2S2O3.5H2O and dilute to 1litre distilled water.

7.5 Procedure

1. 25 mL of sample is pipette into Erlenmeyer flask.
2. If free residual chlorine is present, 0.05 mL (1 drop) of 0.1 N thiosulphatesolution is added.
3. 2 drops of methyl orange indicator is added.
4. These contents are titrated against 0.02 N hydroxide solution. The end point is noted when colour change from orange red to yellow.
5. Then two drops of phenolphthalein indicator is added and titration continued till a pink colour just develops. The volumes of the titrant used are noted down.

7.6 Observation

0.02 N NaOH × Sample (Methyl orange/phenolphthalein indicator)

Description of sample	Trial no.	Burette reading		Volume of NaOH used A
		Initial	Final	

7.7 Calculation

Acidity in mg/L as CaCO3 =A x B x 50,000 V where,
A = mL of NaOH titrant
B = normality of NaOH
V = mL of the sample.

7.8 Result

The acidity of given water sample is .....

7.9 Probing Further Questions

1. What are the units of acidity
2. What is the pH of citric acid?

LAB-6 Chloride

8.1 Objective

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

8.2 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.

8.3 Apparatus

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

8.4 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. **Potassium chromate indicator**: Dissolve 50 g potassium chromate(K2Cr2O4) in a little distilled water. Add silver nitrate solution until a definite red precipitate is formed. Let stand for 12 hours, filter and dilute the filtrate to 1 litre with distilled water.
6. **Standard silver nitrate solution 0.0141 N**: Dissolve 2.395 g AgNO3indistilled water and dilute to 1 litre. Standardise against 0.0141 N NaCl. Store in a brown bottle; 1 mL = 500 µg Cl2.
7. **Standard sodium chloride 0.0141N**: Dissolve 824.1 mg NaCl (dried at140°C) in chloride free water and dilute to 1 litre. 1mL = 500 µg Cl2 .
8. **Aluminium hydroxide suspension**: Dissolve 125 g aluminiumpotassium sulphate in 1 litre water. Warm to 60°C and add 55 mL concentrated NH4OH slowly with stirring. Let stand for 1 hour, transfer the mixture to a large bottle. When freshly prepared the suspension occupies a volume of approximately 1 litre.

8.5 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 (V1).
6. Repeat the procedure for blank and note down the volume (V2).

8.6 Observations

Water sample vs Silver nitrate (0.0141 N)
(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					

V =

V_1 =

V_2 =

N =

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

$$= \frac{(V_1 - V_2) \times 500}{V} = \ldots\ldots\ldots \text{mg/L}$$

8.7 Result

The amount of chloride in the given water sample is .....

8.8 Probing Further Questions

1.How excess chloride can corrode concrete?
2.What is the permissible limit of chloride in drinking water?

LAB-7 Iron

9.1 Objective

To determine the quantity of iron present in the given sample of water

9.2 Principle

Iron is usually present in natural water and is not objectionable, if concentration is less than 0.3 ppm. It may be in true solution in colloidal state that may be peptized by organic matter, in the inorganic and organic iron complexes, or in relatively coarse suspended particles. It may be either ferrous or ferric, suspended or filterable. Iron exists in soils and minerals mainly as insoluble ferric oxide and iron sulphide (pyrite). It occurs in some areas, also as ferrous carbonate (siderite), which is very slightly soluble. The phenanthroline method is the preferred standard procedure for the measurement of iron in water except when phosphate or heavy metal interferences are present. The method depends upon the fact that 1, 10-phenanthroline combine with Fe++ to form an orange-red complex. Its colour conforms to Beer's law and is readily measured by visual or photometric comparison. Small concentration of iron can be most satisfactorily determined by colorimetric analysis. It is also based on Beer's law. By measuring the intensities of transmitted and incident light through a coloured solution and knowing its optical density or transmission, we can prepare a calibration curve and subsequent concentration can be read.

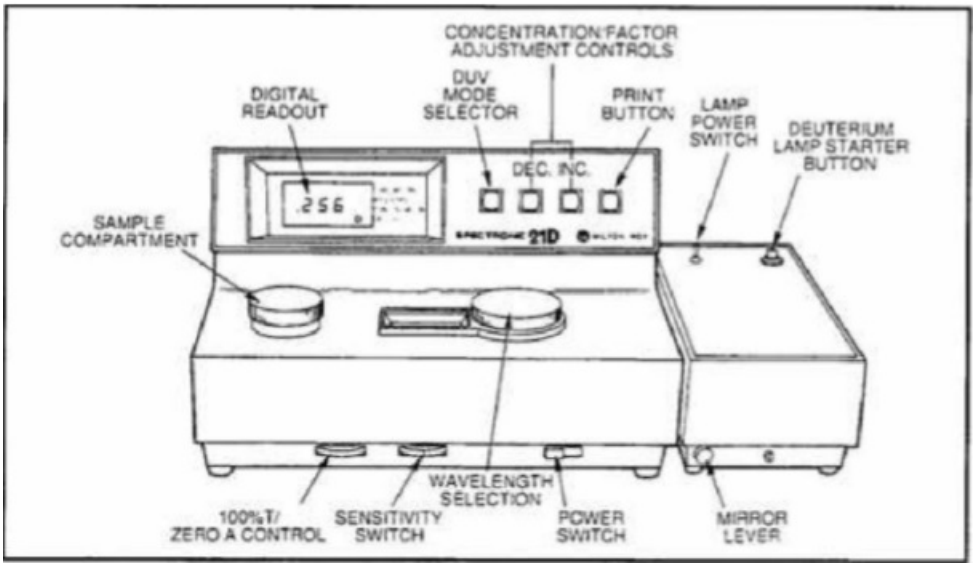
9.3 Apparatus

1. Colorimetric equipment; one of the following is required:
(a) Spectrophotometer, for use at 510 nm, providing a light path of 1 cm or longer.
(b) Nessler tubes, matched, 100 mL, tall form.

2. Glassware like conical flasks, pipettes and glass beads.

9.4 Reagents

1. Hydrochloric acid
2. Hydroxylamine solution
3. Ammonium acetate buffer solution
4. Sodium acetate solution
5. Phenanthroline solution
6. Stock iron solution
7. Standard iron solution (1 mL = 10 µg Fe



9.5 Procedure

1. Pipette 10, 20, 30 and 50 mL. Standard iron solution into 100 mL conical flasks.
2. Add 1 mL hydroxylamine solution and 1 mL sodium acetate solution to each flask.
3. Dilute each to about 75 mL with distilled water.
4. Add 10 mL phenanthroline solution to each flask.
5. Make up the contents of each flask exactly to 100mL by adding distilled water and left stand for 10 minutes.
6. Take 50 mL distilled water in another conical flask.
7. Repeat steps 2 to 5 described above.
8. Measure the absorbance of each solution in a spectrophotometer at 508 nm against the reference blank prepared by treating distilled water as described in steps 6 and 7. Prepare a calibration graph taking meter reading on y-axis and concentration of iron on x-axis.
9. For visual comparison, pour the solution in 100 mL tall form Nessler tubes and keep them in a stand.
10. Mix the sample thoroughly and measure 50 mL into a conical flask.
11. Add 2 mL conc. hydrochloric acid (HCl) and 1mL hydroxylamine solution. Add a few glass beads and heat to boiling. To ensure dissolution of all the iron, continue boiling until the volume is reduced to 15 to 20 mL.
12. Cool the flask to room temperature and transfer the solution to a 100 mL Nessler tube.
13. Add 10 mL ammonium acetate buffer solution and 2 mL Phenanthroline solution and dilute to the 100 mL mark with distilled water.
14. Mix thoroughly and allow at least 10 to 15 minutes for maximum colour development.
15. Measure the absorbance of the solution in a 1cm cell in spectrophotometer at 508 nm.
16. Read off the conc. of iron (mg Fe) from the calibration graph for the corresponding meter reading.
17. For visual comparison, match the colour of the sample with that of the standard prepared in steps 1 to 7 above.
18. The matching colour standard will give the concentration of iron in the sample (µg Fe).

9.6 Observation

Standard iron solution in mL	Iron content in µg	Absorbance

Sample no.	Absorbance	Iron content from graph in µg	Iron as Fe in mg/L

9.7 Result

Iron (Fe) in mg/L = µg Fe/mL of sample = ... mg/L

9.8 Probing Further Questions

1. Ironreacts with which chemical to form an orange-red complex ion?
2. Ferric iron combines with thiocyanate ions to form whichcoloured ferric thiocyanate.

Lab 8 – Dissolved Oxygen

10.1 Objective

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.

10.2 Principle

Dissolved Oxygen (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.

10.3 Apparatus

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

10.4 Reagents

1. Manganous sulphate solution (MnSO4.4H2O)
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)

10.5 Preparation of reagents

1. **Manganous sulphate solution:** Dissolve 480 g MnSO4.4H2O, 400 gMnSO2.2H2O or 364 g MnSO4.H2O in distilled water, filter and dilute to 1 litre.
2. **Alkali-iodide-azide reagent:** Dissolve 500 g NaOH or 700 g KOH and135 g NaI or 150 g KI in distilled water and dilute to 1 litre. Add 10 g sodium azide (NaN3) dissolved in 40 mL

distilled water. The reagent should not give colour with starch when diluted and acidified.
3. **Sulphuric acid concentrated:** 1mL is equivalent to about 3 mL alkali-iodide-azide reagent.
4. **Standard sodium thiosulphate 0.025 N:** Dissolve 6.205 g sodiumthiosulphate (Na2S2O3.5H2O) in freshly boiled and cooled distilled water and dilute to 1 litre. Preserve by adding 5 mL chloroform or 0.4 g NaOH/L or 4 g borax and 5 10 mg HgI2/L. Standardise this with 0.025 N potassium dichromate solution which is prepared by dissolving 1.226 g potassium dichromate in distilled water and diluted to 1 litre.
5. **Standard potassium dichromate solution 0.025 N**: A solution ofpotassium dichromate equivalent to 0.025 N sodium thiosulphate contains 1.226 g/L K2Cr2O7. Dry K2Cr2O7 at 103°C for 2 hrs before making the solution.
6. **Standardisation of 0.025 N sodium thiosulphatesolution:** Dissolveapproximately 2 g KI in an Erlenmeyer flask with 100 to 150 mL distilled water. Add 10 mL of H2SO4, followed by exactly 20 mL, 0.1 N potassium dichromate solution. Place in the dark for 5 minutes, dilute to approximately 400 mL and titrate with 0.025 N sodium thiosulphate solution, adding starch towards the end of titration. Exactly 20 ml 0.025 N thiosulphate will be consumed at the end of the titration. Otherwise, the thiosulphate solution should be suitably corrected.
7. **Starch Indicator:** Add cold water suspension of 5 g soluble starch toapproximately 800 mL boiling water with stirring. Dilute to 1 litre, allow to boil for a few minutes and let settle overnight. Use supernatant liquor. Preserve with 1.25 g salicylic acid/1 litre or by the addition of a few drops of toluene

10.6 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. Restopper and mix by gentle inversion until dissolution is complete.
6. Measure out 203 mL of the solution from the bottle to an Erlenmeyer flask. As 2 mL each of manganese sulphate and azide reagent have been added, the proportionate quantity of yellow solution corresponds to 200 mL of sample is = 200×300 = 203mL 300–4
7. Titrate with 0.025 N sodium thiosulphatesolution 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.

10.7 Observation

Sample x Standard sodium thiosulphate solution (0.025N) (Starch indicator)

Description of sample	Trial no.	Volume of sample (mL)	Burette reading		Volume of titrant mL	D.O. in mg/L
			Initial	Final		
Sample I						
Sample II						
Sample III						

10.8 Result

The amount of dissolved oxygen of given water sample is .....

10.9 Probing Further Questions

1. High amount of Dissolved Oxygen in a lake indicate?
2. According to drinking water specification provided by IS 10500 (2012), what should be range of DO of drinking water?

LAB-9 Nitrate

11.1 Objective

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

11.2 Principle

The reaction with the nitrate and brucine produces yellow colour that can be used for the colorimetric estimation of nitrate. The intensity of colour is measured at 410 nm. The method is recommended only for concentration of 0.1– 2.0 mg/L NO-3—N. All strong oxidising and reducing agent interfere. Sodium arsenite is used to eliminate interference by residual chlorine; sulphanilic acid eliminates the interferences by NO2-N and chloride interference is masked by addition of excess NaCl. High concentration of organic matter also may interfere in the determination.

11.3 Apparatus

1. Spectrophotometer
2. Water bath
3. Reaction tubes
4. Cool water bath

11.4 Reagents

1. Stock nitrate solution
2. Standard nitrate solution
3. Sodium arsenate solution
4. Brucine-sulphanilic acid solution
5. Sulphuric acid solution
6. Sodium chloride solution

11.5 Procedure

1. Nitrate standards are prepared in the range 0.1–1.0 mg/LN diluting 1.00, 2.00, 4.00, 7.00 and 10.0 mL standard nitrate solution to 10 mL with distilled water.
2. If residual chlorine is present 1 drop of sodium arsenite solution is added for each 0.1 mg Cl2 and mixed.
3. Set up a series of reaction tubes in test tube stand. Add 10 mL sample or a portion diluted to 10 mL to the reaction tubes.
4. Place the stand in a cool water bath and add 2 mL NaCl solution and mix well.
5. Add 10 mL H2SO4 solution and again mix well and allow cooling.
6. The stand is then placed in a cool water bath and add 0.5 ml brucinesulphanilic acid reagent. Swirl the tubes and mix well and place the tubes in boiling water bath at temperature 95°C.
7. After 20 minutes, remove the samples and immerse in cool water bath.
8. The sample are then poured into the dry tubes of spectrophotometer and read the standards and sample against the reagent blank at 410 nm.
9. Prepare a standard curve for absorbance value of standards (minus the blank) against the concentration of NO-3N.
10. Read the concentration of NO-3N in the sample from the known value of absorbance.

11.6 Calculation

Nitrate N in mg/L µg NO3- – N =
mL of sample
NO3 in mg/L = mg/L nitrate N × 4.43.

11.7 Observation

The observation are presented in Tables A and B respectively.

Table A: Observation for calibration
Table B:

Stock nitrate solution in mL	Nitrate	Absorbance

Sample no.	Absorbance	Nitrate nitrogen in µg from graph	Nitrate nitrogen in mg

11.8 Result

The amount of nitrate Nitrogen in given water sample is .....

11.9 Probing Further Questions

1. How does nitrogen affect water quality?
2. How do nitrates get into water?

LAB-10 Optimum Coagulant Dosage

12.1 Objective

To determine the optimum coagulant dosage for clarifying the given sample of water by using alum as the coagulant and performing the jar test experiment

12.2 Principle

Coagulants are used in water treatment plants
(i) to remove natural suspended and colloidal matter,
(ii) to remove material which do not settle in plain sedimentation, and
(iii) to assist in filtration.
Alum [Al2(SO4)3. 18H2O] is the most widely used coagulant. When alum solution is added to water, the molecules dissociate to yield SO2–4and Al3+. The +ve species combine with negatively charged colloidal to neutralize part of the charge on the colloidal particle. Thus, agglomeration takes place. Coagulation is a quite complex phenomenon and the coagulant should be distributed uniformly throughout the solution. A flash mix accomplishes this.Jar test is simple device used to determine this optimum coagulant dose required. The jar test, device consists of a number of stirrers (4 to 6) provided with paddles. The paddles can be rotated with varying speed with the help of a motor and regulator. Samples will be taken in jars or beakers and varying dose of coagulant will be added simultaneously to all the jars. The paddles will be rotated at 100 rpm for 1 minute and at 40 rpm for 20 to 30 minutes, corresponding to the flash mixing and slow mixing in the flocculator of the treatment plant. After 30 minutes settling, supernatant will be taken carefully from all the jars to measure turbidity. The dose, which gives the least turbidity,is taken as the optimum coagulant dose.

12.3 Apparatus

1. Jar Test Apparatus
2. Glass Beakers
3. Pipette
4. Nephelometer
5. pH meter

12.4 Reagents

1. Alum solution (1mL containing 10 mg of alum)
2. Lime
3. Acid/alkali

12.5 Procedure

1. Take 1-litre beakers and fill them with sample up to the mark.
2. Keep each beaker below each paddle and lower the paddles, such that each one is about 1cm above the bottom.
3. Find the pH of the sample and adjust it to 6 to 8.5.
4. Pipette 1, 2, 3, 4, 5, 6 mL of the alum solution into the test samples.
5. Immediately run the paddles at 100 rpm for 1 minute.
6. Reduce the speed to 30–40 rpm and run at this rate for 30 minutes.
7. Stop the machine, lift out the paddles and allow to settle for 30 minutes.
8. Find the residual turbidity of the supernatant using nephelometer.
9. Plot a graph with alum dosage along x-axis and turbidity along y-axis.
10.The dosage of alum, which represents least turbidity, gives Optimum Coagulant Dosage (O.C.D.). 11.Repeat steps 1–10 with higher dose of alum, if necessary

12.6 Observation

Trial No.	*Alum Dosage in mg/L*	*Turbidity in NTU*

12.7 Result

Optimum coagulant dosage = ...

12.8 Probing Further Questions

1. A jar test simulates which process.
2. Stirring speed is reduced to promote which action

LAB-11 Chlorine Demand

13.1 Objective

To determine the chlorine demand

13.2 Theory

Most water treatment plants are required to disinfect the water, a process used to kill harmful bacteria. The most frequently used method of disinfection is the addition of chlorine. Here, we will briefly introduce three terms used during chlorination - chlorine dose, chlorine demand, and chlorine residual. These three characteristics are related to each other using the following equation: (Chlorine demand) = (Chlorine dose) - (Chlorine residual) The amount of chlorine added to the water is known as the chlorine dose. This is a measured quantity chosen by the operator and introduced into the water using a chlorinator or hypochlorinator.As the chlorine reacts with bacteria and chemicals in the water, some of the chlorine is used up. The amount of chlorine used up by reacting with substances in the water is known as the chlorine demand. If nothing reacts with the chlorine (as would be the case in distilled water), then the chlorine demand is zero. However, in most cases the operator should count on some of the chlorine dose being used up when it reacts with substances in the water. The amount of chlorine remaining in the water after some of the chlorine reacts with substances in the water is known as the chlorine residual. This lab introduces a test which can be used to calculate the chlorine residual. The chlorine residual is the most important of these three values - dose, demand, and residual - because it represents the actual amount of chlorine remaining in the water to act as a disinfect The test for chlorine residual is performed frequently at most water treatment plants. Since regulations require a certain level of chlorine in water at the far ends of the distribution system, operators should be sure to test the chlorine residual in the distribution system as well as in the clear well.

13.3 Introduction to Testing Procedures

The DPD Colorimetric Method introduced in this lab is one of several procedures which can be used to test for chlorine residual. This method requires compensation for color and turbidity and can detect chlorine concentrations only as low as 10 ug as Cl2/L. Standard Methods introduces several other procedures and explains which procedures are most effective under a variety of circumstances. In every case, remember that chlorine is a relatively volatile substance and that samples should be tested as soon as possible after the water is collected.The chemistry involved in the DPD Colorimetric Method is relatively simple. The buffer lowers the pH of the sample to 4 or less. In this pH range, chlorine in the water is able to react with the added potassium iodide, replacing the iodine which is released into the solution as shown below: 2KI + Cl2 2KCl + I2 When free iodine becomes present in the water, the indicator makes the solution change to a red color, with the intensity of the color equivalent to the amount of chlorine found in the solution.

13.4 Apparatus

This procedure requires a piece of colorimetric equipment, some glassware, and Titration equipment. The colorimetric equipment must be one of the following:
1. Spectrophotometer, for use at a wavelength of 515 nm and providing a light path of 1 cm or longer.
2. Filter photometer, equipped with a filter having maximum transmission in the wavelength range of 490 to 530 nm and providing a light path of 1 cm or longer.

13.5 Reagents

1. Standard potassium permanganate solutions
2. Phosphate buffer solution
3. N,N-Diethyl-p-phenylenediamine (DPD) indicator solution
4. Standard ferrous ammonium sulfate (FAS) titrant
5. Potassium iodide (KI) crystals
6. Chlorine-demand-free water

13.6 Procedure

1. Calibrate the photometric equipment using the following procedure. Note that this procedure uses potassium permanganate solutions. You can use chlorine solutions to calibrate the equipment by following the procedure in StandardMethods on pages 463 to 4-64.
1. Set 100%T on the spectrophotometer or filter photometer using a distilled water blank, in accordance with manufacturer's instructions. (Prepare the distilled water blank in the same manner as you prepare the sample for testing.)
2. Prepare a series of potassium permanganate standards covering the equivalent chlorine range of 0.05 to 4 mg/L. (The procedure for producing the standards is explained in Standard Methods on page 4-64.)
3. Label empty flasks for each standard. Place 5 mL of phosphate buffer and 5 mL of DPD indicator reagent in each labelled flask.
4. Add 100 mL of each prepared potassium permanganate standard solution to the appropriate flask and mix thoroughly.
5. Fill a photometer or colorimeter cell with the solution in each flask and read each standard at a wavelength of 515 nm.
6. In the data section, plot a standard curve of mg/L equivalent chlorine versus %T.

7. Return the cell contents to the appropriate flask and titrate with FAS titrant as a check on any absorption of permanganate by distilled water.
2. Measure the chlorine content of the sample.
a) Pipette 0.5 mL of phosphate buffer solution into an empty test tube.
b) Add 0.5 mL of DPD indicator solution to the test tube.
c) Add 10 mL of sample water and read the color immediately. Use the standard curve to determine the amount of chlorine in the sample. Record this in the Data section as Reading A.
d) Continue by adding one very small crystal of KI (about 0.1 mg) to the test tube and mixing. Read the color immediately. Use the standard curve to determine the amount of chlorine in the sample. Record this in the Data section as Reading B.
e) Continue by adding several crystals of KI (about 0.1 g) to the test tube and mixing. Let the solution stand for about two minutes to allow color to develop, then read the color. Use the standard curve to determine the amount of chlorine in the sample. Record this in the Data

section as Reading C.
f) Place a very small crystal of KI (about 0.1 mg) in a clean test tube. Add 10 mL of the sample and mix. In a separate tube, add 0.5 mL of the phosphate buffer solution and and 0.5 mL of the DPD indicator solution and mix. Add the contents of the second tube to the first tube and mix. Read the color immediately. Use the standard curve to determine the amount of chlorine in the sample. Record this in the Data section as Reading N.

3. Calculate the amount of each type of chlorine using the calculation methods listed in Table 2 in the Data section. For example, let's consider our calculations if the readings were A = 1.0 mg/L, B = 1.3 mg/L, C = 2.7 mg/L, and N = 1.9 mg/L. First, the amount of free chlorine was shown by Reading A to be 1.0 mg/L. The amount of monochloramine is calculated as: B - A = 1.3 mg/L - 1.0 mg/L = 0.3 mg/L So the concentration of monochloramine is 0.3 mg/L. Since N is more than 0, the dichloramine concentration is calculated as: C - N = 2.7 mg/L - 1.9 mg/L = 0.8 mg/L So the concentration of dichloramine is 0.8 mg/L. Finally, since N is more than 0 and there are monochloramines present, the amount of trichloramine is calculated as follows: 2(N - B) = 2(1.9 mg/L - 1.3 mg/L) = 1.2mg/L So the concentration of trichloramine is 1.2 mg/L.

13.7 Observations

13.7.1 Table 1

Reading	**%T**	**mg/L chlorine**
A		
B		
C		
N		

13.7.2 Table 2

Type of Chlorine	**Calculation Method**	**mg/L**
Free Chlorine	A	
Monochloramine	B - A	
Dichloramine	If N=0, then C - B; If N>0, then C - N	
Trichloramine	If N=0, then 0 mg/L; If N>0 and (B - A) = 0, then 2(N - A) If N>0 and (B - A)>0, then 2(N - B)	

13.8 Result

The chlorine demand of given water sample is ..... .

13.9 Probing Further Questions

1. At what pH, chlorine exists as molecular chlorine?
2. By how many times the hypochlorous acid is effective as hypochlorite ions?

LAB-12 Biological Oxygen Demand

14.1 Objective

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

14.2 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 (D1) in the other set ofbottles will be determined immediately. At the end of 5 days, the dissolved oxygen content (D2) in the incubated set of bottles is determined. Then m.g./L B.O.D. (D – D)2 P where,

P = decimal fraction of sample used. D1= dissolved oxygen of diluted sample (mg/L), immediately after preparation. D2= 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.

14.3 Apparatus

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

14.4 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.

14.5 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. Highly alkaline or acidic samples should be neutralised to pH 7. 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.
5. Take the sample in the required concentrations. The following concentrations are suggested:
6. 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.
7. Mix the contents in the measuring cylinder.
8. Add this solution into two B.O.D. bottles, one for incubation and the other for determination of initial dissolved oxygen in the mixture.
9. Prepare in the same manner for other concentrations and for all the other samples
10. 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.
11. 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.
12. Determine the initial dissolved oxygen contents in the other set of bottles and note down the results.
13. Determine the dissolved oxygen content in the incubated bottles at the end of 5 days and note down the results.
14. Calculate the B.O.D. of the given sample.
Note: The procedure for determining the dissolved oxygen content is sameas described in the experiment under “Determination of dissolved oxygen”.

14.6 Observation

Sample No. Or *Description*	*Concentration*	*Dissolved oxygen content mg/L*				*B.O.D. mg/L (5 days 20^0C)*
		Initial D1		*Initial D2*		
		Bottle No.	*D.O. value*	*Bottle No.*	*D.O. value*	

14.7 Result

The bod of given water sample is ……..

14.8 Probing Further Questions

1. What is the temperature required during the incubation period?
2. What is the colour change during titration with Na2S2O3 before adding starch?

LAB-13 Chemical Oxygen Demand

15.1 Objective

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

15.2 Principle

Potassium dichromate is a powerful oxidising agent in acidic medium and is obtained in high state of purity. The reaction involved is:

where, c = 2/3n + a/6 - b/3 C.O.D. results are reported in terms of mg of oxygen. N/8 or 0.125 N solution of oxidising agent is used in the determination. Normality double the strength is used. This allows the use of larger samples. Thus, each ml of 0.25 N solution dichromate is equivalent to 2 mg of oxygen. An excess of oxidising agent is added, the excess is determined by another reducing agent such as ferrous ammonium sulphate. An indicator ferroin is used in titrating the excess dichromate against ferrous ammonium sulphate. Blanks are used also treated and titrated to get the correct value of C.O.D.

15.3 Apparatus

1. Reflux apparatus
2. Burettes
3. Pipettes

15.4 Reagents

1. Standard potassium dichromate solution 0.25N.
2. Sulphuric acid reagent.
3. Standard ferrous ammonium sulphate.
4. Ferroin indicator solution.
5. Mercuric sulphate.
6. Sulphuric acid crystals.

15.5 Preparation of reagents

1. Standard potassium dichromate solution 0.25 N: Dissolve 12.259 gK2Cr2O7primary standard grade previously dried at 103°C for 2 hours and dilute to 1 litre.
2. Sulphuric acid reagent: Concentrated H2SO4containing 22 g silversulphate per 4 kg bottle.Dissolve 22 g Ag2SO2 in 4 kg bottle and keep it for 2 days. This is the reagent.
3. Standard ferrous ammonium sulphate 0.1 N: Dissolve 39 g Fe(NH4)2(SO4)2.6H2O in distilled water. Add 20 mL conc. H2SO4 and cool and dilute to 1 litre. Standardise this against the standard dichromate solution. Dilute 10 mL standard K2Cr2O7 solution to about 100 mL.

Add 30 mL conc. H2SO4 and cool. Titrate with ferrous ammonium sulphate titrant using 2 3 drops of ferroin indicator.

15.6 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.

15.7 Observation

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

15.8 Calculation

C.O.D. mg/L = (V1 - V2) N x 8000
V where, V1 = mL ferrous ammonium sulphate used for blank V2 = mL ferrous ammonium sulphate used for sample N = normality of ferrous ammonium sulphate V = volume of sample used.

15.9 Result

The C O D of given water sample is .....

15.10 Probing Further Questions

1. How does COD affect water quality?
2. What is the COD value of drinking water?