Military Institute of Science and Technology

CE 332

ENVIRONMENTAL ENGINEERING LABORATORY MANUAL





MILITARY INSTITUTE OF SCIENCE AND TECHNOLOGY

Department of Civil Engineering

Course Number: CE 332

Course Title: Environmental Engineering Laboratory

Table of Contents

Exp. No.	Name of the Experiment	Page No.
	Introduction & General Instruction	3
	Water Sampling Methods for Lab Analysis	5
1.	Determination of pH of Water	10
2.	Determination of Color of Water	12
3.	Determination of Turbidity of Water	14
4.	Determination of Total Solids, Dissolved Solids and Suspended Solids in Water	16
5.	Determination of Carbon dioxide in Water	18
6.	Determination of Alkalinity of Water	21
7.	Determination of Hardness of Water	25
8.	Determination of Chloride in Water	29
9.	Determination of Iron in Water	32
10.	Determination of Arsenic in Water	35
11.	Determination of Total Coliform and Fecal Coliform for Microbiological Water Quality Analysis	40
12.	Estimation of Organic Pollution Load through Determination of BOD and COD	44
13.	Chemical Coagulation of Water: Alum Coagulation	52
14.	Determination of Residual Chlorine and Chlorine Demand: Break Point Chlorination	57
15.	Measurement of Ambient Particulate Air Pollution Concentration: PM2.5 and PM10	61
16.	Measurement of Ambient Noise Level	65
	Appendix-A: Water Quality Standards from ECR'2023	69
	Appendix-B: Water Sample Preservation	73
	Appendix-c: Test Report Format	75

Class Schedule

Class Weeks	Scheduled Task
Week -1	Introduction & Water sample collection methods
Week -2	Exp. No.- 1-4
Week -3	Exp. No.- 5-7
Week -4	Exp. No.- 8 & 9
Week -5	Exp. No.- 10 & 11
Week -6	**Midterm Quiz and Viva** (Water sampling & Exp. No.- 1 to 11)
Week -7	Exp. No.- 12
Week -8	Exp. No.- 13
Week -9	Exp. No.- 14
Week -10	Exp. No.- 15 & 16
Week -11	**Final Quiz and Viva** (Exp. No.- 12 to 16)

Introduction & General Instruction

Environmental engineering is the branch of engineering that is concerned with protecting people from the effects of adverse environmental effects, such as pollution, as well as improving environmental quality. Environmental engineers devise solutions for wastewater management, water and air pollution control, recycling, waste disposal, and public health. The Environmental Laboratory ensures that testing capacity is available to support the public health and environmental protection objectives of the government. The Environmental Laboratory includes these technical areas: General Chemistry, Metals Chemistry, Organic Chemistry, Radiation Chemistry and Water Microbiology. In CE 332 laboratory class is designed to determine of physio-chemical and bacteriological characteristics of water and in laboratory analysis of some common physiochemical treatments.

This Lab manual was prepared with the help of "Standard Methods for the Examination of Water and Waste Water", 1995, 20th Edition, American Public Health Association, APHA; and "The Environment Conservation Rules", 2023.

Important Instructions Regarding the Lab Class

- All students must collect their own copy of lab manual (January 2025 version) from the undergraduate Environmental Engineering lab by the 1st week of the commencement of the class.
- Student **must bring this manual in every class** of this sessional; otherwise he/she will not be allowed to attend the lab class.
- Student must bring calculator, pencil, scale, A4 offset paper, graph paper etc. with him as per requirement of the schedule lab test.
- Student must come to the CE332 sessional classes with proper preparation for conducting the lab test as per schedule. The Students must read in advance the chapter(s) of this lab manual related to the test(s) to be performed.
- In this lab class the course teachers may evaluate each student's class performance during each lab test either by asking questions orally or by taking written assessment.
- All students in a class will be divided into groups by the course teachers. Each student has to do all the experiment/test along with his/her designated group.

Laboratory Report Writing Instruction

- Each experimental report **must be** submitted in the report format attached in the supplied lab manual. The student has to photocopy the pages of the report and fill up with the relevant information, calculation etc.
- The lab test report on any experiment must be submitted individually and **on due date**. 50% marks will be deducted for late submission. No report submission is accepted if late by more than a week from due date of submission.
- Students must submit the whole lab report in a transparent plastic file. The color of the file for each group will be instructed by your course teacher.
- The file should contain the followings:
    i. A top cover page for full lab report
    ii. An index page
    iii. Experiment reports
- Each experiment report also should have an individual cover page (Details of the index content and cover page contents will be discussed in the class by the course teachers).
- Repost must be neat and clean and must be written using pen.
- Graphs and other additional pages (if needed) must be placed the report in appropriate sequence as discussed in the following section.

Brief Description of the Laboratory Report

- Objective & scope of the test: A brief statement of the purpose and significance of the test should be described in brief.
- Water Sampling Information (source, location, date and time of collection etc.) should be mentioned in the report.
- Name of apparatus & reagents used to conduct the test should be listed
- Experiment Data Table: All laboratory test data should be submitted in the data table provided in the report format. Observations relating to the behavior of the materials should be included.
- Sample Calculation: Al necessary calculation should be shown in the report. All equations or formulas used should be clearly indicated. Calculations should be properly checked.
- Graph(s): If the test requirement includes drawing any graph(s), should be drawn in correct graph paper and according to the test specification.
- Result(s): all obtained results should be presented in the standard way as stated in the specification
- Discussion on obtained result(s): There should be included a brief discussion in which attention is drawn to the silent facts shown by the tables and diagrams. The test results should be compared with the standard values and conclusion should be drawn.
- Assignment questions: each test report format contains some questions related to the test. Answer to those questions should be brief and to the point. The name of the reference books or websites other than this lab manual used for getting those answers should be mentioned below that answer.

Laboratory Safety Practice

All students are strictly instructed to maintain lab responsibility and safety practice while being in the lab. Please read the "Environmental Lab Safety Manual" carefully and follow each instruction. The manual is uploaded in the departmental website.

Introduction:
Methods of Water Sampling for Lab Analysis

Water testing is carried out to meet the regulatory requirements and adhere to the safety procedures that are needed for pollutant-free water. Water quality is the physical, chemical and biological characteristics of water which are determined by the intended use of the water. Works in the area of water quality tend to be focused on water that is treated for human consumption, industrial use, or to get back in the environment.
Water quality standards are put in place to ensure the efficient use of water for a designated purpose. Water quality analysis is to measure the required parameters of water, following standard methods, to check whether they are in accordance with the standards against which compliance can be assessed.

Water quality analysis is required mainly for monitoring purpose. Some importance of such assessment includes:
1. To check whether the water quality is in compliance with the standards, and hence, suitable or not for the designated use.
2. To monitor the efficiency of a system, working for water quality maintenance
3. To check if any upgradation / change of an existing system is required and to decide what changes should take place.
4. To monitor whether water quality is in compliance with rules and regulations.

Importance of Sampling for Water Quality Analysis
Water sampling is very important for the correct outcome of water analysis. Using the correct technique and the appropriate glassware or plasticware is essential for the correct outcome. For example, metals react with glass and can be adsorbed on the glass; for this reason, water sampling for metal analysis is performed in plastic (usually polyethylene) containers. Another important factor is the correct lifetime of the parameters to be used and the preservation method. Some parameters such as metals require immediate pretreatment such as the addition of acid in the sample, and then, storage and other parameters require immediate analysis (e.g., temperature, nutrients etc.).

On-site Water Quality Analysis
The complexity of water quality as a subject is reflected in the many types of measurements of water quality indicators. The most accurate measurements of water quality are made on-site, because water exists in equilibrium with its surroundings. Measurements commonly made on-site and in direct contact with the water source in question include - temperature, pH, dissolved oxygen, conductivity, oxygen reduction potential (ORP) and turbidity..

Water Quality Analysis in Lab: Collection of water sample
More complex measurements are often made in a laboratory requiring a water sample to be collected, preserved, transported, and analyzed at another location. The process of water sampling introduces two significant problems- a common cause of error in water quality analysis is improper sampling. The results of a water quality analysis of a sample show only what is in the sample. For the results to be meaningful, the sample *must be representative* i.e., it must contain essentially the same constituents as the body of water from which it was taken.
Many water sources vary with time and with location. The measurement of interest may vary seasonally or from day to night or in response to some activity of man or natural populations of aquatic plants and animals. The measurement of interest may vary with distances from the water boundary with overlying atmosphere and underlying or confining soil. The sampler must determine if a single time and location meets the needs of the investigation, or if the water use of interest can be satisfactorily assessed by averaged values with time and/or location,

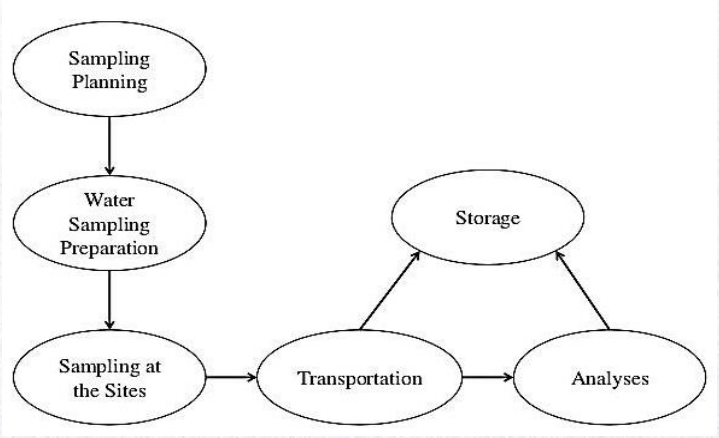
or if critical maxima and minima require individual measurements over a range of times, locations and/or events. The sample collection procedure must assure correct weighting of individual sampling times and locations where averaging is appropriate.
The second problem occurs as the sample is removed from the water source and begins to establish chemical equilibrium with its new surroundings - the sample container. Sample containers must be made of materials with minimal reactivity with substances to be measured; and pre-cleaning of sample containers is important. The water sample may dissolve part of the sample container and any residue on that container, or chemicals dissolved in the water sample may sorb onto the sample container and remain there when the water is poured out for analysis.
The collected sample should be small enough in volume to be transported conveniently and yet large enough for analytical purposes while still accurately representing the material being sampled.

Location of sampling points
The following general criteria are usually applicable:

- Sampling points should be selected such that the samples taken are representative of the different sources from which water is obtained by the public or enters the system.
- These points should include those that yield samples representative of the conditions at the most unfavourable sources or places in the supply system, particularly points of possible contamination such as unprotected sources, loops, reservoirs, low-pressure zones, ends of the system, etc.
- Sampling points should be uniformly distributed throughout a piped distribution system, taking population distribution into account; the number of sampling points should be proportional to the number of links or branches.
- The points chosen should generally yield samples that are representative of the system as a whole and of its main components. Sampling points should be located in such a way that water can be sampled from reserve tanks and reservoirs, etc.
- In systems with more than one water source, the locations of the sampling points should take account of the number of inhabitants served by each source.
- There should be at least one sampling point directly after the clean-water outlet from each treatment plant.

Requirements for Sampling:

- Meet the requirements of the sampling program.
- Handle the sample carefully so that it does not deteriorate or become contaminated or compromised before it is analyzed.
- Ensure sampling all equipment are clean and quality assured before use.
- Use sample containers that are clean and free of contaminants
- Rinse the bag/bottle at least twice with the sample water prior to filling and closing
- Fill bag/bottle as full as possible. Half-filling leaves more room for oxygen which will promote degradation of your sample.
- If sampling a body of running water, point the mouth of the bag upstream and your hands downstream to avoid contamination.
- If sampling from a water faucet, run the faucet for 1 minute before obtaining a sample.
- Make records of every sample collected and identify every bottle e.g., take notes and photographs, fill out tags, etc.
- Place the sample into appropriate, labeled containers.
- All samples must be preserved as soon as practically possible.



Water Sampling Flow Chart

Type of Surface Water Sample

Surface water sampling can be done in any of the following two methods depending on test requirements:

1. Grab sampling
2. Composite sampling

1. Grab sampling

Grab sampling can be done in two ways: i. discrete grab sampling & ii. depth-integrated grab sampling. A discrete grab sample is collected at a specific location at a certain point in any specific time. Whereas depth-integrated grab sample is collected over a part or the entire depth at a specific location.

A grab sample reflects performance only at the point in time that the sample was collected, and then only if the sample was properly collected- such as pH, dissolved oxygen, chlorine residual, nitrites and temperature of a particular point and time. If the environmental medium varies spatially or temporally e.g.in case of waste effluents, then a single grab sample is not representative and more samples need to be collected at suitable time intervals and analyzed separately.

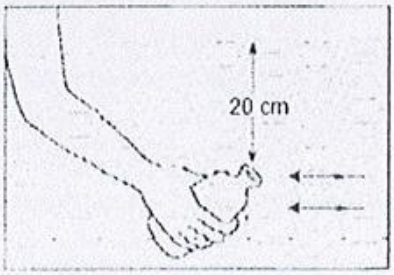
2. Composite sampling

A composite sample, also known as an integrated sample, is a sample which consists of a mixture of several individual grab samples collected at regular and specified time periods, each sample taken in proportion to the amount of flow at that time. Composite samples give a more representative sample of the characteristics of water at the plant over a longer period of time. This is particularly useful in water bodies that have a lot of chemical variability either over space or over short time periods. Composite samples are often used to reduce the cost of analyzing a large number of samples.

Composite samples are often taken using automatic sampling devices. These may be set to take a sample every 8, 12, or 24 hours, with the frequency depending on test requirements, on the size of the treatment plant, and on permit requirements.

Surface Water Sampling Techniques

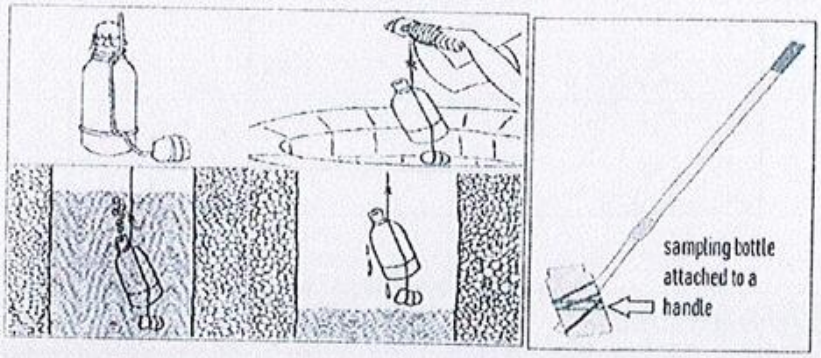
i. When the water source is accessible



- Rinse the sampling vessel with water on site 3~4 times. Care must be taken to avoid contaminating water to be sampled during rinsing.
- Submerge the sampling vessel gently (150-250 mm below the surface), fill it with the water sample and close it tightly.
- If the collected water sample may be frozen, leave some space for expansion equivalent to about 10% of the sampling vessel.

ii. When the water source is inaccessible

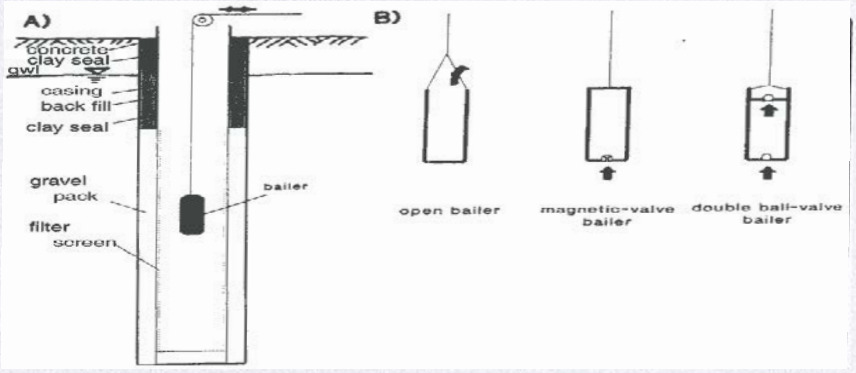
A rope attached to the bucket is often used. Scoops with adjustable shafts are convenient. Items made of synthetic resins such as polypropylene can also be used.



Groundwater (from well)

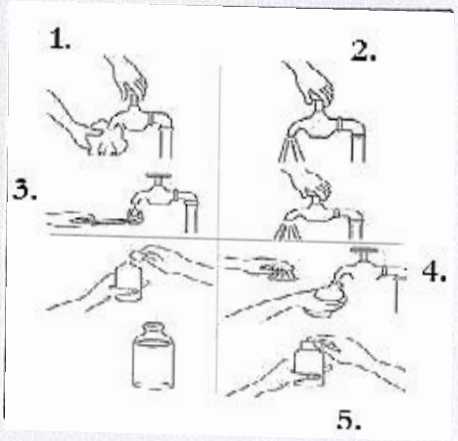
Groundwater samples will typically be collected from the discharge line of a pump or from a *bailer*, either from the pour stream of an up-turned bailer or from the stream from a bottom-emptying device. A bailer is a hollow tube used to retrieve groundwater samples from monitoring wells. Bailers are tied to a piece of rope or a piece of wire and lowered into the water column. Once lowered, the bailer uses a simple ball check valve to seal at the bottom in order to pull up a sample of the groundwater table.





Sampling water from a tap for microbiological analysis

1. Carefully clean and disinfect the inside and outside of the tap.
2. Open the tap and let water flow for 2-3 minutes or until the water temperature has stabilized.
3. Turn off the tap and sterilize the spout by heating it with a blow lamp, gas torch or by igniting a piece of cotton wool soaked with methylated spirits until any water in the tap boils.
4. Take care not to allow the container to touch the tap.
5. Take a water sample with the sample container



Sampling Information

The following information should be included in the label attached to the sample. Labeling the sample with appropriate information is an important part in sampling program. Use water proof ink to record all the information:

- Location (and name) of sampling site, with coordinates and any other relevant information.
- Details of sampling point.
- Date of sampling.
- Method of sampling.
- Time of sampling.
- Name of sampler.
- General environment and climatic conditions.
- Nature of pre-treatment.
- Preservation procedure.
- Water quality parameters collected in the field.
- Any information which may affect the results of analysis.

Sample Preservation

There is usually a delay between the collection and analysis of a sample. The nature of the sample can be changed during this period. Therefore proper preservation is required in the way to laboratory after collection, and in the laboratory up to when analysis starts.

Complete and unequivocal preservation of samples- whether domestic wastewater, industrial wastes, or natural waters, is practically impossible as because - complete stability for every constituent never can be achieved.

At best, preservation techniques only retard chemical (especially, hydrolysis of constituents) and biological changes that inevitably continue after sample collection. No single method of preservation is entirely satisfactory; the preservative is chosen with due regard to the determinations to be made. Commonly used preservation methods are - pH control, chemical addition, the use of amber and opaque bottles, refrigeration, filtration, and freezing.

Note:

See the appendix for the details of the preservation conditions for different water quality parameters.

Experiment No. 1

Determination of pH of Water

Introduction

pH stands for the "power of hydrogen". The numerical value of pH is determined by the molar concentration of hydrogen ions (H^+). This is done by taking the negative logarithm of the H+ concentration [-log(H^+)].

$$pH = -\log \{H^+\} \quad -(1)$$

Where, $\{H^+\}$ is the concentration or activity of hydrogen ion or proton in moles/liter (M). For example, if a solution has a H+ concentration of 10^{-6} M, the pH of the solution will be -log(10^{-6}), which equals 6.

In contrast, pOH is a measure of hydroxide ion (OH–) concentration. Water dissociates to form hydrogen ion (H^+) and hydroxyl ion (OH^-) according to the following equation:

$$H_2O = H^+ + OH^- \quad -(2)$$

The sum of pH and pOH is always 14 [pH + pOH = 14]. This is because the product of proton concentration and hydroxide concentration must always equal the equilibrium constant for the ionization of water, which is equal to 10^{-14}. At equilibrium, we can write,

$$K_w = \frac{\{H^+\}\{OH^-\}}{\{H_2O\}} \quad -(3)$$

Since concentration of water is extremely large (approximately 55.5 moles/L) and is diminished very little by the slight degree of ionization, it may be considered as a constant and its activity is taken as 1.0. Thus, Eq- 3 can be written as:

$$K_w = \{H^+\}\{OH^-\} \quad (4)$$

Where, K_w = Equilibrium Constant

At 25°C de-ionized or distilled water dissociates to yield 10^{-7}M of hydrogen ion (H^+) and 10^{-7} M of hydroxyl ion (OH^-). Therefore for pure water at 25°C, $K_w = 10^{-7} \times 10^{-7} = 10^{-14}$. This is known as the ion product of water or ionization constant for water. Hence, according to Eq.-1, pH of deionized water is equal to 7.0. Therefore, the pH can be represented by a scale ranging from 0 to 14, with 7 being neutral. So, pH is also a measure of the acidic or alkaline condition of water. pH of less than 7 indicate acidity, whereas a pH of greater than 7 indicates a base.

Groundwater is often found to be slightly acidic due to the presence of excess carbon dioxide. Aeration removes carbon dioxide and hence causes a rise in pH value. However, some natural waters are sometimes found to be slightly alkaline due to the presence of bicarbonate and, less often, carbonate. Water with pH outside the desirable neutral range may exhibit sour taste and accelerate the corrosion of metallic plumbing fittings and hot water services.

Environmental Significance

If the pH of water is too high or too low, the aquatic organisms living within it will die. pH can also affect the solubility and toxicity of chemicals and heavy metals in the water. The majority of aquatic creatures prefer a pH range of 6.5-9.0, though some can live in water with pH levels outside of this range.

pH values greater than 11 can cause skin and eye irritations, as does a pH below 4. A pH value below 2.5 will cause irreversible damage to skin and organ linings. Lower pH levels increase the risk of mobilized toxic metals that can be absorbed, even by humans, and levels above 8.0 cannot be effectively disinfected with chlorine, causing other indirect risks. In addition, pH levels outside of 6.5-9.5 can damage and corrode pipes and other systems, further increasing heavy metal toxicity.

A controlled value of pH is also desired in water supplies, sewage treatment and chemical process plants. In water supply pH is important for coagulation, disinfection, water softening and corrosion control. In biological treatment of wastewater, pH is an important parameter, since organisms involved in treatment plants are operative within a certain pH range. According to Bangladesh Environment Conservation Rules (2023), drinking water guideline value for pH is 6.5 - 8.5.

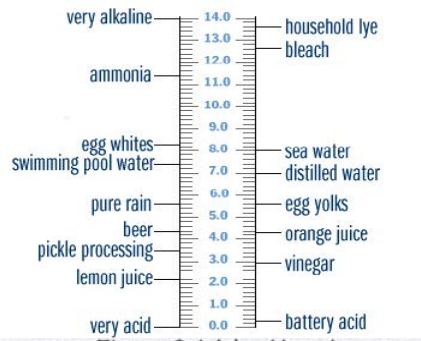
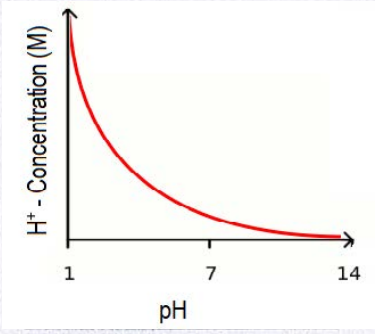


Figure-2.1 (a): pH scale



(b): H^+ ion activity vs pH

Theory on Experimental Method

Measurement of pH is carried out either colorimetrically (using pH indicator paper) or by electrochemical method (i.e., using a H^+ ion sensitive pH probe/meter). The second method is more expensive but precise whereas first one is cheap but serious erroneous result may produce due to interference of color, turbidity, high saline content, etc. However, before measuring pH with pH meter it is necessary to calibrate (standardize) the instrument using some buffer solutions of known pH value.

Reagent

- Standard pH solutions (buffer) for calibration of pH meter.

Apparatus

- pH meter



Figure-3.2: Digital pH meter

Procedure

1. Calibrate the pH meter using standard pH solutions. The calibration procedure would depend on the pH range of interest.
2. Take about 100 ml of the test sample in a beaker. Do not to agitate the sample to prevent exchange of gases between the sample and the atmosphere.
3. Insert pH meter in the sample. Allow sometime for attainment of equilibrium. Turn on the pH meter and take reading of pH.

Experiment No. 2

Determination of Color of Water

Introduction

Pure water should not pose any color. Color in water may result from the presence of natural metallic ions (iron and manganese),humus and peat materials, plankton, weeds, and industrial wastes. Impurities in water may exist either in the colloidal from or in suspended state. Color caused by dissolved and colloidal substances is referred as "true color" and that caused by suspended matter, in addition to dissolved and colloidal matters, is called "apparent color" as it can be easily removed by filtration. Ground water may show color due to the presence of iron compounds.

Environmental Significance

Though presence of color in water is not always harmful to human but in most cases it is. Even if the water is not harmful, aesthetically people do not prefer to use water with color. Moreover, disinfection by chlorination of water containing natural organics (which produces color) results in the formation of trihalomethanes including chloroform and a range of other chlorinated organic substances leading to problems which is a major concern in water treatment. So it is important to limit the color of water for domestic supplies. According to Bangladesh Environmental Conservation Rules 2023 (ECR'23), drinking water standard value for color is 15 Pt-Co Unit.

Theory on Experimental Method

Color is determined by visual comparison of the sample with known concentrations of colored solutions. Comparison also may be made with special, properly calibrated glass color disks. The platinum-cobalt method of measuring color is the standard method; the unit (Pt-Co Units) of color being that produced by 1 mg platinum–cobalt salt/L in the form of the chloro-platinate ion which is approximate the yellow-brown color of natural waters. The platinum-cobalt method is useful for measuring color of potable water and of water in which color is due to naturally occurring materials. It is not applicable to most highly colored industrial wastewaters. In the laboratory color of water is usually measured using spectrophotometer which uses light intensity of a specific wavelength (455 ηm). The color test measures (inversely) an optical property of water sample which result from the absorption of light of specific wavelength by the soluble color substances present in water (Figure-2.2). Before measuring the color of water it is necessary to plot standard calibration curve for color using different standard platinum-cobalt solutions of known concentrations within the range of interest.

Reagent

- Standard potassium chloro-platinate solution

Apparatus

- Spectrophotometer (HACH, DR 4000U)
- Filtration system including filter paper, funnel, holder, beaker etc.



Fig.-4.1: HACH, DR 4000U spectrophotometer

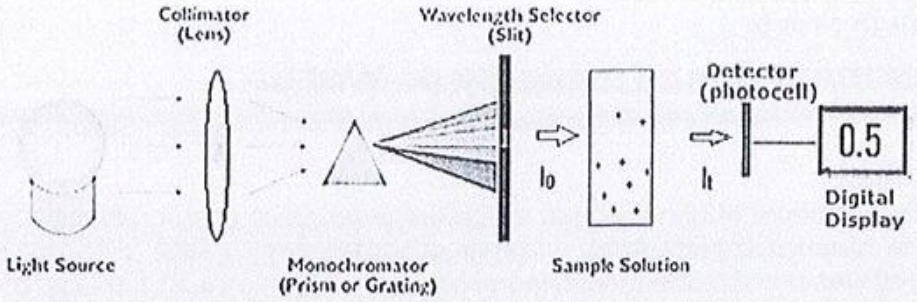


Fig. -4.2: Optical property used in measurement of color in water using spectrophotometer

Procedure

1. Prepare standard samples having color within a specific range by mixing different concentration of standard potassium chloro-platinate solution with distilled water. Using these samples to prepare a color calibration curve (absorbance vs color concentration) for the spectrophotometer.
2. Place a filter paper properly on a funnel and put a clean beaker below the funnel. Pour raw test sample on filter. Filtrate water is collected in the beaker. Discard initial portion of filtrate water. Take 50-mL of filtered test sample in a beaker.
3. Tale 50 –mL raw sample in another beaker. That gives the apparent color present in water sample.
4. Take 50-mL distilled water in another beaker. Use this sample as blank for color measurement.
5. Set the spectrophotometer to determine color concentration of the sample.
6. Put the blank sample inside the spectrophotometer cell and set the reading "zero".
7. Bring out the blank sample and place the filtrate sample inside the spectrophotometer
8. After a while the display shows the true color concentration of the sample.
9. Repeat procedure 5 to 7 for the raw water sample and record the apparent color value.

Experiment No. 3

Determination of Turbidity of Water

Introduction

Turbidity is a measure of the degree to which the water loses its transparency due to the presence of suspended particulates. In other words the term "turbid" is applied to water containing suspended matter that interferes with passage of light through the water or in which visual depth is restricted. Turbidity may be caused by a wide variety of suspended substances of various sizes ranging in size from colloidal to coarse particles, depending on the degree of turbulence. In rivers under flood conditions, most of the turbidity will be due to relatively coarse particles whereas in lakes and other waters existing under relatively quiescent conditions, most of the turbidity will be due to colloidal and extremely fine particles.

Environmental Significance

Turbidity is important for water supply engineers as turbid water is not aesthetically acceptable to people. For filtration process, turbid water is not suitable as it causes quick clogging of filter bed and thus requires the use of pre-treatment plant. Turbidity is also an important parameter in disinfection process. Disinfection is usually accomplished by means of chlorine, ozone, or chlorine dioxide. To be effective, there must be contact between the agent and the organisms to be killed. However, in cases in which turbidity is caused by municipal wastewater solids, many of the pathogenic organisms may be encased in the particles and protected from the disinfectant. In addition, the suspended particles absorb heat from the sunlight, making turbid waters become warmer, and so reducing the concentration of oxygen in the water. The suspended particles scatter the light, thus decreasing the photosynthetic activity of plants and algae, which contributes to lowering the concentration even more. According to Bangladesh Environment Conservation Rules (2023), drinking water standard for Turbidity is 10 NTU (Nephelometric turbidity unit).

Theory on Experimental Method

Turbidity is usually measured in Nephelometric turbidity units (NTU) or Jackson turbidity units (JTU). Both methods involve measurement of turbidity using either a nephelometer or Jackson turbidimeter to compare a reference solution to the sample. Nephlometer is now practiced as more reliable, sensitive, and easier to use instrument that depend upon the principle of nephelometry. The second method was a rather crude instrument in which the turbidity of a suspension was measured by the depth of suspension through which the outline of a flame from a standard candle just disappeared.

Jackson turbidimeter used silica as a standard reference material (1 mg SiO_2/L = 1 unit of turbidity). Whereas in nephelometer, a light source illuminates the sample and one or more photoelectric detectors are used with a readout device to indicate the intensity of scattered light at right angle to the path of the incident light (Fig.-1).In nephlometer standardized preparations of formazin polymer as standard reference material. In additions to formazin standards, commercially available standards such as styrene divinylbenzene beads are also available. Turbidity value as low as 0.02 NTU can be determined by this procedure, provided that water with sufficiently low turbidity can be obtained for use in instrument calibration.

Samples with turbidity greater than 40 NTU are diluted with turbidity-free water until values within the range of 30 to 40 NTU are obtained. The turbidity is then determined by multiplying the measured by the dilution factor.

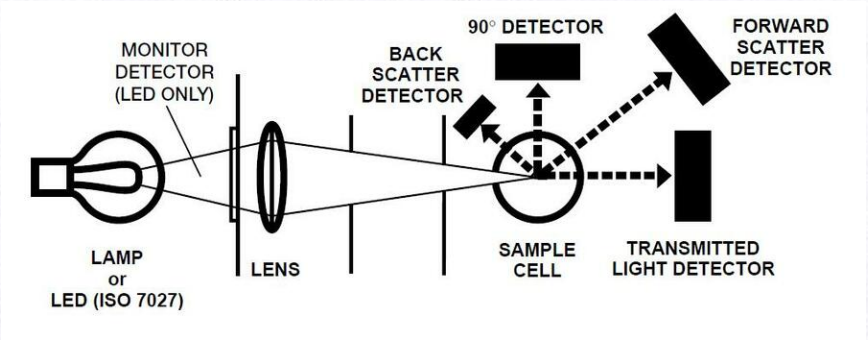


Fig.-5.1: Measurement of turbidity using Nephelometric turbidimeter - intensity of light scattered at 90º to the path of the incident light is considered for measurement of turbidity.

Reagent

- Formazin polymer standards

Apparatus

- DR LANGE Turbidimeter instrument set



Fig.-5.2: Turbidimeter

Procedure

1. Standardized the turbidimeter using formazin standards.
2. Fill the clean sample cell with the test sample and place it in the sample cell holder. Cover the sample cell with the light shield.
3. Turn on the switch and read the turbidity value directly from the screen.

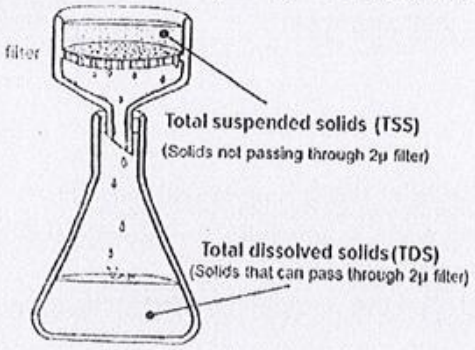
Experiment No. 4

Determination of Total Solids, Dissolved Solids and Suspended Solids in Water

Introduction

The solids are incorporated in water from its source (e.g., minerals with which it has been in contact). The term total dissolved solids refer to materials that are completely dissolved in water. These solids are filterable in nature. It is defined as residue upon evaporation (at 103-105°C) of filterable sample. The term total suspended solids can be referred to materials which are not dissolved in water and are non-filterable in nature. It is defined as residue upon evaporation of non-filterable sample on a filter paper

Total Solids (TS) refer to the matter that remains as residue upon evaporation and drying at 103 -105°C. Total solids (TS) include total suspended solids (TSS) - the portion of total solids retained by a filter, and total dissolved solids (TDS) - the portion that passes through the filter.



Total solids (TS) = Total suspended solids (TSS) + Total dissolved solids (TDS)

In potable water, most of the solids remain in dissolved form and consists of inorganic salts, small amounts of organic matter, and dissolved gases. The total dissolved solids content of potable water usually ranges from 20 to1000 mg/L. Groundwater usually has higher dissolved solids.

Environmental Significance

Dissolved minerals, gases and organic constituents may produce aesthetically displeasing color, taste and odor. Some dissolved organic chemicals may deplete the dissolved oxygen in the receiving waters and some may be inert to biological oxidation, yet others have been identified as carcinogens. Suspended material is aesthetically displeasing and provides adsorption sites for chemical and biological agents. Suspended solids determination is extremely valuable in the analysis of polluted waters. Presence of such solids in water prevents entering of light into water, thus reducing the growth of oxygen producing plants.
Estimation of total dissolved solids is useful to determine whether the water is suitable for drinking purpose, agriculture and industrial purpose. According to Bangladesh Environment Conservation Rules (2023), potable water should not contain more than 1000 mg/L of total dissolved solids (TDS). Water with high dissolved solids generally is of inferior palatability and may induce unfavourable physiological reaction in the user. Concentrations of TDS above 500 mg/L result in excessive scaling in water pipes, water heaters, boilers and household appliances. Water high in suspended solids may be aesthetically unsatisfactory for such purposes as bathing.

Theory on Experimental Method

Water has a boiling point of 100°C. It may rise by a few degrees due to the presence of impurities. When water with solids is kept at 103 -105°C for 24 hours, the water gets evaporated and the solids either in dissolved state or in suspended state remain as residue. A rapid assessment of the dissolved solids content of water can be obtained by specific conductance measurements. Such measurement indicates the capacity of a sample to carry an electrical current, which in turn is related to the concentration of ionized substances in the water. Most dissolved inorganic substances in water are in ionized form and so contribute to the specific conductance.

Although the nature of the various ions, their relative concentrations, and the ionic strength of the water affect this measurement, such measurement can give practical estimate of the dissolved mineral content of water. The TDS content can be approximated by multiplying the specific conductance in micro Siemens per cm (µS/cm) by an empirical factor varying from 0.55 to 0.90 depending on the chemical composition of the TDS.

Apparatus

- Beaker (150 mL) : 2 pcs
- Measuring cylinder (100 mL) : 1 pc
- Funnel : 1 pc
- Dropper : 1pc
- Filter paper (2 µm) : 2 pcs

Procedure

Measurement of Total Solids (TS):

1. Take a clear dry 150 - mL capacity glass beaker (which was kept at 103°C in oven for 1 hour) and put appropriate identification mark on it. Weigh the empty beaker (W_1).
2. Thoroughly mix the sample. Measure 100 - mL of the sample using the measuring cylinder and pour in to the beaker.
3. Place the beaker in an oven maintained at 103°C for 24 hours. After 24 hours, cool the beaker and weigh. Measure the total weight of solids residue and beaker (W_2).
4. Calculate total solids (TS) as follows:

$$\text{Total solids, TS (mg/L)} = \frac{(W_2 - W_1)(\text{mg}) \times 1000}{\text{volume of sample taken(mL)}}$$

Measurement of Dissolved Solids (TS) :

1. Same as above (step-1 of TS measurement).
2. Take a 100 mL of sample and filter it through a double layered filter paper and collect the filtrate in a beaker.
3. Repeat the same procedure as in steps- 3 and 4 of TS measurement and determine the dissolved solids content as follows:

$$\text{Total Dissolved Solids, TDS (mg/L)} = \frac{\text{solid residue(mg)} \times 1000}{\text{volume of sample taken(mL)}}$$

Measurement of Suspended Solids:

Total Suspended Solids, TSS (mg/L) = TS (mg/L) - TDS (mg/L)

Experiment No. 5

Determination of Carbon dioxide in Water

Introduction

The total carbon dioxide present in water is the sum of the free carbon dioxide and the carbon dioxide existing in the form of bicarbonate and carbonate ions. The presence of free carbon dioxide in water is caused by the decay of organic matter, dissolution of carbon dioxide from underground sources, and the dissolution from the atmosphere. Since the carbon dioxide content of the normal atmosphere is quite low (less than 0.04% by volume), this is not a source of appreciable carbon dioxide except where flue gases containing a high percentage of carbon dioxide are brought in relatively close contact with the water. Surface waters usually contain less than 10 ppm (mg/L) of free CO_2, while some ground water sources are found to contain free carbon dioxide exceeding 50 ppm (mg/L).

Environmental Significance

Carbon dioxide gas on solution in water produces carbonic acid (Equation-1) resulting in lowering of pH value and thus produces corrosive characteristic in water. Severe corrosion of heat exchanger, pipes, valves, etc. can result where appreciable quantities of free carbon dioxide are present in water. Corrosion from carbon dioxide in boiler system is most frequently encountered in steam and return lines. Even though the boiler feed water contains no free carbon dioxide, appreciable concentration of this corrosive gas may come from carbonate and bicarbonate of the boiler feed water. Although Bangladesh Environment Conservation Rules (ECR, 1997) does not set any limit for its presence in drinking water, limits for carbon dioxide has been prescribed for many industrial uses.

Theory on Experimental Method

This test for determination of free carbon dioxide in water involves titration of the water sample with standard sodium hydroxide solutions in the presence of phenolphthalein indicator (which changes color from colorless to pink at a pH of about 8.3). Carbon dioxide reacts with sodium hydroxide to form sodium bicarbonate with a consequent increase in pH value. The reaction of sodium hydroxide with free carbon dioxide reaches a completion at a pH of about 8.3. As soon as this pH is reached, a slight pink color is developed and this is taken as end point of reaction. The reactions may be considered to occur as follows:

$$CO_2 + H_2O = H_2CO_3 \quad (1)$$

$$H_2CO_3 + NaOH = HCO_3^- + Na^+ + H_2O \quad (2)$$

$$HCO_3^- + NaOH = CO_3^{2-} + Na^+ + H_2O \quad (3)$$

Determination of CO_2-Acidity

Phenolphthalein acidity (often called CO_2 acidity) of water is defined as the amount of standard base (usually $\frac{1}{50}$ N NaOH) required raising the pH of a sample of water to the phenolphthalein end point of 8.3. CO_2 acidity is expressed as $CaCO_3$ (calcium carbonate required to neutralize H_2CO_3)

Hence, Acidity could be easily determined from the results of CO_2 determination as follows:

$$\text{Phenolphthalein Acidity as mg/L } CaCO_3 = \frac{CO_2\ (\text{mg/L}) \times 50}{44} \quad (4)$$

Acidity Determination					
Indicator	**Methyl Orange**		**Phenolphthalein**		
Color	**Orange**	**Yellow**	**Color less**	**Pink**	
pH Scale [End Point]	4.5	6.3	8.3	10.3	
Type of Acidity	**Mineral Acidity (Methyl Orange Acidity)**	**CO_2 Acidity (Phenolphthalein Acidity)**		----------	
	Total Acidity / Titrable acidity				**No Acidity**
Add 0.02 N Caustic Soda (NaOH)	Titration upto pH 4.5 (Orange color turns into Yellow color)	Titration upto pH 8.3 (Pink color just appears)		----------	
Measurement	ml of 0.02N, NaOH required is equivalent to Mineral Acidity	ml of 0.02N, NaOH required is equivalent to CO_2 Acidity		----------	
Chemical Reaction	----------	$H_2CO_3 + OH^- \rightarrow HCO_3^- + H_2O$		$HCO_3^- + OH^- \rightarrow CO_3^{2-} + H_2O$	$H_2O \rightarrow H^+ + OH^-$
Major Species	Carbonic Acid (H_2CO_3)	Carbonic Acid (H_2CO_3)	Bicarbonate (HCO_3^-)	Bicarbonate (HCO_3^-) Carbonate (CO_3^{2-})	Carbonate (CO_3^{2-}) Hydroxide (OH^-)

Many people use titratable acidity and total acidity as synonyms, but they are not. The titratable acidity is always less than the total acidity, because not all of the hydrogen ions expected from the acids are found during the determination of titratable acidity. However, titratable acidity is easier to measure.

Reagents

- Phenolphthalein indicator
- Standard $\frac{1}{44}$N sodium hydroxide

Apparatus

- Beaker : 2 pc
- Measuring cylinder : 1 pc.
- Dropper : 1 pc.
- Stirrer : 1 pc.
- Burette : 1 pc.

Procedure

1. Take 100 mL of sample into a beaker and same quantity of distilled water into another. Add 10 drops of Phenolphthalein indicator to each. If a pink color develops, no carbon dioxide is present in the water sample. Otherwise, go to the next step.

2. Add $\frac{1}{44}$N sodium hydroxide solution from a burette to the sample and stir gently until a slight permanent pink color appears as compared with the distilled water. Measure the mL of sodium hydroxide required for titration.

Calculation

Free CO_2 in water (mg/L) = Multiplying Factor (MF) x mL of $\frac{1}{44}$ N NaOH added

Where, MF = $\frac{\text{normality of NaOH x equivalent wt.of } CO_2 \text{ x } 1000}{\text{mL of sample taken}}$

Phenolphthalein acidity as $CaCO_3$ (mg/L) – use equation (4)

Note:

Since excess CO_2, if present in water, easily escapes to the atmosphere, tests for presence of CO_2 in water should be performed immediately after collection of water sample, especially for groundwater samples which usually contain high carbon dioxide. If this is not possible, the sample bottle should be completely filled and capped and the sample should be kept at a temperature lower than that at which it was collected. Where the free CO_2 of the water sample is high, there may be some loss of carbon dioxide to the atmosphere during the titration process. To check this, it is advisable to secure a second sample. Complete determination of CO_2 on the first sample. Then take the second sample (100 mL) and immediately add the full amount of N sodium hydroxide solution used in the titration of the first sample. Add 10 drops of phenolphthalein indicator and if the sample remains colorless, add additional $\frac{1}{44}$ N sodium hydroxide to the end point (till the slight pink color appears) and accepting this second test as more accurate titration.

Experiment No. 6

Determination of Alkalinity of Water

Introduction

Alkalinity is the measure of water's capacity to absorb hydrogen ion (neutralize acids) without significant p H change. The term acid neutralizing capacity (ANC) is sometimes used. The alkalinity of natural waters is due primarily to the salts of weak acids, although weak or strong bases may also contribute and such substances act as buffer to resist a drop in pH resulting from acid additions. Bicarbonates represent the major form of alkalinity, since they are formed in considerable amounts from the action of CO_2 upon basic materials in the soil.

$$CO_2 + CaCO_3 + H_2O = Ca^{2+} + 2HCO_3^- \qquad (1)$$

Other salts of weak acids, such as borates, silicates, and phosphates, may be present in small amounts. A few organic acids, e.g., humic acid, form salts that add to the alkalinity of natural waters.

Although many materials may contribute to the alkalinity of water, three major types of chemical constituents cause the major portion of the alkalinity in natural waters: (i) hydroxides, (ii) carbonates, and (iii) bicarbonates. For most practical purposes, alkalinity due to other materials in natural waters is insignificant and may be ignored. The predominance of different carbonate species (closed system) is shown in Fig- 1.

$$\text{Total Alkalinity} = \Sigma\,[\,HCO_3^- + 2CO_3^{2-} + OH^- - H^+\,]$$

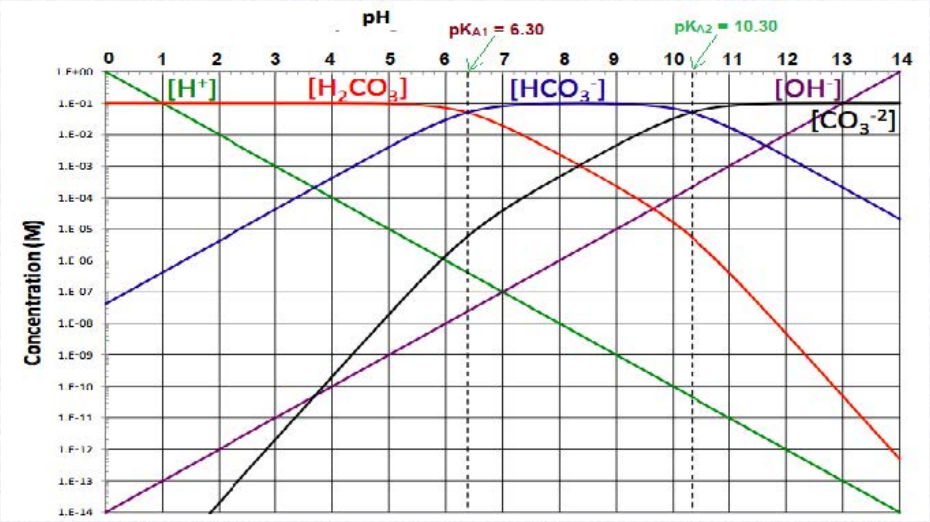


Fig.- 8.1: Logarithmic concentration diagram for 0.01 M Carbonic Acid (Source: Sawyer et al., 1994)

Environmental Significance

As mentioned earlier alkalinity is a measure of the buffer capacity and in this sense is used to a great extent in wastewater treatment practice. Alkalinity is very important in iron removal from water by oxidation-precipitation process. It has been found that rate of oxidation of dissolved ferrous iron (Fe^{2+}) into insoluble ferric iron (Fe^{3+}) is very slow for low alkaline waters (alkalinity less than 130 mg/L as $CaCO_3$). Because the alkalinity of many waters is primarily a function of carbonate, bicarbonate and hydroxide content, it is taken as an indication of the concentration of these constituents.

Alkalinity of water has little public health significance. Highly alkaline waters are usually unpalatable. Typical alkalinity of different natural water sources are given in the table below:

Water sources	Alkalinity (mg/L as $CaCO_3$)
Rainwater	< 10
Typical surface water	20 - 200
Surface water in regions with alkaline soils	100 - 500
Groundwater	50 - 1000
Seawater	100 - 500

Theory on Experimental Method

Alkalinity is measured volumetrically by titration with $\frac{1}{50}$ N(i.e.,0.02 N) H_2SO_4 and is reported in terms of equivalent $CaCO_3$. For samples with initial pH above 8.3, the titration is made in two steps-

i) In the first step, the titration is conducted until the pH is lowered to 8.3, the point at which phenolphthalein indicator turns from pink to colorless.
ii) The second phase of the titration is conducted until the pH is lowered to about 4.5, corresponding to the methyl orange (or bromocresol green) end point.

When the pH of a sample is less than 8.3, a single titration is made to a pH of 4.5. The first end point of pH 8.3 corresponds to the equivalence point for the conversion of carbonate to bicarbonate (Eq.-1,Figure-1). When alkalinity is measured to the phenolphthalein end point (pH 8.3), it is called phenolphthalein alkalinity, which is majorly due to the presence of either hydroxide or carbonate or both.

$$CO_3^{2-} + H^+ = HCO_3^- \qquad (2)$$

The use of a pH of about 4.5 for the end point for the second step of the titration corresponds approximately to the equivalence point for the conversion of bicarbonate ion to carbonic acid (Eq. 2, Fig. 1). When alkalinity is measured to the methyl orange (or bromocresol green) end point, it is called methyl orange alkalinity or total alkalinity, which is due to the presence of hydroxide, carbonate, and bicarbonate.

$$HCO_3^- + H^+ = CO_3^{2-} \qquad (3)$$

<table>
<tr><th colspan="6">Alkalinity Determination</th></tr>
<tr><td>Indicator</td><td colspan="2">Methyl Orange/
Bromeresol Green</td><td colspan="3">Phenolphthalein</td></tr>
<tr><td>Color</td><td>Orange/
Red</td><td>Yellow /
Green</td><td>Color less</td><td colspan="2">Pink</td></tr>
<tr><td>pH Scale
[End Point]</td><td colspan="5">4.5 — 6.3 — 8.3 — 10.3
<-------------></td></tr>
<tr><td rowspan="2">Type of Alkalinity</td><td>No Alkalinity</td><td colspan="2">M-Alkalinity</td><td>----------</td><td>Caustic Alkalinity</td></tr>
<tr><td></td><td colspan="2"></td><td colspan="2">P-Alkalinity</td></tr>
<tr><td></td><td></td><td colspan="4">Total Alkalinity</td></tr>
<tr><td>Add 0.02 N Sulfuric Acid (H_2SO_4)</td><td>----------</td><td colspan="2">Titration upto pH 4.5 (Yellow color turns into orange color)</td><td colspan="2">Titration upto pH 8.3 (Pink color just disappears)</td></tr>
<tr><td>Measurement</td><td>----------</td><td colspan="2">ml of 0.02N, H_2SO_4 required is equivalent to M-Alkalinity</td><td colspan="2">ml of 0.02N, H_2SO_4 required is equivalent to P-Alkalinity</td></tr>
<tr><td rowspan="3">Chemical Reaction Major Species</td><td rowspan="3">Un-dissociated Carbonic Acid (H_2CO_3)
Carbonic Acid (H_2CO_3)</td><td colspan="2">$H_2CO_3 \leftarrow HCO_3^- + H^+$</td><td>$HCO_3^- \leftarrow CO_3^{2-} + H^+$</td><td>$H_2O \leftarrow H^+ + OH^-$</td></tr>
<tr><td>Carbonic Acid (H_2CO_3)</td><td>Bicarbonate (HCO_3^-)</td><td>Bicarbonate (HCO_3^-)
Carbonate (CO_3^{2-})</td><td>Carbonate (CO_3^{2-})
Hydroxide (OH^-)</td></tr>
<tr><td colspan="2">At pH 6.3, $(H_2CO_3) = (HCO_3^-)$</td><td colspan="2">At pH 10.3, $(HCO_3^-) = (CO_3^{2-})$</td></tr>
</table>

Reagents

- Standard 0.02N sulfuric acid

- Phenolphthalein indicator
- Methyl orange indicator

Apparatus

- Beaker : 2 pcs
- Measuring cylinder : 1pc
- Dropper : 1pc
- Stirrer : 1pc

Procedure

1. Take 100 mL of the sample into one beaker and the same amount of distilled water into another beaker.
2. Measure pH of the sample with pH meter.
3. Add 3 drops of phenolphthalein indicator to each. If the sample becomes pink, go to step-4. Otherwise, go to step- 5.
4. Add 0.02N H_2SO_4 acid from a burette until the pink color just disappears. Record the mL of the acid used.
5. Add 3 drops of methyl orange indicator to each beaker. If the sample becomes yellow, add 0.02N H_2SO_4 acid until the first change in color is noted. The end point is a slight orange tinge. Record the mL of acid used.

Calculation

Phenolphthalein Alkalinity (mg/L as $CaCO_3$)
= Multiplying Factor (MF) x mL of 0.02N H_2SO_4 (added up to pH 8.3) (4)

and, Total Alkalinity (mg/L as $CaCO_3$)
= MF x mL of 0.02N H_2SO_4 (added up to pH approx. 4.5) (5)

Where, MF = $$\frac{\text{normality of } H_2SO_4 \text{ x equivalent wt.of } CaCO_3 \text{ x } 1000}{\text{mL of sample taken}}$$

Calculation of Hydroxide, Carbonate and Bicarbonate Alkalinity

In water analysis it is often desirable to know the kinds and amounts of the various forms of alkalinity present. This information is especially needed in water-softening processes and in boiler-water analysis. The most common approach is computation of the concentrations of the different alkalinity forms using various equilibrium equations for carbonic acid. This method gives reasonably accurate results for constituents, even when present in fractional mg/L range.

A total alkalinity, as well as a pH measurement, is required. In addition, a dissolved solids measurement to correct for ion activity and a temperature measurement for the selection of a proper equilibrium constant are needed. However, reasonably accurate results are obtained without these two data if the dissolved solids concentration is not too high and water temperature is around 25°C. The following section describes only the third procedure to determine hydroxide, carbonate and bicarbonate alkalinities.

Calculation from Equilibrium Equations

The relevant equilibrium equations are as follows:

$$H_2O = H^+ + OH^- \quad (6a)$$

$$K_w = \{H^+\}\{OH^-\} = 10^{-14} \text{ (at } 25^oC) \quad (6b)$$

$$H_2CO_3 = H^+ + HCO_3^- \quad (7a)$$

$$K_{A1} = \frac{\{H^+\}\{HCO_3^-\}}{\{H_2CO_3\}} = 10^{-6.3} \text{ (at 25°C)} \quad (7b)$$

$$HCO_3^- = H^+ + CO_3^{2-} \quad (8a)$$

$$K_{A2} = \frac{\{H^+\}\{CO_3^{2-}\}}{\{HCO_3^-\}} = 10^{-10.3} \text{ (at 25°C)} \quad (8b)$$

The alkalinity of water can be expressed as follows:

Total alkalinity (mg/L as $CaCO_3$)/50,000 = $\sum[(HCO_3^-) + 2(CO_3^{2-}) + (OH^-) - (H^+)]$ (9)

From the above equations, the following expressions for different alkalinities can be derived:

Carbonate alkalinity (mg/L as $CaCO_3$)

$$= \frac{50{,}000 \left\{ \left(\frac{\text{Alkalinity}}{50{,}000} + H^+ \right) - \left(\frac{K_w}{H^+} \right) \right\}}{\left[1 + \left(\frac{H^+}{2K_{A2}} \right) \right]} \quad (10)$$

Bicarbonate alkalinity (mg/L as $CaCO_3$)

$$= \frac{50{,}000 \left\{ \left(\frac{\text{Alkalinity}}{50{,}000} + H^+ \right) - \left(\frac{K_w}{H^+} \right) \right\}}{\left[1 + \left(\frac{2K_{A2}}{H^+} \right) \right]} \quad (11)$$

It should be noted that the equilibrium constants vary with temperature and activities of ions vary with concentration of ions in water. If temperature of water varies significantly from 25°C and ionic strength of water is high (i.e., high concentration dissolved ions), appropriate corrections need to be incorporated in the above equations. Otherwise these equations would give reasonably accurate results.

Sometimes actual concentrations of carbonate and bicarbonate ions may be desired. Conversions to mg/L of CO_3^{2-} and HCO_3^- are as follows:

mg/L CO_3^{2-} = mg/L carbonate alkalinity x 0.6 (12)

mg/L HCO_3^- = mg/L bicarbonate alkalinity x 1.22 (13)

Molar concentrations may be obtained as follows:

(CO_3^{2-}) = (mg/L CO_3^{2-}) / 60,000 (14)

(HCO_3^-) = (mg/L HCO_3^-) / 61,000 (15)

Experiment No. 7

Determination of Hardness of Water

Introduction

Hard waters are generally considered to be those waters that require considerable amounts of soap to produce foam or lather and that also produce scale in hot-water pipes, heaters, boilers, and other units in which the temperature of water is increased substantially. The hardness of water varies considerably from place to place. In general, surface water is softer than groundwater. The hardness of water reflects the nature of the geological formations with which it has been in contact.

Hardness is caused by multivalent metallic cations. Such cations are capable of reacting with soap to form precipitates and with certain anions present in water to form scale. The principal hardness causing cations are the divalent calcium, magnesium, strontium, ferrous iron, and manganous ions. These cations and the important anions with they are associated are shown in Table-1 in the order of their relative abundance in natural waters. Aluminum and ferric ions are sometimes considered as contributing to the hardness of water. However, their solubility is so limited at pH values of natural waters that ionic concentrations are negligible. The hardness of water is derived largely from contact with the soil and rock formations.

Table-1. : Principal hardness causing cations and the major anions associated with them

Cations causing Hardness	Associated Anions
Ca^{2+}	HCO_3^-
Mg^{2+}	SO_4^{2-}
Sr^{2+}	Cl^-
Fe^{2+}	NO_3^-
Mn^{2+}	SiO_3^{2-}

Hardness caused by each cation can be calculated as follows:

$$\text{Hardness (mg/L as } CaCO_3) = \frac{M^{2+}\,(\text{mg/L}) \times 50}{\text{Equivalent weight of } M^{2+}} \qquad \text{--(1)}$$

Where, M^{2+} = concentration of divalent metal cation (mg/L) and 50 is the equivalent weight of $CaCO_3$.

$$\text{Total Hardness} = \sum\left[\frac{50}{20.04}Ca^{++} + \frac{50}{12.15}Mg^{++} + \frac{50}{27.92}Fe^{++} + \frac{50}{27.46}Mn^{++}\right] \qquad \text{--(2)}$$

$$= \sum[2.49\,Ca^{++} + 4.12\,Mg^{++} + 1.79\,Fe^{++} + 1.82\,Mn^{++}] \qquad \text{--(3)}$$

Environmental Significance

Hard waters are as satisfactory for human consumption as soft waters. Because of their adverse action with soap, however, their use for cleansing purpose is quite unsatisfactory, unless soap costs are disregarded. Soap consumption by hard waters represents an economic loss to the water user. Sodium soaps react with multivalent metallic cations to form a precipitate, thereby losing their surfactant properties. In recent years these problems have been largely alleviated by the development of soaps and detergents that do not react with hardness.

Boiler scale, the result of the carbonate hardness precipitation, may cause considerable economic loss through fouling of water heater and hot water pipes. Changes in pH in the

water distribution systems may also result in deposits of precipitates. Bicarbonates begin to convert to the less soluble carbonates at pH values above 9.0.
Magnesium hardness, particularly associated with the sulfate ion has a laxative effect on persons unaccustomed to it. Magnesium concentrations of less than 50 mg/L are desirable in potable waters, although many public water supplies exceed the amount. Calcium hardness presents no public health problem. In fact, hard water is apparently beneficial to the human cardiovascular system. Water can be generally classified in terms of the degree of hardness as described in table-2 below. According to ECR'2023 standard hardness should be below 500 mg/L as $CaCO_3$ based on taste and household use considerations.

Table-2: Water quality with respect to hardness

Water Quality	Hardness (mg/L as $CaCO_3$)
Soft	<50
Moderately hard	50-150
Hard	150-300
Very hard	>300

Theory on Experimental Method

Hardness is usually expressed in terms of $CaCO_3$. Perhaps the most accurate method of determining hardness is by a calculation based upon the divalent ions found through complete cation analysis (Equation–1 to 3).This method is preferred where complete analyses are available.
However, complete analyses of metal ions are not always available and in laboratory total hardness is usually determined by the *EDTA Titrimetric Method.* This method yields very precise and accurate results and is the method of choice in most laboratories.
The EDTA Titrimetric method involves the use of solutions of ethylene-diamine-tetra-acetic acid (EDTA) or its sodium salt as the titrating agent. These compounds are chelating agents (A chemical compound in the form of a heterocyclic ring, containing a metal ion attached by coordinate bonds to at least two non-metal ions) and thus form extremely stable complexes with Ca^{2+}, Mg^{2+} and other divalent cations causing hardness, as shown in Eq. -4.

$$M^{2+} + EDTA = [M \,.\, EDTA]_{complex} \qquad \text{--(4)}$$

The successful use of EDTA for determining hardness depends upon having an indicator present to show when EDTA is present in excess, or when all the ions causing hardness have been complexed.
The Eriochrome Black T dye serves as an excellent indicator to show when all the hardness ions have form complex with EDTA. When a small amount of Eriochrome Black T, having a blue color, is added to hard water with a pH of about 10.0, it combines with a few of the $Ca^{2+}$and Mg^{2+} ions to form a weak complex ions which produces wine red in color in the solution (Eq.-5).

$$M^{2+} + \text{Eriochrome Black T} = (M \,.\, \text{Eriochrome Black T})_{complex} \qquad \text{--(5)}$$
(wine red color)

During titration with EDTA, all free hardness ions are complexed according to Eq. 2. Finally, the EDTA disrupts the weak wine red complex compounds (M . Eriochrome Black T) and forms more stable complex with the divalent ions. This action frees the Eriochrome Black T indicator, and the wine red color changes to a distinct blue colour (Eq.- 4), indicating the end of titration.

$$(M.\text{Eriochrome Black T})_{complex} + EDTA = (M.EDTA)_{complex} + \text{Eriochrome Black T} \qquad \text{--(6)}$$
(wine red color) *(blue color)*

Reagents

- Buffer solution (for attaining a pH close to 10.0).
- Eriochrome Black T dye.
- Standard EDTA solution

Apparatus

- Beaker : 1 pcs
- Measuring cylinder : 1pc
- Dropper : 1pc
- Stirrer : 1pc
- HACH Auto-titration device: 1 set

Procedure

1. Take 50 mL sample in a 150 mL beaker.
2. Add one ml of standard buffer solution (supplied by HACH) to raise the pH of water sample to about 10.0 (Note: If 100 mL sample is taken, add 2 mL buffer).
3. Add one packet of Eriochrome Black T dye (supplied by HACH) indicator to the beaker. The sample would turn wine-red (if hardness is present).
4. Fit the cartridge containing standard EDTA solution to the titrator device (HACH).
5. Turn the flow control knob of the device until the solution starts to come out of the tube fitted to the cartridge. Take initial reading of the counter.
6. Immerse the tube fitted to the cartridge into the water sample and start titrating (under constant stirring) by turning the flow control knob of the auto-titrator. Continue until the wine-red color of the sample changes to blue. Take final reading of the counter.

Calculation

Total hardness (mg/L as $CaCO_3$) = MF x (initial counter reading - final counter reading)

$$\text{Where, MF} = \frac{\text{normality of EDTA x equivalent wt. of } CaCO_3 \text{ x } 1000}{\text{mL of sample taken}}$$

(Normality of EDTA titrant used in the lab is 0.002 N)

Therefore for 100 mL sample, MF =1

Total hardness (mg/L as $CaCO_3$) = initial counter reading - final counter reading

And for 50 mL sample, MF =2

Total hardness (mg/L as $CaCO_3$) = 2 x (initial counter reading - final counter reading)

Types of Hardness

In addition to total hardness, it is often desirable and sometimes necessary to know the type of hardness present. Hardness is classified in two ways:

(1) With respect to the metallic ion (calcium and magnesium hardness), and

(2) With respect to the anions associated with the metallic ions (carbonate and non-carbonate hardness).

Calcium and Magnesium Hardness

Calcium and magnesium cause by far the greatest portion of the hardness occurring in natural waters. Sometimes, it is important to know the amounts of calcium and magnesium hardness in water. For example, it is necessary to know the magnesium hardness or the amount of Mg^{2+} in order to calculate lime requirement in lime-soda ash softening. The calcium and magnesium hardness may be calculated from the complete chemical analysis. If calcium hardness is determined, magnesium hardness is obtained by subtracting calcium hardness from total hardness as follows:

Total hardness - calcium hardness = magnesium hardness (5)

Carbonate and Non-carbonate Hardness

The part of total hardness that is chemically equivalent to the bicarbonate plus carbonate alkalinities present in water is considered to be carbonate hardness. Since alkalinity and hardness are both expressed in terms of $CaCO_3$, the carbonate hardness can be found as follows:

When alkalinity < total hardness, Carbonate hardness (mg/L) = alkalinity (mg/L) - (6)
When alkalinity ≥ total hardness, Carbonate hardness (mg/L) = total hardness (mg/L) - (7)

Carbonate hardness is of particular interest because the bicarbonate and carbonate ions with which it is associated tend to precipitate this portion of the hardness at elevated temperatures such as occur in boilers or during the softening process with lime. Carbonate hardness was formerly called *temporary hardness* because it can be caused to precipitate by prolonged boiling (Eq.- 8).

$$Ca^{2+} + 2HCO_3^- = CaCO_3\,(s) + CO_2 + H_2O \quad (8)$$
$$Ca^{2+} + 2HCO_3^- + Ca(OH)_2 = 2CaCO_3\,(s) + 2H_2O \quad (9)$$

The amount of hardness which is in excess of the carbonate hardness is called non-carbonate hardness, and can be estimated as follows:

Non-carbonate hardness (NCH) = total hardness - carbonate hardness (10)

Non-carbonate hardness was formerly called permanent hardness because it cannot be removed or precipitated by boiling. Non-carbonate hardness cations are associated with sulfate, chloride, and nitrate anions.

Pseudo-Hardness

Sea, brackish, and other waters that contain appreciable amounts of Na^+ interfere with the normal behavior of soap because of the common ion effect. Sodium is not a hardness-causing cation, and so this action which it exhibits when present in water in high concentration is termed *pseudo-hardness*.

Understanding Composition of a Water Sample from its Alkalinity and Hardness Values

The table below shows the major cations and anions that may exist in any water sample depending on the relationship among pH, alkalinity and hardness. Here Consider: 300 mg/L as $CaCO_3$ as "high" hardness /alkalinity and 30 mg/l as $CaCO_3$ as "low" hardness /alkalinity.

Table -3: Different Forms of Hardness Causing Compounds Depending on pH, Harness and Alkalinity

Alkalinity (mg/L as $CaCO_3$)	Hardness (mg/L as $CaCO_3$)	pH value	Major Compounds	Type of Hardness/Alkalinity
300	300	< 8.3	$Ca(HCO_3)_2$ $Mg(HCO_3)_2$	Carbonate Hardness
		> 10.3	$CaCO_3$ $MgCO_3$	
		> 12.0	$Ca(OH)_2$ $Mg(OH)_2$	Caustic Hardness
300	30	< 8.3	$Na(HCO_3)_2$ $K(HCO_3)_2$	Non Hardness Alkalinity
		> 10.3	Na_2CO_3 K_2CO_3	
		> 12.0	NaOH KOH	Caustic Alkalinity
30	300	----	$CaCl_2$ $CaSO_4$ $MgCl_2$ $MgSO_4$	Non Carbonate Hardness
Negligible	Negligible	---	NaCl Na_2SO_4 KCl	----------

Experiment No. 8

Determination of Chloride in Water

Introduction

Chlorides occur in all natural waters in widely varying concentration. The chloride ion is highly mobile and concentrations in water are not affected by chemical reactions. Hence chloride does not biodegrade, readily precipitate, volatilize, or bioaccumulate. The chloride content normally increases as the mineral content increases. Upland and mountain supplies usually are quite low in chlorides, whereas river and groundwater usually have a considerable amount. Sea and ocean waters represent the residues resulting from partial evaporation of natural waters that flow into them and chloride levels are very high.

Chlorides gain access to natural waters in many ways. The solvent power of water dissolves chlorides from topsoil and deeper formations. Spray from the ocean is carried inland as droplets or as minute salt crystals, which result from evaporation of the water in the droplets. These sources constantly replenish the chlorides in inland areas where they fall. Ocean and seawaters invade the rivers that drain into them, particularly the deeper rivers. The salt water, being denser, flows upstream under the fresh water, which is flowing downstream. There is a constant intermixing of the salt water with the fresh water above. Groundwater in areas adjacent to the ocean is in hydrostatic balance with seawater. Over-pumping of groundwater produces a difference in hydrostatic head in favor of the seawater, and it introduce into the fresh water area. Such intrusion has occurred in many areas of the coastal southern region of Bangladesh.

Human excreta, particularly the urine, contain chloride in an amount about equal to the chlorides consumed with flood and water. This amount average about 6 gm of chlorides per person per day and increases the amount of Cl^- in municipal wastewater about 15 mg/L above that of the carriage water. Thus, wastewater effluents add considerable chlorides to receiving streams. Many industrial wastes (e.g., tannery waste) also contain appreciable amount of chlorides.

Environmental Significance

Chlorides in reasonable concentrations are not harmful to human. At concentrations above 250 mg/L they give a salty taste to water, which is objectionable to many people. However, The taste threshold of the chloride anion in water is dependent on the associated cation. Taste thresholds for sodium chloride and calcium chloride in water are in the range 200–300 mg/L. The taste of coffee is affected if it is made with water having a chloride concentration above 400 mg/L as NaCl or 530 mg/L as $CaCl_2$. Again, in many areas of the world where water supplies are scarce, source containing as much as 2,000 mg/L are used for domestic purposes without the development of adverse effects, once the human system becomes adapted to the water. According to Bangladesh Environment Conservation Rules (1997), drinking water standard for chloride is 150 - 600 mg/L; but for coastal regions of Bangladesh, the limit has been relaxed to 1000 mg/L.

It may act corrosively on metal pipes and be harmful to plant life. Chloride ions are adsorbed on the rebar surface. The Cl- promotes the hydration of the iron ions and thus facilitates the corrosion of steel. It also interfere with irrigation system, industrial processes etc.

Before the development of bacteriological testing procedures, chloride and nitrogen determination is being used for detecting contamination of groundwater by wastewater. Chlorides are used to some extent as tracers in sanitary engineering practice; however, they have been replaced to a great extent by organic dyes. Moreover measurement of total salinity and Cl^- is also important for irrigation water.

Theory on Experimental Method

A number of suitable analytical techniques are available for chloride in water:

i. Mohr method – silver nitrate titration with potassium as the chromate indicator,
ii. Mercuric nitrate method- mercuric nitrate titration with diphenyl-carbazone as the indicator.
iii. Potentiometric titration with silver nitrate,
iv. Automated iron(iii) mercury(ii) thiocyanate colorimetry,
v. Chloride ion-selective electrode,
vi. Silver colorimetry, and
vii. Ion chromatography etc.

In this class chloride is determined using Mohr method. This method involves simple titration process. However, in Mohr method color change occurs gradually and true end point (reddish brown precipitate) cannot be detected readily in solutions containing low concentrations of chloride (below 20 ppm) and even in higher concentrations, satisfactory titrations can be made only by a highly skilled technologist..

The "Standard Method" recommends a 0.0141 N silver nitrate solution which corresponds to $\frac{1}{71}$ N solution or a solution in which each millilitre is equivalent to 0.50 mg of chloride ion.

In the titration, chloride is precipitated as a white silver chloride (Eq. -1)

$$Ag^+ + Cl^- = AgCl \text{ (white precipitate)} \qquad (1)$$

The end point of titration cannot be detected visually unless an indicator capable of demonstrating the presence of excess Ag^+ is present. The indicator normally used is potassium chromate, which supplies chromate ions. As the concentration of Cl^- ions becomes exhausted, the silver ion concentration increases and a reddish brown precipitate of silver chromate is formed (Eq.- 2).

$$2Ag^+ + CrO_4^{2-} = Ag_2CrO_4 \text{ (reddish brown precipitate)} \qquad (2)$$

This is taken as evidence that all chloride has been precipitated. Since an excess Ag^+ is needed to produce a visible amount of Ag_2CrO_4, the indicator error is subtracted from all titrations.

Several precautions must be observed in the determination of chloride in order to ensure accuracy:

1. A uniform sample size must be used, preferably 100 ml (or 50 mL), so that ionic concentrations needed to indicate the end point will be constant.
2. The pH must be in the range of 7 to 8 because Ag^+ is precipitated as AgOH at high pH levels and the CrO_4^{2-} is converted to $Cr_2O_7^{2-}$ at low pH levels.
3. A definite amount of indicator must be used to provide a certain concentration of CrO_4^-, otherwise Ag_2CrO_4 may form too soon or not soon enough.

The indicator error or blank varies somewhat with the ability of individuals to detect a noticeable color change. The usual range is 0.2 to 0.4 mL of titrant. An error of 0.2 mL will be used in the class.

Reagents

- Potassium chromate indicator
- Silver nitrate solution (0.0141 N)

Apparatus

- Measuring cylinder (100 mL) : 1 pc.
- Beaker : 1 pc.
- Dropper : 1 pc.
- Stirrer : 1 pc.

Procedure

1. Take 50 mL of the sample in a beaker and add 5 drops (about 1 mL) of potassium chromate indicator to it.
2. Add standard (0.0141 N) silver nitrate solution to the sample from a burette, a few drops at a time, with constant stirring until the first permanent reddish color appears. This can be determined by comparison with distilled water blank. Record the mL of silver nitrate used.
3. If more than 7 or 8 mL of silver nitrate solution are required, the entire procedure should be repeated using a smaller sample diluted to 50 mL with distilled water.

Calculation

Chloride, Cl^- (mg/L) = (mL of $AgNO_3$ used - "error"/"blank") x Multiplying Factor (MF) --- (1)

Where, $MF = \dfrac{\text{normality of } AgNO_3 \text{ x equivalent wt.of } Cl^- \text{ x } 1000}{\text{mL of sample taken}}$

If a diluted sample is used for chloride measurement,

Chloride (Cl^-) concentration of original sample (mg/L) = Cl^- measured in Eq.-1 x (DF) --- (2)

Where,

Dilution Factor (DF) $= \dfrac{\text{Volume (mL) of diluted watersample (mix)}}{\text{Volume (mL) of original sample in the mix}}$ --- (3)

Experiment No. 9

Determination of Total Iron in Water

Introduction

Iron is the second most abundant metal in the earth's crust, of which it accounts for about 5%. Iron is mainly present in water in two forms: either the soluble ferrous iron or the insoluble ferric iron. Iron exists in soils and minerals as insoluble ferric oxide/hydroxide and iron sulphide (pyrite). In some areas, it also occurs as ferrous carbonate, which is very slightly soluble. Since groundwater usually contains significant amounts of carbon dioxide, appreciable amounts of ferrous carbonate may be dissolved according to eq.-1.

$$FeCO_3 + CO_2 + H_2O = Fe^{2+} + 2HCO_3^- \qquad (1)$$

If reducing (anaerobic) conditions exist in groundwater environment, the insoluble ferric iron [Fe^{3+}] is reduced to more soluble ferrous iron [Fe^{2+}] and iron concentration in water increases. Water containing ferrous iron is clear and colorless because the iron is completely dissolved. When exposed to air it oxidized to ferric form and cause reddish brown precipitates in water. Manganese in water also shows similar behavior. Thus, iron, as well as manganese, creates serious problems in public water supplies, specially in the system which uses groundwater as the source of water supply.

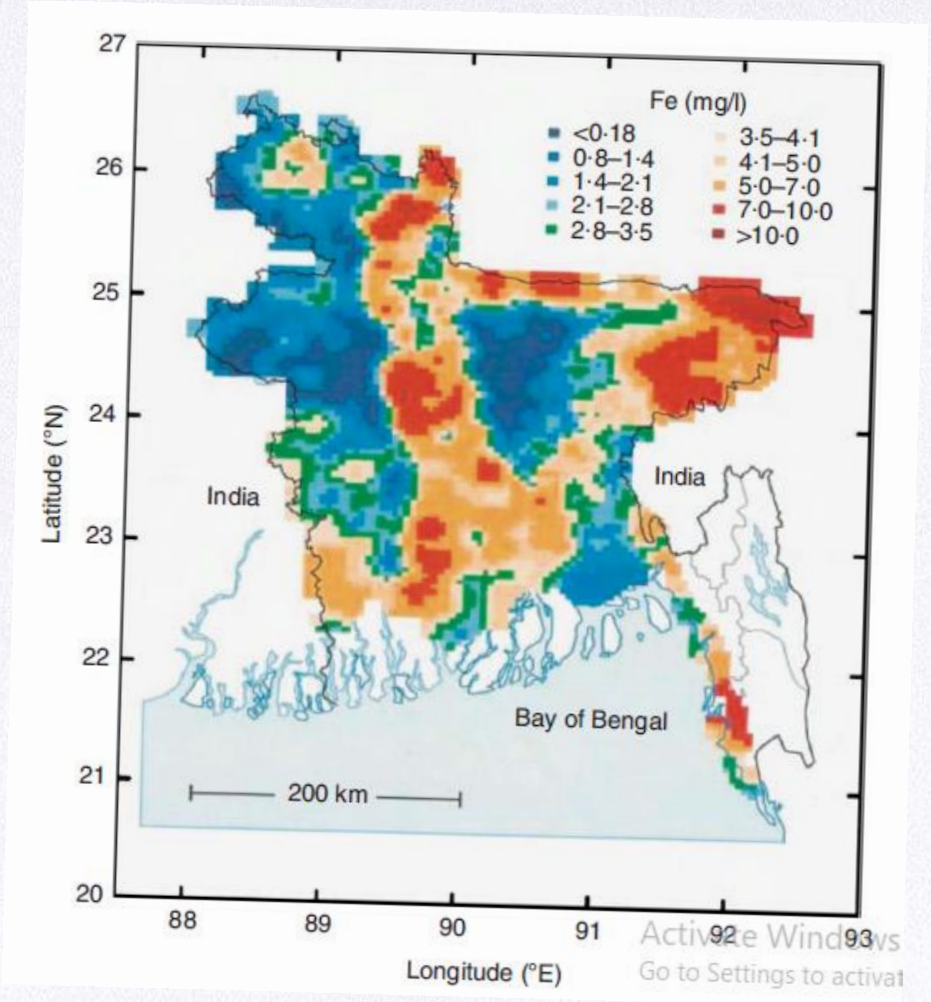


Fig-11.1: Iron concentration in groundwater in Bangladesh (British Geological Survey 2001)

Not only arsenic (As), but also iron (Fe) contamination in groundwater is now a vital problem in Bangladesh. According to the National Drinking Water Quality Survey (NDWQS) in 2009, approximately 70% and 61% of drinking water sources in Bangladesh, mainly groundwater, have been found to contain elevated level of iron (>0.3 mg/L) and manganese (>0.1 mg/L), respectively. According to the 2001 British Geological Survey (Figure 9.1), either side of the Brahmaputra basin, south of the Ganges basin and north-eastern haor regions are predominantly high groundwater iron areas, while the Barind and Madhupur tracts are the areas with predominantly low iron in groundwater. In several areas of Bangladesh, groundwater withdrawals are causing a large Fe contamination in groundwater levels during dry season.

Environmental Significance

As far as is known, human suffer no harmful effects from drinking waters containing iron and manganese. Such waters, when exposed to the air so that oxygen can enter, become turbid and highly unacceptable from the aesthetic viewpoint owing to the oxidation of iron and manganese to the Fe^{3+}, and Mn^{4+} states which form colloidal precipitates. The rates of oxidation are not rapid, and thus reduced forms can persist for some time in aerated waters. This is especially true when the pH is below 6 with iron oxidation and below 9 with manganese oxidation. The rates may be increased by the presence of certain inorganic catalysts through the action of microorganisms.

Both iron and manganese interfere with laundering operations, impart objectionable stains to plumbing fixtures and cause difficulties in distribution systems by supporting growths of iron bacteria. Iron also imparts a taste to water, which is detectable at very low concentrations. According to Bangladesh Environment Conservation Rules (1997), drinking water standard for iron is 0.3 - 1.0 mg/L.

Theory on Experimental Method

Ferric iron is seldom found in true solution in natural waters, unless they are highly acidic, because of the formation of insoluble ferric hydroxides. Ferrous iron is more likely to be found in true solution, although it is easily oxidized to the ferric state and precipitated in alkaline waters as ferric hydroxide.

Since some iron may exist as iron hydroxide precipitates, therefore it is necessary to bring precipitated form(s) of iron back in to solution before oxidizing total iron content in water. For this hydrochloric acid is added to the test sample to dissolve the insoluble ferric forms.

For determination of total iron by the following procedure, it must be ensured that all iron exists in ferric form (Fe^{3+}). This is most readily accomplished by using potassium permanganate, an oxidizing agent.

$$5Fe^{2+} + MnO_4^- + 8H^+ = 5Fe^{3+} + Mn^{2+} + 4H_2O \qquad (2)$$

Ferric iron is determined by producing a red-colored iron compound, ferric thiocyanate, by the addition of potassium thiocyanate (Eq.-3).

$$Fe^{3+} + 3\,KCNS = \underset{(red)}{Fe(CNS)_3} + 3\,K^+ \qquad (3)$$

The quantity of ferric iron is determined by comparison with the red color produced by standard iron solutions.

Reagents

- Hydrochloric acid
- Potassium permanganate solution
- Potassium thiocyanate solution
- Standard iron stock solution

Apparatus

- Nessler tube : 2 pc.
- Measuring cylinder : 1 pc.
- Dropper : 1 pc.

Procedure

1. Place 100 mL of the water sample in a Nessler.
2. Add 5 mL of dilute hydrochloric acid
3. Add two drops of potassium permanganate solution
4. Add 5 mL of potassium thiocyanate solution. The solution would turn brown if iron is present.
5. Compare the brown color formed with the standard prepared as follows:
   a. Add 100 mL of distilled water in a Nessler tube
   b. Add 5 mL of the dilute hydrochloric acid.
   c. Add two drops of potassium permanganate solution.
   d. Add 5 mL of potassium thiocyanate solution.
   e. Add 0.2 mL at a time of the standard iron solution until the color of the standard and the sample match.

Calculation

Total iron concentration of test sample (mg/L) =

$$\frac{\text{Standard iron solution added in dilstilled water (mL)} \times \text{Conc.of standard iron solution (mg/L)}}{\text{mL of sample taken}}$$

*For diluted sample multiply the obtained result with dilution factor

Experiment No. 10

Determination of Arsenic in Water

Introduction

Arsenic, the 20th most abundant element in the earth's crust and the 12th most abundant element in the biosphere, is a common trace inorganic contaminant in drinking water and is identified as a significant health risk. Arsenic is known for its high toxicity and its ability to induce cancer after long-term ingestion. Presence of elevated levels of arsenic in groundwater (especially from shallow aquifer) has become a major concern in Bangladesh. Arsenic pollution of groundwater is particularly challenging in Bangladesh since tubewell water extracted from shallow aquifers is the major source of drinking water for most of its population. The rural water supply is almost entirely based on groundwater supply through use of hand pump tubewells; the urban water supply is also heavily dependent on groundwater.

Awareness about the presence of arsenic in groundwater has been growing since late 1993 when arsenic was first detected in the district of Chapai Nawabgonj bordering the West-Bengal district of India. A detail survey carried out by the DPHE and BGS (British Geological Survey) in 1999 identified the presence of arsenic in 268 Upazilas out of 464 in the country (BGS and DPHE, 2001). In 2005, the number of affected Upazilas increased to 270. In 2002 and 2003, 4.7 million tube wells in Bangladesh have been screened for arsenic. Of those, 1.4 million tube wells were found to contain arsenic above the Bangladesh drinking water standard of 50 microgram per liter. According to the Multiple Indicator Cluster Survey (MICS) carried out by GoB and the Unicef in 2012-13, about 24.8 per cent of the population of Bangladesh are drinking water in the household with arsenic above the WHO provisional guideline value of 10 ppb, and 12.4 per cent of the population are exposed to water with arsenic exceeding the Bangladesh standard of 50 ppb (GoB and Unicef, 2015).

In Bangladesh, the arsenic in groundwater is of geologic origin and is probably only apparent now because it is only the last 25 – 35 years that groundwater has been extensively used for drinking in rural areas. Weathering of arsenic-rich base metal sulfides in the upstream of the Ganges basin appears to be a major source of arsenic-rich iron oxyhydroxides in the sediments of Bangladesh. Arsenic-rich iron oxyhydroxides appear to be the major source of arsenic, from which arsenic is released as a result of dissolution and desorption. Reducing environment in the alluvial aquifer, primarily due to the presence of organic matter, promotes dissolution of iron oxyhydroxides and release of arsenic. Use of phosphate fertilizer can potentially enhance release of arsenic as a result of replacement of arsenic by phosphate ions on the adsorption sites of iron oxyhydroxides. Natural and anthropogenic processes that may lead to release/mobilization of arsenic in the subsurface are being investigated.

According to ECR 1997, drinking water standard for arsenic in Bangladesh is 50 μg/L (or 0.05 mg/L). The WHO guideline value for arsenic in drinking water is 10 μg/L and the USEPA is also planning to revise its standard from 50 μg/L to 10 μg/L.

Arsenic Chemistry

Arsenic occurs in water in several different forms, depending upon the pH and the redox potential, E_h. Some of the most important compounds and species are shown in Table 10. 1. In groundwater, arsenic primarily exists as inorganic arsenic. Inorganic trivalent arsenic, [As(III)] or arsenite is the dominant form in reducing environment; while inorganic pentavalent arsenic [As(V)] or arsenate is the dominant form in oxidizing or aerobic environment.

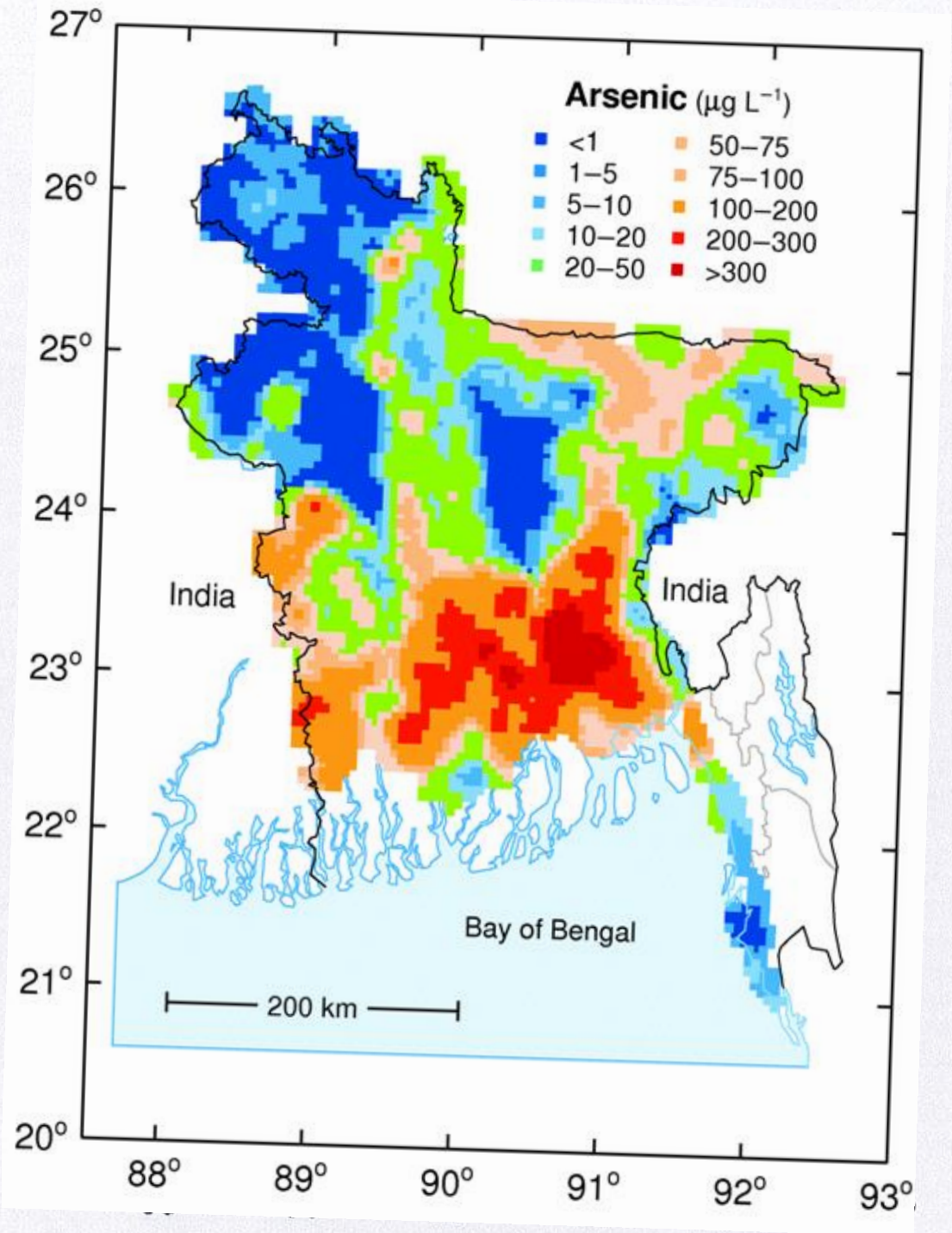


Figure 14.1: Distribution of Arsenic in Groundwater of Bangladesh

In groundwater environment where the conditions are mostly reducing, a significant part of the arsenic exists as As(III). As shown in Table 10.1, in the pH range of most groundwater (i.e., pH 6 – 9), dominant chemical form of As(III) is H_3AsO_3; while dominant chemical forms of As(V) in this pH range are: $H_2AsO_4^-$, $HAsO_4^{2-}$.

Table -10.1: Arsenic compounds and species and their environmental and toxicological importance in water

Compounds	Example	Environmental Significance/ Dominant pH region	Toxicity
Arsine	As^{3-}	Minor importance	Most toxic As species
Elemental Arsenic	As	Minor importance	Least toxic As species
Trivalent Arsenic	As(III)	Anaerobic	10 times more toxic than As(V)
Arsenite, Inorganic	H_3AsO_3, $H_2AsO_3^{1-}$, $HAsO_3^{2-}$, AsO_3^{3-}	pH = 0-9 pH = 10-12 pH = 13 pH = 14	
Methylated As(III) Organo-As(III)		Minor importance	Less toxic than Inorganic As(III)
Pentavalent arsenic	As(V)	Aerobic	10 times less toxic than As(III)
Arsenate, Inorganic	H_3AsO_4, $H_2AsO_4^{1-}$, $HAsO_4^{2-}$, AsO_4^{3-}	pH = 0-2 pH = 3-6 pH = 7-11 pH = 12-14	
Methylated As(V) Organo-As(V)		Minor importance	Less than Inorganic As(V)

Health Effects of Arsenic

In a population drinking arsenic contaminated water, a great variety of specific as well as non-specific symptoms may be observed. Table 2 lists some of the effects of arsenic reported to be due to exposure through drinking water.

In a recent study by the National Institute of Preventive and Social Medicine (NIPSOM), arsenic related diseases (arsenicosis) have been identified in 37 districts (Ahmad et al., 1998). A total of 6000 cases were identified in 162 villages in the 37 districts, mostly in the rural areas. Three stages of manifestations of chronic arsenicosis were observed in the study (Ahmad et al., 1998), but most of the patients were found in the first and second stages.

The most common presentations were melanosis, keratosis, hyperkeratosis and depigmentation. Cancers were found among 0.8% and actinic keratosis and Bowen's diseases were observed among 3.1% of the cases. It is important to note that the study found that the cases at initial and second stages of toxicity showed improvement when patients stopped taking arsenic contaminated water and increased intake of protein rich food, vitamin A, E and C.

Table- 10.2: Toxicological effects due exposure to high arsenic concentration in drinking water (WHO, 1996, Khan, 1997)

Effect	Symptoms	Remarks
Blackfoot Disease Arsenical dermatosis	Dermal lesion, Peripheral neuropathy Keratosis, Hyperkeratosis, Hyperpigmentaion	May necessitate operation
None specific	Nausea, Abdominal Pain, Diarrhoea, Vomiting, Conjunctivitis, Oedema.	Mainly due to acute intoxication
Pregnancy disorders	Spontaneous abortions, miscarriages	-
Heart Disease	Coarctation of aorta, Cardiovascular disturb.	Among children
Cancer	Bladder, Kidney, Skin & Lungs, Liver & Colon	-
Mortality	-	Mainly due to cancer

Analytical Methods for Measuring Arsenic

The most commonly used methods for detection of arsenic in water include:

1. Inductively coupled plasma (ICP) method
2. Hydride generation atomic absorption spectrophotometric method
3. Graphite furnace atomic absorption spectrophotometric method
4. Hydride generation-scraper-spectrophotometric (SDDC) method
5. Hydride generation-scraper-indicator paper-field kit

The first three methods involve high-cost equipment and provide more accuracy and lower detection limit (minimum detection limit, MDL = 1 µg/L). The last two methods are relatively low cost methods but accuracy of determination is less. In our laboratory we have two atomic absorption spectrophotometers and can measure arsenic concentration following methods (2) and (3).

Inductively coupled plasma (ICP) method

An ICP source consists of a flowing stream of argon gas ionized by applied radio frequency field typically oscillating at 27.1 MHz. The water sample is atomized at temperature about 6000 to 8000 ^{0} K. The light emitted from ICP Hydride generation atomic absorption spectrophotometric method is focused on entrance slit and using radio frequency determines absorbance of arsenic.

Hydride generation atomic absorption spectrophotometric method

This method is also known as Flame Atomic Absorption Spectrophotometric (FAAS) method. In this method arsenic is reduced to gaseous arsine in a reaction vessel. The method is two types: i) manual hydride generation and ii) continuous hydride generation. In manual method zinc is added to speed the reaction whereas continuous in continuous hydride generation no zinc is needed. In continues measurement hydride generator a peristaltic pump is used to meter and mix reagents and a gas–liquid separator unit uses flow of argon to strip out hydrogen and arsine gas.

Graphite furnace atomic absorption spectrophotometric method

Also known as Electro thermal atomic absorption spectrophotometric method (ETAAS). This method is based on the same principle as direct flame atomization but an electrically heated atomizer or graphite furnace replaces the standard burner head. Typically, determinations are made by heating the sample in three or more stages. First, a low current heats the tube to dry the sample. The second, or charring, stage destroys organic matter and volatilizes other matrix components at and intermediate temperature. Finally, a high current heat the tube to incandescence and, in an inert atmosphere, atomizes the element being determined. A photoelectric detector measures the intensity of transmitted radiation. The inverse of the transmittance is related logarithmically to the absorbance, which is directly proportional to the number density of vaporized ground-state atoms.

Preparation of Sample for Arsenic Determination:

Preparation of Sample:

Arsenic is often present in very small concentration in water. Therefore, water samples are often *pre-concentrated* in order to increase the quantity of arsenic in a given volume. Pre-concentration is usually done by acidifying 200 to 500 mililiter water sample and then reducing the volume to about 35 mililiter in a water bath.

Precaution:

Arsenic is often found in groundwater with high concentration of iron (Fe). After extraction, Iron present in groundwater in ferrous (Fe^{2+}) form is converted to ferric (Fe^{3+}) form in contact with atmospheric oxygen and forms ferric hydroxide $Fe(OH)_3$ (s) flocs. Arsenic, especially As(V), is strongly adsorbed onto these ferric hydroxide flocs. Therefore, care should be taken while taking water sample from a bottle in which red coloured ferric hydroxide flocs are visible. It is better to acidify the bottle first (thus dissolving the ferric hydroxide flocs) and then take water sample for pre-concentration.

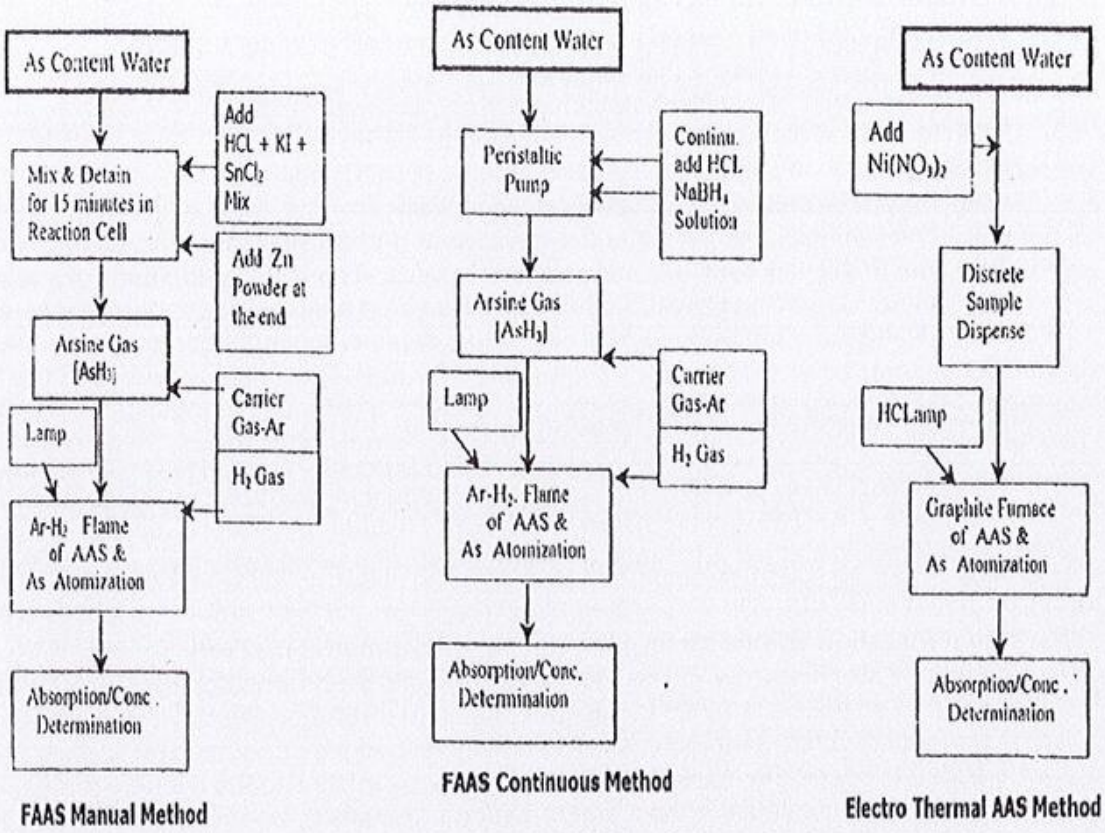


Fig. – 14.2: Different types of Atomic Absorption Spectrophotometric determination of arsenic

Apparatus
- Atomic Absorption Spectrophotometer (AAS)
- Glass tubes, Micro pipette etc.

Reagent
- Nickel nitrate (as matrix modifier)

Procedure
1. Take 9 mL sample and 1 mL nickel nitrate in a container.
2. Draw 20 µL using micro pipette and insert into the hole of graphite tube.
3. A hollow cathode lamp of 193.7 ηm is placed inside the spectrophotometer and switch on the machine. The temperature is raised up to 3000°C to and As atomic cloud is formed (details will be shown in the class).
4. Repeat the procedure and take the average of three measurements.

Calculation
Calculate arsenic concentration using the calibration curve (will be supplied in the class).

Actual As concentration (ppb) = As conc.(ppb) obtained from calibration curve of AAS x D.F.

Where, D.F. = Dilution Factor = $$\frac{\text{volume of sample} + \text{volume of } NiNO_3}{\text{volume of sample(mL)}}$$

Experiment No. 11

Microbiological Quality of Water: Determination of Total Coliform and Fecal Coliform

Introduction

A variety of different microorganisms are found in untreated water. Most of these organisms do not pose a health hazard to humans. Certain organisms, referred to as pathogens, cause disease to humans which include species of bacteria, viruses and protozoa. These organisms are not native to aquatic systems and usually require an animal host for growth and reproduction. Pathogens are likely to gain entrance sporadically, and they do not survive for very long period of time; consequently they could be missed in a sample submitted to the laboratory. Although it is possible to detect the presence of various pathogens in water, the isolation and identification of many of these is often extremely complicated, time-consuming and expensive proposition. Hence in most cases (except when presence of any particular microorganism is suspected) the microbiological quality of water is checked using some *indicator organisms*.

An indicator organism is one whose presence presumes that contamination has occurred and suggests the nature and extent of the contaminants. An indicator organism should be a microorganism whose presence is evidence of fecal contamination of warm blooded animals. Indicators may be accompanied by pathogens, but typically do not cause disease themselves. It is considered that the ideal indicator organisms should have the following characteristics:

- always be present when pathogens are present
- always be absent where pathogens arc absent
- numbers should correlate the degree of pollution
- be present in greater number than pathogens
- there should be no after-growth or re-growth in water
- there should be greater or equal survival time than pathogens
- be easily and quickly detected by simple laboratory tests
- should have constant biochemical and identifying characteristics
- harmless to human

No organisms or group of organisms meet all of these criteria; but the *coliform* bacteria fulfil most of them, and this group is most common indicator used in microbial examination of water.

Total coliforms are grouped into two categories - i) Fecal coliform (thermo-tolerant coliform) and ii) Non-fecal coliform. Total coliforms are defined as gram negative bacteria which ferment lactose at 35 or 37°C, with the production of acid, gas and aldehyde within 24 or 48 hours. Fecal coliforms are a subgroup of total coliforms, which live in the warm blooded animals and have the same properties as the total coliforms but tolerate and grow at the higher selective temperature range of 44 to 44.5°C. In addition, they form indole from tryptophan. And these combined properties, when positive, are regarded as presumptive *Escherichia coli* (presumptive E. coli). Some coliform species are frequently associated with plant debris or may be common inhabitants in soil or surface waters which are called non-fecal coliforms.

Total coliform (TC) = Fecal coliform (FC) + Non-fecal coliform.

Thus, the total coliform group should not be regarded as an indicator of organisms exclusively of fecal origin. The use of total coliform as an indicator may therefore be of little value in assessing the fecal contamination of surface water, unprotected shallow wells etc. where contamination by coliform of non fecal origin also may occur. However, measurement of total coliform is of particular relevance for treated and/or chlorinated water supplies; in this case the absence of total coliform would normally indicate that the water has been sufficiently

treated/ disinfected to destroy various pathogens. Measurement of fecal coliform is a better indicator of general contamination by material of fecal origin. The predominant species of fecal coliform group is *Escherichia coli* (E. coli), which is exclusively of fecal origin, but strains of *Klebsella pneumoniae* and *Enterobacter* species may also be present in contaminated water.

Using coliform as indicators of the presence and absence of pathogens sometimes may cause the following drawbacks:

- False positive result can be obtained from the bacterial *genus aeromonas*- a gram-negative bacteria which resemble coliform group bacteria in their fermentative ability.
- False negative result can be obtained when coliforms are present along with high population of other bacteria. The latter bacteria can act to suppress coliform activity.
- A number of pathogens have been shown to survive longer in natural waters and/ or through various treatment processes than coliform.

But the use of coliform was established first and there does not appear to be any distinct advantages to warrant shifting to other indicator organisms. Since bacteria are used as indicator organisms, the microbiological examination of water is commonly called bacteriological examination.

Methods of Bacteriological Examination of Water

Basically there two methods of bacteriological analysis of water: (a) Multiple Tube or Most Probable Number (MPN) method, and (b) Membrane Filter (MF) method.

The most probable number (MPN) is the number of organisms that are most likely to have produced laboratory results in a particular test. The MPN method is used to quantify the concentration of the viable microorganisms in a sample and involves inoculating decimal dilutions into tubes of a broth medium, observing results and using a standard MPN table. This method is extensively used in food microbiology. In our lab we will perform membrane filter (MF) method which is discussed below.

Membrane Filter Method:

The membrane filter (MF) method gives a direct count of total coliforms and fecal coliforms present in a given sample of water. The method is based on the filtration of a known volume of water through a membrane filter consisting of a cellulose compound with a uniform pore diameter of 0.45μm; the bacteria are retained on the surface of the membrane filter. When the membrane containing the bacteria is incubated in a sterile container at an appropriate temperature with a selective differential culture medium, characteristic colonies of coliforms and fecal coliforms develop, which can be counted directly. This technique is popular with environmental engineers.

This method is not suitable for turbid waters, but otherwise it has several advantages. Its particular advantages and limitations are as follows:

Advantages:

- results are obtained more quickly as the number of coliforms can be assessed in less than 24 hours, whereas the multiple tube technique requires 48 hours both for a negative or a presumptive positive test;
- saving in work, certain supplies and glassware;
- method gives direct results;
- easy to use in laboratories, or even in the field if portable equipment is used.

Disadvantages:

- high turbidity caused by clay, algae, etc. prevents the filtration of a sufficient volume of water for analysis and it may also produce a deposit on the membrane which could interfere with bacterial growth;
- presence of a relatively high non-coliform count may interfere with the count.
- water containing particular toxic substances which may be absorbed by the membranes, can affect the growth of the coliforms.

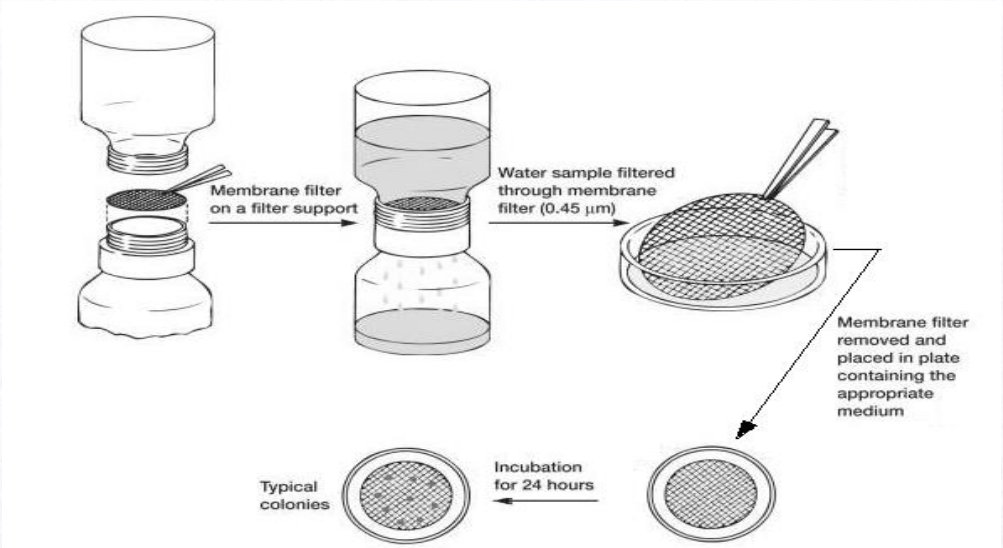


Fig-15.1 : Membrane Filter method

Reagents

- Distilled water
- Appropriate culture mediums (broth)

Apparatus

- Erlenmeyer flask : 1 pc.
- Vacuum pump : 1 set
- Petri Dish : 2 pcs.
- Membrane filter paper (0.45 μm) : 2 pcs.
- Incubator : 2
- Measuring cylinder, beaker, dropper etc.

Test Procedure

This section describes the general procedures. It should be noted that different types of filtration units and equipment are available in the market for performing the tests.

Determination of Total Coliforms (TC):

1. Connect the Erlenmeyer (side-arm) flask to the vacuum source (turned off) and place the porous support in position. If an electric pump is used, it is advisable to put a second flask between the Erlenmeyer and the vacuum source; this second flask acts as a water trap and thus protects the electric pump.
2. Open a Petri-dish and place a pad in it. With a sterile pipette add 2 mL of selective broth (culture) medium to saturate the pad.
3. Assemble the filtration unit by placing sterile membrane filter on the porous support, using forceps sterilized earlier by flaming.
4. Place the upper container in position and secure it with the special clamps. The type of clamping to be used will depend on the type of equipment.
5. Pour the volume of sample chosen as optimal, in accordance with the type of water, into the upper container. If the test sample is less than 10 mL, at least 20 mL of sterile dilution water should be added to the top container before filtration applying the vacuum.
6. After the sample has passed through the filter, disconnect the vacuum and rinse the container with 20-30 mL of sterile dilution water. Repeat the rinsing after all the water from the first rinse has passed through the filter.

7. Take the filtration unit apart and using the forceps, place the membrane filter in the Petri-dish on the pad with the grid side up. Make sure that no air bubbles are trapped between the pad and the filter.
8. Invert the Petri-dish for incubation. Incubate at 35°C or 37°C for 18-24 hours with 100% humidity (to ensure this, place a piece of wet cotton wool in the incubator). If ointment containers or plastic dishes with tight-fitting lids are used, humidification is not necessary.

Bacterial Colony observation:
Colonies of coliform bacteria are a medium red or dark red color, with a greenish gold or metallic surface sheen. This sheen may cover the entire colony or appear only in the centre of the colony. Colonies of other types should not be counted. The colonies can be counted with the aid of a lens. The number of total coliforms per 100 ml is then given by:

Determination of Fecal Coliforms (FC):
The procedure for fecal coliforms is similar to that used for determining total coliforms. Filter the sample as described, and place the membrane filler on the pad saturated with appropriate culture medium.

1. Place the dishes in an incubator at 44±0.5 °C for 24 hours at 100% humidity. Alternatively, tight-fitting or sealed Petri-dishes may be placed in water-proof plastic bags for incubation.
2. Submerge the bags in a water-bath maintained at 44±0.5°C for 24 hours. The plastic bags must be below the surface of the water throughout the incubation period. They can be held down by means of a suitable weight, e.g., a metal rack.

Bacterial Colony observation:
Colonies of fecal coliform bacteria are blue in color. This color may cover the entire colony, or appear only in the center of the colony. Colonies of other types should not be counted. The colonies can be counted with the aid of a lens. The number of fecal coliforms per 100 ml is then given by:

Calculation

$$\text{Total coliform (nos./ 100 mL)} = \frac{\text{no. of coliform colonies counted} \times 100}{\text{mL of sample filtered}}$$

$$\text{Fecal coliform (nos./ 100 mL)} = \frac{\text{no. of coliform colonies counted} \times 100}{\text{mL of sample filtered}}$$

Experiment No. 12

Estimation of Organic Pollution Load through Determination of BOD and COD

Introduction

One of the most important water quality parameters for natural water bodies is the amount of dissolved oxygen (DO) present in the water. Biodegradable organic substances, discharged into water bodies (pond, lake, canal, khal, river, ocean) with wastewater/effluent (domestic and industrial), get oxidized in the receiving body of water (by naturally present microorganisms) reducing the amount of available DO. As DO drops, fish and other aquatic life are threatened, and in extreme cases, killed.

There are several measures to estimate pollution load from biodegradable organic substances. The most common measures are:

(A) Biochemical Oxygen Demand (BOD); and

(B) Chemical Oxygen Demand (COD).

(A) Determination of Biochemical Oxygen Demand (BOD)

When biodegradable organic matter/waste is released into a water body, microorganisms (especially bacteria) feed on the wastes, breaking it down to simpler organic and inorganic substances. When this decomposition takes place in an aerobic environment (i.e., in the presence of oxygen; Eq. 1), it produces non-objectionable, stable end products (e.g., CO_2, SO_4, PO_4, and NO_3) and in the process draws down the dissolved oxygen (DO) of water.

$$\text{Organic matter} + O_2 \xrightarrow[\text{(aerobic bacteria)}]{} CO_2 + H_2O + \text{New cells} + \text{Stable products} \quad \text{-----(1)}$$

When insufficient oxygen is available or when oxygen is exhausted by the aerobic decomposition of wastes, then different set of microorganisms carry out the decomposition under anaerobic condition (Eq. 2) producing highly objectionable products including H_2S, NH_3, and CH_4.

$$\text{Organic matter} \xrightarrow[\text{(anaerobic bacteria)}]{} CO_2 + CH_4 + \text{New cells} + \text{Unstable products} \quad (2)$$

The amount of oxygen required by micro-organisms to oxidize organic wastes aerobically is called *biochemical oxygen demand (BOD)*; usually expressed in mg of oxygen required per liter of water/wastewater (mg/L).

According to Bangladesh Environment Conservation Rules (2023), drinking water standard for biochemical oxygen demand (BOD_5) is 0.2 mg/L (at 20°C) and standards for BOD_5 in industrial wastewater/effluent discharge is as followings:

BOD_5 at 20°C	Discharge into Inland Water	In Sewer for Secondary Treatment	Discharge near Coastal Area
	30	250	100

Modeling of BOD

It is often assumed that the rate of decomposition of organic waste is proportional to the amount of waste available. If L_t represents the amount of oxygen demand left after time t, then, assuming a first order reaction, we can write,

$$dL_t/dt = -k L_t \quad (3)$$

Where, k = BOD reaction rate constant (time^{-1}).

Integrating, we get, $L_t = L_o e^{-kt}$ (4)

Where, L_o= the ultimate carbonaceous oxygen demand, which is the total amount of oxygen required by microorganisms to oxidize the carbonaceous portion of the waste.

Now, $L_o = BOD_t + L_t$ (5)

After combining, we get, $BOD_t = L_o (1 - e^{-kt})$ (6)

Fig.-10.1(a) and 10.1(b) shows the graphical representations of Eq. (4) and Eq. (6), respectively.

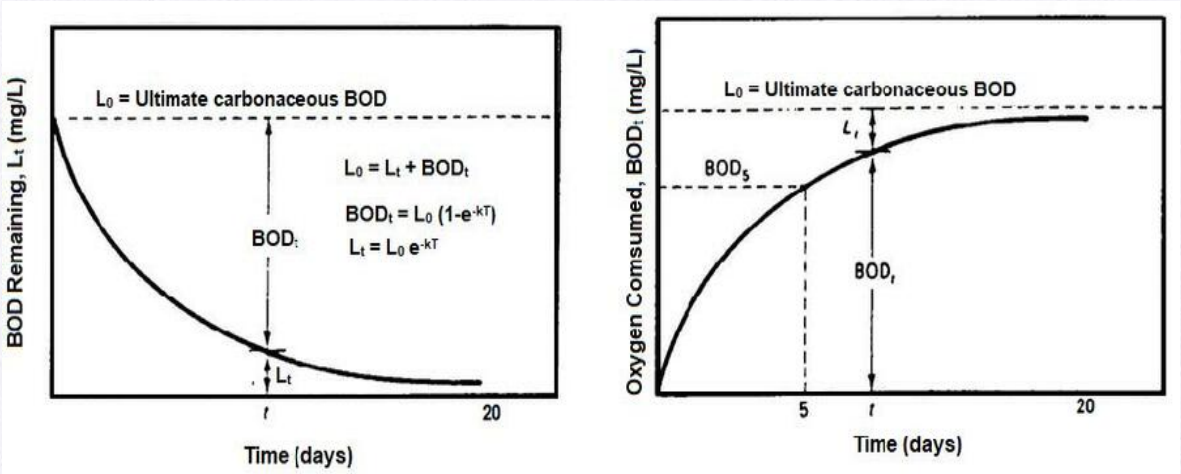


Fig.- 1.1: Idealized carbonaceous oxygen demand: (a) the BOD remaining as a function of time, and (b) the oxygen consumed or the biochemical oxygen demand as a function of time.

Besides the oxidation of carbonaceous organic matter, a significant additional demand may be caused by the oxidation of nitrogenous compounds. To distinguish this *nitrogenous biochemical oxygen demand* from the *carbonaceous biochemical oxygen demand*, the two are sometimes referred to as NBOD and CBOD, respectively. Fig.- 2 illustrates the two demands for a typical municipal waste. Note that the NBOD does not begin to exert itself for at least 5 to 8 days, so most 5-day BOD tests are not affected by the nitrification process.

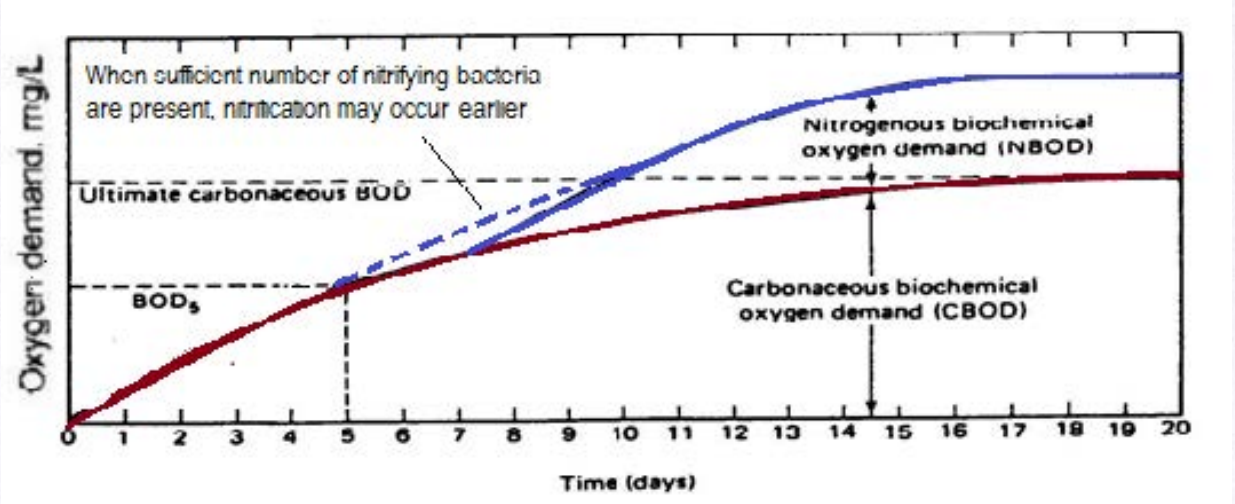


Fig.-1.2: Illustration of Carbonaceous and Nitrogenous Biochemical Oxygen Demand

Theory on Experimental Method

The oxygen demand for decomposition of biodegradable organic matter is a function of time. It is a standard practice to measure and report the oxygen demand over a 5-day period, realizing that the ultimate oxygen demand (for complete degradation of organic matter) is considerably higher and would take a much longer time to determine in the laboratory.

The 5-day BOD or BOD_5 is the amount of oxygen consumed by microorganisms during the first 5 days of biodegradation. The test is usually carried out at 20°C. In its simplest form, a BOD_5 test would involve putting a sample of waste/wastewater into a bottle, measuring the dissolved oxygen(DO) of the sample at the beginning of the test and again at the end of five days. The difference in DO would be the BOD_5 of the water /wastewater. Light must be kept out of the bottle to keep algae from adding oxygen by photosynthesis and the stopper is used to keep air from replenishing DO from outside.

Dilution of wastewater:

Since the oxygen demand of typical wastewater/effluent is several hundred mg/L, and since the saturated value of DO for water at 20°C is only 9.1 mg/L, it is usually necessary to dilute the sample to keep final DO above zero. If during the five days of experiment, the DO drops to zero, then the test is invalid since more oxygen would have been removed had more been available.

The five-day BOD of a diluted sample is given by

$$BOD_5 = [DO_i - DO_f] \times D.F. \qquad (7)$$

where,

$$DF = \text{Dilution Factor} = \frac{\text{volume of (wastewater, Vw + dilution water, Vd)}}{\text{volume of wastewater, Vw}} = \frac{Vm}{Vw}$$

here, $V_m = V_w + V_d$

A wide variety of dilution waters have been used for BOD work. Natural surface waters have been used, but they suffer from a number of disadvantages including variable BOD, variable micro-organism population, and variable mineral content. Tap water has also been used, but it also suffers from many of the limitations of surface water plus the possibility of toxicity from chlorine residuals. Synthetic dilution water prepared by adding different salts to distilled water has been found to be the most ideal for use as dilution water.

Seeding of wastewater:

In some cases, it becomes necessary to *seed* the dilution water with microorganisms to ensure that there is an adequate bacterial population to carry out the biodegradation. In such cases, two sets of BOD bottles must be prepared, one for just the seeded dilution water (called the "blank") and the other for the mixture of wastewater and dilution water. The changes in DO in both bottles are measured. The oxygen demand of the wastewater (BOD_w) is then determined from the following relationship:

$$BOD_m V_m = BOD_w V_w + BOD_d V_d \qquad (8)$$

Where, BOD_m, BOD_w, & BOD_d represent the BOD of the mixture of wastewater and dilution water, BOD of the wastewater &BOD of the dilution water alone respectively.

Rearranging and simplifying equation-8, BOD_w can be obtained –

$$BOD_w = BOD_m \times DF - BOD_d \times (DF-1) \qquad (9)$$

Different types of microorganisms have been used for "seeding" purpose. Experience has shown that domestic wastewater, particularly from combined sewer systems provides a well-

balanced population of mixed micro-organisms. Usually 2 mL of wastewater per liter of diluted water is sufficient. Some river waters are also satisfactory, but care must be taken to avoid using waters that contain algae or nitrifying bacteria in significant amount. The dilution water should always be "seeded" to ensure a uniform population of micro-organisms.

Aeration of wastewater:

Aeration is a treatment method to increase dissolved oxygen concentration in water /wastewater. For organic waste loading analysis aeration is used serve the following two purposes:

1. ***To increase DO in water for determination of BOD:***

Polluted water and wastewater often contain very little DO. For determination of BOD, the initial DO of the sample needs to be raised, so that at the end of 5-day incubation period, some DO remains in water (to be measured). This is usually carried out through "aeration" (i.e. flow of air) of the dilution water, which increases DO of the dilution water by allowing oxygen present in air to dissolve into it.

2. ***Aeration as a means to reduce BOD:***

Aeration is a common technique in wastewater treatment for reduction of BOD. Through aeration (i.e., supply of excess air into the wastewater), the decomposition/degradation of organic matter present in wastewater (by bacteria) is accelerated, thereby reducing its BOD. Aeration ensures activity of aerobic bacteria, which has been identified to metabolize organic waste load in the water faster than the anaerobic bacteria. In this class, the possible effect of simple aeration on BOD would be assessed.

Theory of DO Determination

Dissolved oxygen (DO) determination is done by *Winkler Method.* The reactions involved in the various steps are presented below:

Manganous sulfate reacts with potassium hydroxide in the alkaline potassium iodide solution to produce a white precipitate of manganous hydroxide.

$$MnSO_4 + 2KOH = \underset{\text{white ppt}}{Mn(OH)_2\downarrow} + K_2SO_4 \qquad (10)$$

If the white precipitate is obtained, there was no dissolved oxygen in the sample and there is no need to proceed further. A brown precipitate shows that oxygen is present and reacted with the manganous hydroxide and forms manganic basic oxide.

$$2Mn(OH)_2 + O_2 = \underset{\text{brown ppt}}{2MnO(OH)_2\downarrow} \qquad (11)$$

Upon the addition of (sulfuric) acid, this precipitate is dissolved, forming manganic sulfate:

$$MnO(OH)_2 + 2H_2SO_4 = Mn(SO_4)_2 + 3H_2O \qquad (12)$$

This compound immediately reacts with potassium iodide, liberating iodine and resulting in the typical iodine (blue) coloration of the water.

$$Mn(SO_4) + 2KI = MnSO_4 + K_2SO_4 + I_2 \qquad (13)$$

The quantity of iodine liberated by these reactions is equivalent to the quantity of oxygen present in the sample. The quantity of iodine is determined by titrating a portion of the solution with a standard solution of sodium thiosulfate solution. The end point of titration is indicated by the disappearance of blue color produced by the reaction between iodine and starch (which is added as an indicator in the titration).

$$2Na_2S_2O_3 + I_2 = Na_2S_4O_6 + 2NaI \qquad (14)$$

Reagents

- Manganous sulfate solution
- Alkaline potassium iodide solution
- 0.025N sodium thiosulfate
- Starch solution (indicator)
- Concentrated sulfuric acid.

Apparatus

- BOD bottle : 6 nos.
- Beaker (250 ml) : 1 no.
- Measuring cylinder : 1 no.
- Dropper : 1 no.
- Stirrer : 1 no.

Procedure: *(Determination of DO)*

BOD_5 is to be determined for two types of sample –

i) Wastewater being aerated for a specified period of time and
ii) Non-aerated wastewater. Both samples may be diluted, if needed.

For each type of wastewater (aerated and non-aerated) fill two BOD bottles with sample (or diluted sample); the bottles should be completely filled. Determine initial DO (DO_i) in one bottle for both type of wastewater immediately after filling with sample (or diluted sample). Keep the other set of bottles in dark at 20°C and after particular days (usually 5-days) determine DO (DO_f) in the samples (or diluted samples). Dissolved oxygen (DO) is determined according to the following procedure:

1. Add 1 mL of manganous sulfate solution to the BOD bottle by means of pipette, dipping in end of the pipette just below the surface of the water.
2. Add 1 mL of alkaline potassium iodide solution to the BOD bottle in a similar manner.
3. Insert the stopper and mix by inverting the bottle several times.
4. Allow the "precipitates" to settle halfway and mix again.
5. Again allow the "precipitates" to settle halfway.
6. Add 1 mL of concentrated sulfuric acid. Immediately insert the stopper and mix as before.
7. Allow the solution to stand at least 5 minutes.
8. Withdraw 100 mL of solution into an Erlenmeyer flask and immediately add 0.025N sodium thiosulfate drop by drop from a burette until the yellow color almost disappears.
9. Add about 1 mL of starch solution and continue the addition of the thiosulfate solution until the blue color just disappears. Record the ml. of thiosulfate solution used (disregard any return of the blue color).

Calculation

Dissolved oxygen, DO (mg/L) = mL of 0.025N sodium thiosulfate added x MF

$$\text{Where, MF} = \frac{\text{normality of } N_2S_2O_3 \times \text{equivalent wt. of oxygen} \times 1000}{\text{mL of sample taken}}$$

Calculate:

(a) BOD_5 of the non-aerated wastewater sample (domestic sewage, as received), according to Eq. 3, 4 or 5.
(b) BOD_5 of the aerated wastewater according to Eq. 3, 4 or 5.
(c) If this wastewater is discharged into a river at a rate of 4.6 cfs over a period of 16 hrs per day, estimate the daily BOD_5 load (in kg/day) discharged into the river.

(B) Determination of Chemical Oxygen Demand (COD)

All organic substances are not degradable by microorganism (biologically inert substances). If waste/ wastewater having considerable amount of biologically inert organic matter is tested for BOD, the result would not represent the actual organic loading of the waste/ wastewater. Hence, the chemical oxygen demand (COD) test allows measurement of oxygen demand of the waste in terms of the total quantity of oxygen required for oxidation of the waste to carbon dioxide and water. The test is based on the fact that all organic compounds, with a few exceptions, can be oxidized by the action of strong oxidizing agents under acid conditions.

$$\text{Organic matter} + \text{Chemical Oxidizing agent} = CO_2 + H_2O \qquad (1)$$

The reaction in eq.-1 involves conversion of organic matter to carbon dioxide and water regardless of the biological assimilability of the substance. For example, glucose and lignin (biologically inert substance) are both oxidized completely by the chemical oxidant. As a result, COD values are greater than BOD values, especially when biologically resistant organic forms are present.

Thus one of the chief limitations of COD test is its inability to differentiate between biodegradable and non-biodegradable organic matter. In addition, it does not provide any evidence of the rate at which the biologically active material would be stabilized under conditions that exist in nature.

The major advantage of COD test is the short time required for evaluation. The determination can be made in about 3 hours rather than the 5-days required for the measurement of BOD. For this reason, it is used as a substitute for the BOD test in many instances.

Environmental Significance

COD is often measured as a rapid indicator of organic pollutant in water. It is normally measured in both municipal and industrial wastewater treatment plants and gives an indication of the efficiency of the treatment process. COD has further applications in power plant operations, chemical manufacturing, commercial laundries, pulp & paper mills, environmental studies and general education.

According to Bangladesh Environment Conservation Rules (2023), drinking water standard for COD is 4.0 mg/L. For wastewater effluent discharges, allowable concentration of COD varies from 200, 400 & 250 mg/L depending on discharge point of the effluent e.g., inland water, public sewer & near coastal area respectively (see appendix).

Theory on Experimental Method

Potassium dichromate or potassium permanganate is usually used as the oxidizing agent in the determination of COD. In this class potassium permanganate would be used in the determination of COD. Potassium permanganate is selective in the reaction and attacks the carbonaceous and not the nitrogenous matter.

In any method of measuring COD, an excess of oxidizing agent must be present to ensure that all organic matter is oxidized as completely as possible within the power of the reagent. This requires that a reasonable excess be present in all samples. It is necessary, therefore, to measure the excess in some manner so that the actual amount can be determined. For this purpose, a solution of a reducing agent (e.g., ammonium oxalate) is usually used.

Reagents

- Diluted sulphuric acid solution
- Standard potassium permanganate solution
- Standard ammonium oxalate solution
- COD reagent vial

Apparatus

- Beaker (250 mL) : 1 no

- Dropper : 1 no
- burette : 1 no
- Stirrer : 1 no
- Water bath : 1 no
- Burner : 1 no
- COD reactor : 1 no

Procedure (COD measurement using $KMnO_4$)

1. Pipette 100 mL of the sample into a 250 mL Erlenmeyer flask.
2. Add 10 mL of diluted sulfuric acid and 10 mL of standard $KMnO_4$ solution.
3. Heat the flask in a boiling water bath for exactly 30 minutes, keeping the water in the bath above the level of the solution in the flask. The heating enhances the rate of oxidation reaction in the flask.
4. If the solution becomes faintly colored, it means that most of the potassium permanganate has been utilized in the oxidation of organic matter. In such a case, repeat the above using a smaller sample diluted to 100 mL with distilled water.
5. After 30 minutes in the water bath, add 10 mL of standard ammonium oxalate $[(NH_4)_2C_2O_4]$ solution into the flask. This 10 mL ammonium oxalate, which is a reducing agent, is just equivalent to the 10 mL potassium permanganate (oxidizing agent) added earlier. The excess of reducing agent $[(NH_4)_2C_2O_4]$ now remaining in the flask is just equivalent to the amount of the oxidizing agent ($KMnO_4$) used in the oxidation of organic matter.
6. The quantity of ammonium oxalate remaining in the flask is now determined by titration with standard potassium permanganate. Titrate the content of the flask while hot with standard potassium permanganate to the first pink coloration. Record the mL of potassium permanganate used.

Determination of COD using potassium dichromate in the COD reactor

In this method COD is measured by oxidizing organic wastes using $K_2Cr_2O_7$ as oxidizing agent. COD reagent vials contain premeasured solutions of sulfuric acids and potassium dichromate. The measurement procedure is as follows:

i) Turn on the COD reactor and set to 150°C.
ii) Shake the sample and add 2 mL of sample into COD vial (high range or low range) and replace the cap.
iii) Once the reactor is at the proper temperature, place the vial containing sample in the reactor and set the timer for two hours.
iv) After two hours remove the vial from the reactor and allow cooling to room temperature.
v) Set the spectrophotometer to the appropriate wavelength (620 ηm) and read the absorbance.

Calculations

COD (Using $KMnO_4$)

COD (mg/L) = mL of $KMnO_4$ added in Step-6 x MF

$$\text{Where, MF} = \frac{\text{normality of } KMnO_4 \times \text{equivalent wt. of oxygen} \times 1000}{\text{mL of sample used}}$$

(Normality of $KMnO_4$ used in the lab is 0.0125 N)

Calculate: for both $KMnO_4$ & $K_2Cr_2O_7$ oxidizing agent

(a) COD of the wastewater sample (domestic sewage, as received).

(b) COD of the same wastewater, but which has been aerated for a specified period of time

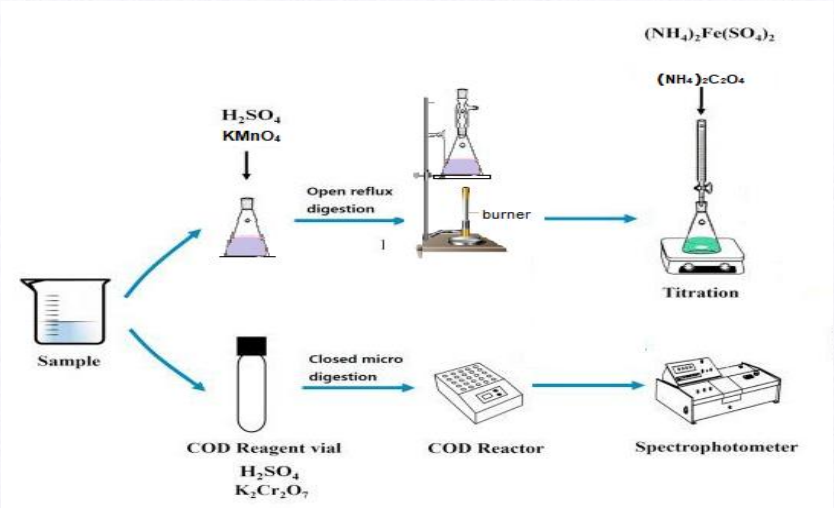


Fig-1.3: COD measurement using $KMnO_4$ & $K_2Cr_2O_7$ as oxidizing agent

Experiment No. 13

Chemical Coagulation of Water: Alum Coagulation

Introduction

All waters, especially surface waters contain dissolved, suspended particles and/or inorganic matter, as well as several biological organisms, such as bacteria, algae or viruses. This material must be removed because it can affect the water quality that can cause turbidity and color. In general, suspended particles are simply removed by conventional physical treatment like sedimentation and filtration. Dissolved molecules cannot be removed by conventional physical treatment. But colloidal elements are difficult to separate from water because the particles will not settle by gravity and are so small that they pass through the pores of most common filtration media. Table-1 shows estimated time (using Stoke's law) required to settle 1 m through water for different types of particles. Thus, the removal of colloids is the main objective and the most difficult aspect in conventional water treatment.

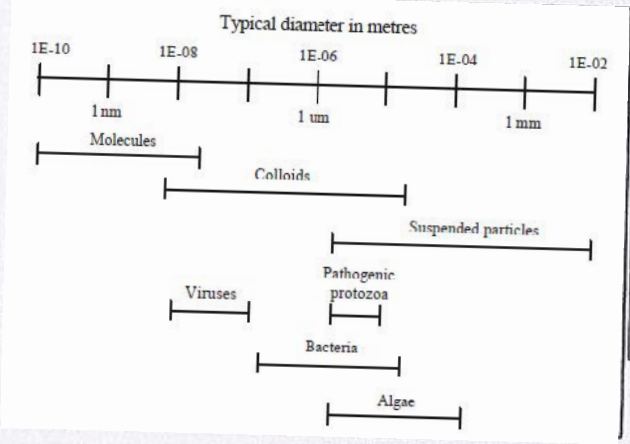


Fig-12.1: Size range of particles of concern in water treatment

Table-1: Time required for settling 1 m through water according to particle size

Particle diameter (cm)	Settling speed (cm/sec)	Time required to settle 1 m
0.05 (Sand)	10.4	10 Sec
0.01 (Fine Sand)	0.42	4 Minutes
0.001 (Silt, Clay)	0.0042	7 Hours
0.0001 (Bacteria)	0.000042	28 Days

There are two types of colloids: hydrophilic colloids and hydrophobic colloids. Hydrophobic colloids, including clay and non-hydrated metal oxides, are unstable. The colloids are easily destabilized. Hydrophilic colloids like soap- are stable. When these colloids are mixed with water, they form colloidal solutions that are not easily destabilized. Most suspended solids smaller than 0.1 mm found in waters carry negative electrostatic charges. Since the particles have similar negative electrical charges and electrical forces to keep the individual particles separate, the colloids stay in suspension as small particles.

To remove colloids, small particles have to be destabilized first and then they will form larger and heavier flocs which can be removed by conventional physical treatment. Chemical agents (coagulants) are induced in water and the positive electric charge of the coagulant reduces the negative charge of the colloids. Thus the process of destroying the stabilizing forces in colloidal particles is referred as chemical coagulation and the aggregation of colloids into larger unit is defined as flocculation. Chemical coagulation-flocculation is a treatment method widely used for removal of small sized and colloidal impurities from water.

Selection of correct chemical dose is very important for this treatment process. At higher coagulant doses, "charge reversal" is possible which may result in re-suspension of the colloids. Hence optimum coagulant doses are determined through laboratory model tests where the water to be treated are subjected to a range of doses of a coagulant and the removal efficiencies are observed.

Environmental Significance

Besides efficient removal of turbidity and color, coagulation with alum and ferric chloride or ferric sulphate is also widely used for removal of heavy metal ions (e.g., lead, arsenic) from water. In this process heavy metal ions are primarily removed by adsorption (and subsequent precipitation) onto coagulated flocs of metal (either aluminum or iron) hydroxides. Coagulation with alum and ferric chloride/sulphate are being successfully used for removal of arsenic from water.

Mechanism of Coagulation Process

The chemical used for this purpose is called are called *coagulants*. The most common *coagulants* used in water and wastewater treatment are aluminum and ferric salts such as alum, ferric chloride and ferric sulfate. Alum (aluminum sulfate) is a good coagulant for water containing appreciable organic matter. Once dissolved in water, aluminum forms hydroxo-complexes and solids [e.g., $Al(OH)_3(s)$, $Al(OH)^{2+}$, $Al(OH)_2^{+}$, $Al(OH)_4^{-}$; [eqs.1-5] and as a result pH of water is lowered, especially if alkalinity of water is low. Theoretically, each mg/L of alum consumes approximately 0.50 mg/L (as $CaCO_3$) of alkalinity. For water with low alkalinity, this may result in significant reduction in pH that may interfere with formation of aluminum hydroxide flocs. If the alkalinity is insufficient, coagulant aids such as lime [$Ca(OH)_2$], soda ash (Na_2CO_3), activated silica and poly-electrolytes are used to provide the necessary alkalinity.

Iron coagulants (e.g., Ferric Chloride) can be operated over a wider pH range and are generally effective in removing turbidity and color from water. However, they are usually more costly.

$$Al_2(SO_4)_3 \cdot 14H_2O \text{ (alum)} = 2\,Al^{3+} + 3\,SO_4^{2-} \quad (1)$$

$$Al^{3+} + 3\,H_2O = Al(OH)_3\,(s) + 3\,H^{+} \quad (2)$$

$$Al^{3+} + H_2O = Al(OH)^{2+} + H^{+} \quad (3)$$

$$Al^{3+} + H_2O = Al(OH)_2^{+} + 2H^{+} \quad (4)$$

$$Al^{3+} + H_2O = Al(OH)_4^{-} + H^{+} \quad (5)$$

Stability of Colloids

The magnitude of the zeta potential (Zp) is usually used to indicate colloidal particle stability. The higher the zeta potential, the greater are the repulsion forces between the colloidal particles and, therefore, the more stable is the colloidal suspension. Low Zp indicates relatively unstable systems, i.e. particles tend to aggregate.

In stable condition, negatively charged colloid is surrounded by a bound layer of water in which ions of opposite charge (counter-ion, i,e.+ve) drawn from the bulk solution with greater concentration near surface of the colloid. This arrangement produces a net charge that is strongest at the bound layer and decreases exponentially with distance from the colloid. When two colloids come in close proximity there are two forces acting on them.

(i)Electrostatic Repulsion:

An electrostatic potential is created by the counter ions surrounding each colloid. As two similarly charged particles approach each other, their diffuse counter-ion atmospheres begin to interfere and cause the particles to be repulsed.

(ii) The Vander Waals' forces of Attraction:

The second force, an attractive force called the Vander Waals force, support contact. This force is inversely proportional to the sixth power of the distance between the particles and also decays exponentially with distance. It decreases more rapidly than electrostatic potential, but is a stronger force at close distances.

The sum of the two forces as they relate to one colloid in close proximity to another is repulsive at greater distances and becomes attractive only after passing through a maximum net repulsive force, called the 'energy barrier' at some distance between colloids. Once the force becomes attractive, contact between the particles takes place.

Destabilization of Colloidal Dispersion
Particle destabilization can be achieved by four mechanisms:

(a) Change characteristics of medium- Compression of double layer.
The ionization of alum in water produces sulfate (SO42-) and aluminium (Al3+) ions. The Al3+ reacts immediately with water to form a variety of hydroxo-complexes (see Eq. 1-5). These ions become part of the ionic cloud surrounding the colloids. A high concentration these multivalent cations reduces the depth of "ionic layer" surrounding each colloid by compressing the layer toward the surface of the colloid. If the ionic layer is sufficiently compressed, then Vander Waals' attractive forces become predominant and the net force becomes attractive (see Fig.12.2).

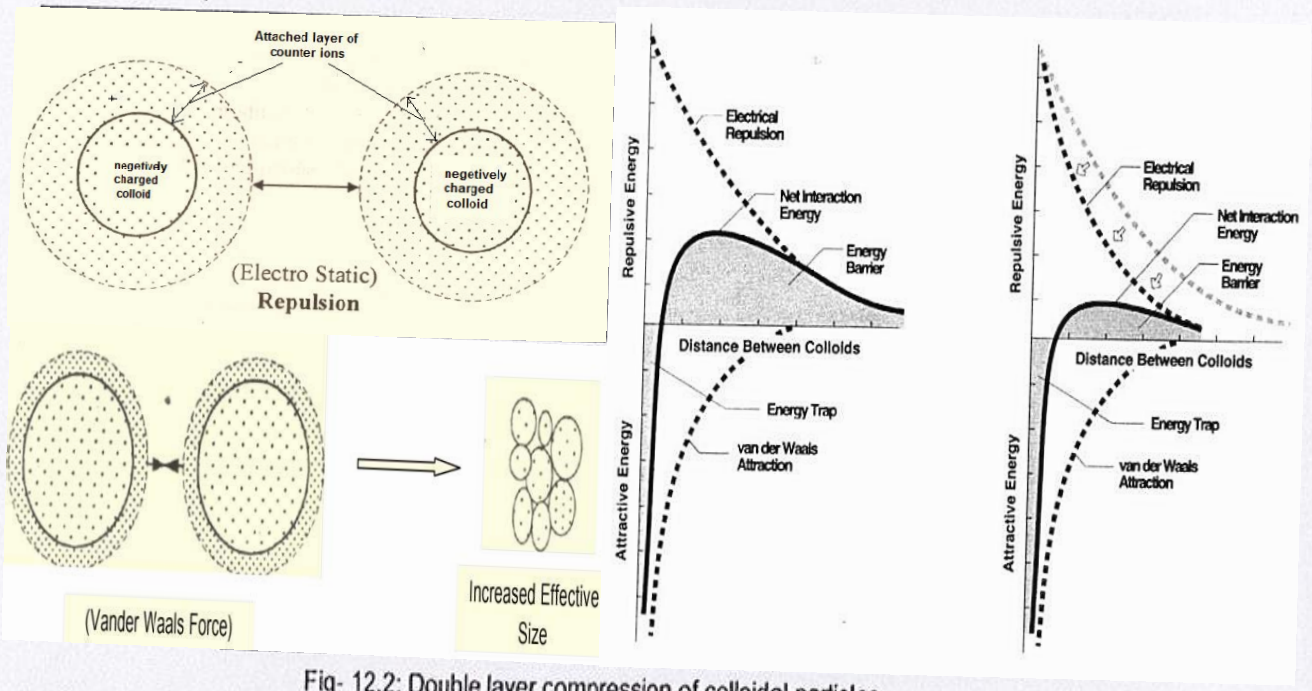


Fig- 12.2: Double layer compression of colloidal particles

(b) Change characteristics of colloid particles-Adsorption and charge neutralization.
Some chemical species are capable of being adsorbed at the surface of colloidal particles. Once the surface charge is neutralized, the ionic cloud dissipates, contact among particles occurs freely. Reduction of surface charge by adsorption is a much different mechanism than reduction by double layer compression. At higher coagulant doses, "charge reversal" (i.e., particles becomes positively charged due to adsorption of positively charged ions, and thus repulsing each other) is possible which may result in re-suspension of the colloids.

(c) Enmeshment in a precipitate: Sweep Floc
If certain metal salts are added to water or wastewater in sufficient amounts, rapid formation of precipitates will occur. Colloids may serve as condensation nuclei for these precipitates or may become enmeshed as the precipitates settle. Removal of colloids in this manner is frequently referred to as sweep-floc coagulation. At low colloid concentrations a large excess of coagulant is required to produce a large amount of precipitate that will enmesh the relatively few colloidal particles as it settles. At high colloid concentrations, coagulation will occur at a lower chemical dosage because the colloids serve as nuclei to enhance precipitate formation.

(d) Adsorption and inter-particle bridging
Natural organic polymers (e.g., starch, cellulose) and some synthetic organic polymeric compounds have large molecular size. They may be anionic, cationic or non-ionic. Polymer molecule gets attached to a colloidal particle at one or more sites due to columbic attraction

(if the polymer and particle are opposite charge) or due to ion- exchange, Vander waals forces (if the polymer and particle are similar charge). The "tail" of the adsorbed polymer will extend out into the bulk solution and can become attached to vacant site on the surface of another particle to form a chemical bridge. The bridging action results in the formation of a floc particle having favorable settling characteristics.

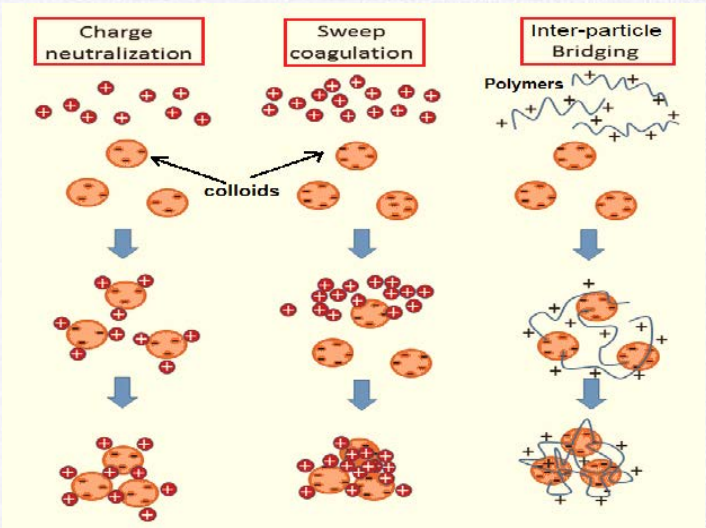


Fig- 12.3: Charge neutralization, Sweep floc & Inter-particle bridging

Experimental Setup for Determination of Coagulation Dose

In the class "jar test" will be used to determine optimum coagulant dose for removal of suspended solids from water.

Reagents

- Standard Alum solution.

Apparatus

- Jar test apparatus
- pH meter
- Turbidity meter
- Glass beakers (1000 mL- 6 nos)

Procedure

1. Determine pH and turbidity of the water to be treated.
2. Fill six 1-litre capacity beakers each with 500 mL of water to be treated.
3. Add required (as instructed in the Lab) coagulant dose (standard alum solution) to each beaker.
4. Stir the beakers with glass stirrer for one minute to mix the coagulant thoroughly in water.
5. Place six beakers on a "jar test" apparatus (Fig. 12.4). Subject the samples to one minute of rapid mixing (about 40 rpm) followed by 15 minutes of slow mixing (about 25 rpm).

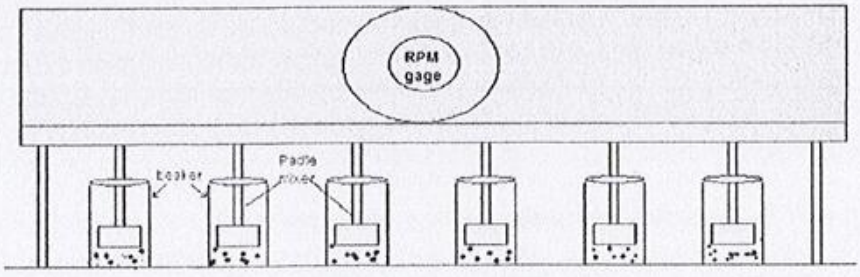


Fig- 12.4: Jar test apparatus

6. Allow the flocs to settle down for about 15 minutes. Observe the characteristics of the flocs and the settling rates. Also observe which of the 6 beakers is most clear.
7. Collect the supernatant from each beaker and measure pH and turbidity of each sample.
8. Plot pH versus alum dose in a graph paper and observe effect of alum dose on pH.
9. Plot turbidity (NTU) versus the coagulant (alum) dose (mg/L) in a graph paper. Determine optimum dose of alum from this plot.

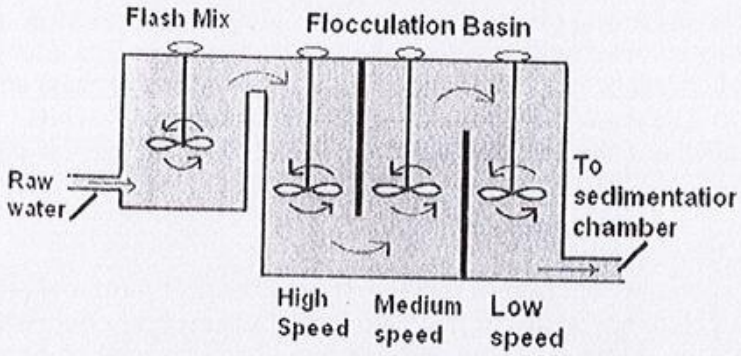


Fig- 12.5: Typical arrangement in a water treatment plant for removal of suspended solids (including colloidal particles) from water by coagulation-flocculation and subsequent sedimentation process

Experiment No. 14

Determination of Residual Chlorine and Chlorine Demand: Break Point Chlorination

Introduction

Chlorination of public water supplies and polluted waters serves primarily to destroy or de-activate disease-producing microorganisms. Disinfection with chlorine is widely practised. Chlorination may produce some adverse effects including taste and odor problem. In recent years, chlorination has been found to produce trihalomethanes (THMs) and other organics of health concern (THMs are suspected human carcinogens). Thus, use of alternative disinfectants, such as chlorine dioxide and ozone that do not cause this particular problem, is increasing.

Theory of Chlorination

Disinfectant capabilities of chlorine depend on its chemical form in water, which in turn is dependent on pH, temperature, organic content of water, and other water quality factors. Chlorine is used in the form of free chlorine [e.g., chlorine gas] or as hypochlorites [e.g., NaOCl and $Ca(OCl)_2$]. Chlorine applied to water either as free chlorine or hypochlorite initially undergoes hydrolysis to form free chlorine consisting of aqueous molecular chlorine, hypochlorous acid and hypochlorite ion.

$$Cl_2 + H_2O = HOCl + H^+ + Cl^- \quad (1)$$

$$NaOCl = OCl^- + Na^+ \quad (2)$$

$$Ca(OCl)_2 = 2OCl^- + Ca^{2+} \quad (3)$$

$$HOCl = H^+ + OCl^- \quad (4)$$

The relative proportion of these "free chlorine" forms is pH and temperature-dependent. Fig.-12.1 shows a distribution diagram for the various chlorine species (Cl_2, HOCl, OCl^-) over a broad pH range

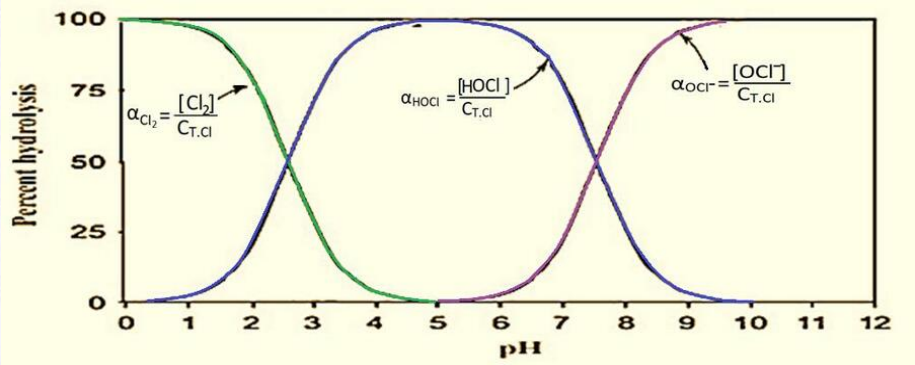


Fig.- 13.1. Distribution diagram for chlorine species at 25°C (Total Chlorine=10^{-3} M)

The diagram above shows that Cl_2 can be significant only at low pH values (below pH 2); while HOCl is dominant between pH 3 and 6. Between pH 6 and 9, the relative fraction of HOCl decreases, while the corresponding fraction of OCl^- increases. The dissociation of HOCl is also temperature dependent. The effect of temperature is such that at a given pH, the fraction of HOCl will be lower at higher temperatures. Generally, the disinfectant capabilities of HOCl are greater than that of OCl^-, especially at short contact times (Montgomery, 1985).

Reactions of Chlorine with Impurities in Water

Reactions with Ammonia:

Free chlorine reacts readily with ammonia and certain nitrogenous compounds to form what are collectively known as "combined chlorine". The inorganic chloramines consist of three species: monochloramine (NH_2Cl), dichloramine ($NHCl_2$) and trichloramine or nitrogen trichloride (NCl_3). The presence and concentrations of these combined forms depend on a number of factors including the ratio of chlorine to ammonia-nitrogen, chlorine dose, temperature, pH and alkalinity.

$$NH_3 + HOCl = NH_2Cl + H_2O \quad (5)$$
$$NH_2Cl + HOCl = NHCl_2 + H_2O \quad (6)$$
$$NHCl_2 + HOCl = NCl_3 + H_2O \quad (7)$$

In addition to chlorinating ammonia, chlorine also reacts to oxidize ammonia to chlorine-free products (e.g., nitrogen gas and nitrate) as shown below.

$$3\,Cl_2 + 2\,NH_3 = N_2(g) + 6\,H^+ + 6\,Cl^- \quad (8)$$
$$4\,Cl_2 + NH_3 + 3\,H_2O = 8\,Cl^- + NO_3^- + 9\,H^+ \quad (9)$$

The mono- and dichloramines have significant disinfecting power and are therefore of interest in the measurement of chlorine residuals. Combined chlorine in water supplies may be formed in the treatment of raw waters containing ammonia; chlorinated wastewater effluents, as well as certain chlorinated industrial effluents normally contain only combined chlorine.

Reactions with Other Impurities:

Chlorine combines with various reducing agents and organic compounds thus increasing the chlorine demand which must be satisfied before chlorine is available to accomplish disinfection.

Fe^{2+}, Mn^{2+}, NO_2^-, and H_2S are examples of inorganic reducing agents present in water supplies that will react with chlorine. Chlorine can react with phenols to produce , which can impart tastes and odor to water. Chlorine also reacts with humic substances present in water to form trihalomethanes (THMs, e.g., chloroform, bromoform, etc.) which are suspected human carcinogens (Note: According to USEPA, maximum allowable level of THMs in drinking water is 100 µg/L).

Break Point Chlorination

If chlorine is added to water containing reducing agents and ammonia (either naturally present or added to water to produce combined chlorine), a hump-shaped breakpoint curve is produced as shown in Fig. – 13.2. The different segment of the curve is described below:

a. If the water is free of ammonia and other compounds that may react with chlorine, the application of chlorine will yield free available chlorine residual of same concentration. This is denoted by the "no demand line" or the "zero demand line".
b. Chlorine first reacts with reducing agents such as H_2S, Fe^{2+}, Mn^{2+} and develops no measurable residual as shown by the portion of the curve from Origin up to point A.
c. Addition of chlorine beyond point A results in forming mainly mono- and di-chloramines. With mole ratios of chlorine to ammonia up to 1:1 [i.e., Cl_2:NH_3-N = 1:1], both mono and di-chloramines are formed. Chloramines thus formed are effective disinfectants and are shown as combined available chlorine residual in Fig. – 13.2 (from A to B).
d. Further increase in the mole ratio of chlorine to ammonia result in formation of some trichloramine and oxidation of part of ammonia to N_2 and NO_3^- (see Eq. 8, 9). These reactions are essentially complete when 1.5 mole of chlorine has been added for each mole of ammonia nitrogen originally present in water [i.e., Cl_2:NH_3-N = 1.5:1]. This is represented by the portion of the curve from B to C (see Fig.13. 2).

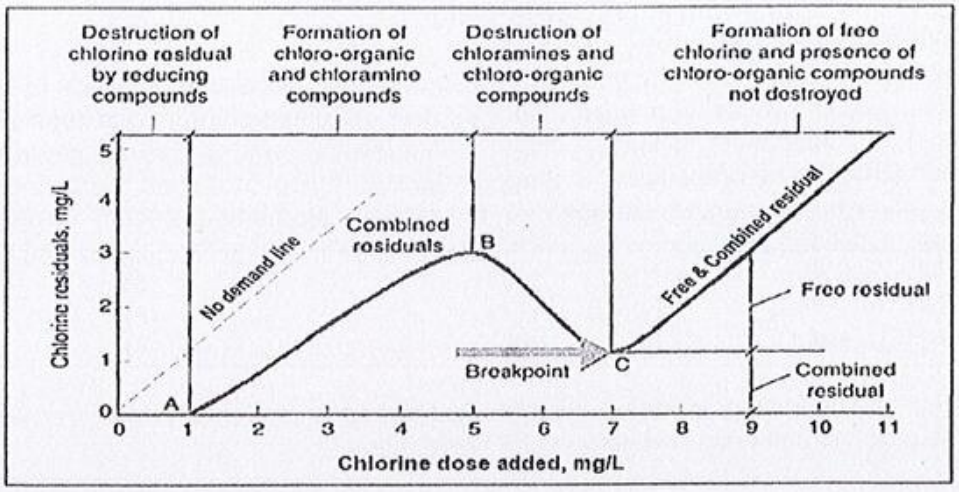


Fig – 13.2: Generalized curve obtained during breakpoint chlorination of a water sample containing ammonia

e. Addition of chlorine beyond point C would produce free chlorine residuals and is referred to as "*breakpoint chlorination*". In other words, chlorination of water to the extent that all ammonia is converted to N_2 or higher oxidation state is referred to as "*breakpoint chlorination*".
f. The difference between the amount of chlorine added to the water and the amount of residual chlorine (i.e., free and combined available chlorine remaining) at the end of a specified contact period is termed as "*chlorine demand*".

Environmental Significance

Chlorine residuals determination is used to control chlorination of domestic and industrial wastewaters. Active chlorine (free and combined) should be determined at each stage in the treatment process of drinking water and in the water mains in order to guarantee bacteriologically impeccable water. While free chlorine residuals have good disinfecting powers, they are usually dissipated quickly in the distribution system. For this reason, final treatment with ammonia is often practiced to convert free chlorine residuals to longer-lasting combined chlorine residuals.

Chlorine determination is important to avoid bad odour and change in the taste of water. It is determined in the swimming pools to avoid ill effects due to excess chlorination. Determination of chlorine residual in water distribution is useful to find the source of contamination or leakage points, so as to supply wholesome water to the consumer.

Reagents

- Starch Indicator
- Standard 0.025 N Sodium thiosulfate
- Potassium Iodine crystal
- Concentrated Acetic Acid
- Bleaching powder stock solution (chlorine water)

Procedure

1. Place 200-mL portion of the water to be chlorinated in each of six 250-mL flasks.
2. Add required quantity (as instructed by your teacher) of "chlorine water" (stock solution of bleaching powder in water) in each of the flasks. The chlorine content of the "chlorine water" (determined earlier in the laboratory) would be provided to you by your teacher. Calculate the chlorine dose for each of the six flasks.
3. Shake each flask gently and allow standing for 30 minutes.
4. Determine residual chlorine of water from each flask by the starch-iodine method as described below:

Determination of Residual Chlorine by Starch-Iodine Titration Method:

The starch-iodine method is based on the oxidizing power of free and combined chlorine residuals to convert iodide ion into free iodine at pH 8 or less, as shown below.

$$Cl_2 + 2I^- = I_2 + 2\,Cl^-$$

In the starch-iodine method, the quantity of chlorine residuals is determined by measuring the amount of iodine in water by titration with a reducing agent - sodium thiosulfate ($Na_2S_2O_3$). The end point of titration is indicated by the disappearance of blue color, produced by the reaction between iodine and starch (which is added as indicator during the titration).

$$I_2 + 2\,Na_2S_2O_3 = Na_2S_4O_6 + 2\,NaI$$

or,

$$I_2 + 2\,S_2O_3^{2-} = S_4O_6^{2-} + 2I^-$$

I_2 + starch $\rightarrow$ blue color

(Qualitative test for the presence of iodine/chlorine)

The titration is carried out at pH 3 to 4, because the reaction with thiosulfate is not stoichiometric at neutral pH due to partial oxidation of the thiosulfate to sulfate.

Determination of Iodine (equivalent to residual chlorine) in water

i) Place 200 mL of the sample in an Erlenmeyer flask.
ii) Add about 2 mL of concentrated Acetic acid to the water.
iii) Add 1g of potassium iodide (estimated on a spatula) and dissolve it by thoroughly mixing it with a stirring rod.
iv) Add 0.025 N sodium thiosulfate drop by drop from a burette until the yellow color of liberated iodine almost disappears. Perform the titration quickly, since iodine liberate faster
v) Add 1 mL of starch solution to the water.
vi) Continue addition of standard sodium thiosulfate solution until the blue color just disappears. (In many cases residual chlorine is very low and starch needs to be added before starting up the titration.)
vii) Record the quantity (in mL) of sodium thiosulfate solution used.

Calculation:

Residual chlorine (mg/L)= mL of 0.025N sodium thiosulfate used x M.F.

$$\text{Where, M.F.} = \frac{\text{normality of } Na_2S_2O_3 \text{ x equivalent wt.of } Cl_2 \text{ x } 1000}{\text{mL of sample taken}}$$

Experiment No. 15

Measurement of Ambient Particulate Air Pollution Concentration: PM2.5 and PM10

Introduction

This laboratory exercise aims to provide hands-on experience in measuring ambient particulate air pollution levels. By the end of this lab, you will be proficient in operating particulate air pollution measuring devices, conducting measurements, and interpreting the significance of the data.

Environmental Significance

According to the World Health Organization (WHO), ambient and household air pollution combine to cause seven million premature global deaths annually, making air pollution the world's leading environmental health risk. Its consequences include elevated mortality rates from stroke, heart disease, chronic obstructive pulmonary disease, lung cancer, and acute respiratory infections. Air pollutants originate from diverse sources, both natural and human-made, such as fossil fuel combustion in electricity generation, transportation, industry, and households, industrial processes, agriculture, waste treatment, and natural events like volcanic eruptions and windblown dust.

Air pollution encompasses gaseous and particulate contaminants. Gaseous pollutants include sulfur dioxide (SO_2), nitrogen oxides ($NOx = NO + NO_2$), ozone (O_3), carbon monoxide (CO), volatile organic compounds (VOCs), toxic air pollutants, and gaseous metal forms. Particulate pollution varies in size and composition, with two extensively studied subsets: $PM_{2.5}$ (fine inhalable particles, <2.5 micrometers) and PM_{10} (inhalable particles, generally <10 micrometers). These particles comprise sulfate, nitrate, elemental carbon, organic carbon, and crustal material.

Air pollutants can be primary or secondary. Primary pollutants are directly emitted, such as particulates, carbon monoxide, and nitrogen oxides from car exhausts. Secondary pollutants form in the atmosphere through chemical reactions, like ground-level ozone created by NOx and VOC reactions in sunlight. Particulate matter, mainly $PM_{2.5}$ and PM_{10}, also forms in complex atmospheric reactions involving sulfur dioxide, nitrogen oxides, and VOCs. Secondary pollutants pose unique challenges due to their variable formation and limited understanding, contributing to issues like photochemical smog.

Criteria Air Pollutants and Air Quality Standards

Criteria air pollutants, also known as criteria pollutants, are a group of common air pollutants that have been designated by government environmental agencies, such as the U.S. Environmental Protection Agency (EPA), as having known adverse effects on human health and the environment. These pollutants are considered "criteria" because they are used as indicators or criteria to assess and regulate air quality, and their concentrations in the atmosphere are often used as a measure of overall air quality. The six common criteria air pollutants identified by the U.S. EPA are: Particulate Matter (PM_{10} and $PM_{2.5}$), Ground-Level Ozone (O_3), Nitrogen Dioxide (NO_2), Sulfur Dioxide (SO_2), Carbon Monoxide (CO), and Lead (Pb)

The Bangladesh Air Pollution Control Rules of 2022 establish ambient air quality standards for various criteria pollutants, as detailed in Table 15.1. Table 15.2 displays the ambient air quality guidelines for common criteria air pollutants recommended by the World Health Organization (WHO).

Air Quality Index (AQI)

Communicating air pollutant concentrations to the public is challenging. A color-coded Air Quality Index (AQI) simplifies this process, covering ozone, particulate matter, carbon monoxide, nitrogen dioxide, and sulfur dioxide. The US-EPA's AQI scale, shown in Table 15.3, ranges from 0 to 500, with higher values indicating worse air quality. For example, 50 or below represents good air quality, while values exceeding 300 indicate hazardous conditions. Six distinct categories, each associated with a specific color, make it easy for people to assess health concerns and air quality quickly.

Theory of Measurement of Pollutant Concentration:

For PM_{10} measurement in Gravimetric Method, an air sampler draws ambient air at a constant flow rate through a specially designed inlet, where suspended particulate matter within the PM_{10} size range is collected on a filter (e.g., Glass microfiber filters; Grade EPM 2000: 2.0µm) during a specified sampling period. Before and after use, each filter is weighed (after moisture equilibration) to determine the net weight (mass) gain attributed to the collected PM_{10}. The total sampled air volume, corrected to reference conditions (25°C, 101.3 kPa), is calculated based on the measured flow rate and sampling time. The PM_{10} mass concentration in ambient air is computed as the total mass of collected particles in the PM_{10} size range divided by the volume of air sampled, expressed in micrograms per standard cubic meter.

For $PM_{2.5}$ measurement in Gravimetric Method, an air sampler draws ambient air at a constant volumetric flow rate through a specially designed inlet and an inertial particle size separator (impactor). The separator isolates suspended particulate matter in the $PM_{2.5}$ size range, which is then collected on a polytetrafluoroethylene (PTFE) filter during the specified sampling period. Before and after sample collection (after moisture and temperature conditioning), each filter is weighed to determine the net mass gain attributed to the collected $PM_{2.5}$. The total sampled air volume is determined by the sampler using the measured flow rate at the actual ambient temperature and pressure and the sampling time. The $PM_{2.5}$ mass concentration in ambient air is calculated as the total mass of collected particles in the $PM_{2.5}$ size range divided by the actual volume of air sampled, expressed in micrograms per cubic meter of air.

To calculate the AQI value for a measured pollutant concentration, first, identify the two breakpoints from Table 15.3 that encompass the concentration. For instance, if you have a measured PM2.5 concentration of 30 µg/m³, it falls between the breakpoints of 12.1 and 35.4 µg/m³ in Table 15.3. Next, use the equation provided in Table 15.3 (simple linear interpolation) to calculate the index and round it to the nearest integer. AQI values exceeding 500 are classified as 'Beyond the AQI' and are treated similarly to the Hazardous category. When calculating AQI for multiple pollutants, the highest value can be reported as the overall AQ.

Table -15.1: Bangladesh Standard for Ambient Air Quality (as per Bangladesh Air Pollution Control Rules, 2022)

তফসিল ১

পরিবেষ্টক বায়ুর মানমাত্রা (**Ambient Air Quality Standards**)

[বিধি ৩ এর উপ-বিধি (১) এবং বিধি ৫ এর উপ-বিধি (১) দ্রষ্টব্য]

বায়ুদূষক	মানমাত্রা	গড় সময়
কার্বন মনোঅক্সাইড (CO)	০৫ মিলিগ্রাম/ঘনমিটার*	৮ ঘণ্টা
	২০ মিলিগ্রাম/ঘনমিটার*	১ ঘণ্টা
লেড (Pb)	০.২৫ মাইক্রোগ্রাম/ঘনমিটার*	বার্ষিক
	০.৫০ মাইক্রোগ্রাম/ঘনমিটার*	২৪ ঘণ্টা
নাইট্রোজেন ডাইঅক্সাইড (NO_2)	৪০ মাইক্রোগ্রাম/ঘনমিটার*	বার্ষিক
	৮০ মাইক্রোগ্রাম/ঘনমিটার*	২৪ ঘণ্টা
বস্তুকণা$_{১০}$(PM_{10})	৫০ মাইক্রোগ্রাম/ঘনমিটার*	বার্ষিক
	১৫০ মাইক্রোগ্রাম/ঘনমিটার*	২৪ ঘণ্টা
বস্তুকণা$_{২.৫}$($PM_{2.5}$)	৩৫ মাইক্রোগ্রাম/ঘনমিটার*	বার্ষিক
	৬৫ মাইক্রোগ্রাম/ঘনমিটার*	২৪ ঘণ্টা
ওজোন (O_3)	১৮০ মাইক্রোগ্রাম/ঘনমিটার*	১ ঘণ্টা
	১০০ মাইক্রোগ্রাম/ঘনমিটার*	৮ ঘণ্টা
সালফার ডাইঅক্সাইড (SO_2)	২৫০ মাইক্রোগ্রাম/ঘনমিটার*	১ ঘণ্টা
	৮০ মাইক্রোগ্রাম/ঘনমিটার*	২৪ ঘণ্টা
অ্যামোনিয়া (NH_3)	১০০ মাইক্রোগ্রাম/ঘনমিটার*	বার্ষিক
	৪০০ মাইক্রোগ্রাম/ঘনমিটার*	২৪ ঘণ্টা

নোট: * এই তফসিলে বায়ু মানমাত্রা বলিতে পরিবেষ্টক বায়ুর মানমাত্রা (Ambient Air Quality Standards)-কে বুঝাইবে।

(ক) গড়মান বৎসরে একবারের অধিক অতিক্রম করিবে না।

(খ) লক্ষ্যমাত্রা অর্জিত হইবে যখন বার্ষিক গড়মান নির্ধারিত মানমাত্রার মধ্যে থাকিবে।

Table -15.2: WHO recommend ambient air quality guidelines, 2005 and 2021

Recommended 2021 AQG levels compared to 2005 air quality guidelines

Pollutant	Averaging time	2005 AQGs	2021 AQG level
$PM_{2.5}$, μg/m³	Annual	10	5
	24-hour[a]	25	15
PM_{10}, μg/m³	Annual	20	15
	24-hour[a]	50	45
O_3, μg/m³	Peak season[b]	–	60
	8-hour[a]	100	100
NO_2, μg/m³	Annual	40	10
	24-hour[a]	–	25
SO_2, μg/m³	24-hour[a]	20	40
CO, mg/m³	24-hour[a]	–	4

μg = microgram [a] 99th percentile (i.e. 3–4 exceedance days per year). [b] Average of daily maximum 8-hour mean O_3 concentration in the six consecutive months with the highest six-month running-average O_3 concentration. Note: Annual and peak season is long-term exposure, while 24 hour and 8 hour is short-term exposure.

Table - 15.3: US-EPA's breakpoint and Air Quality Index (AQI)

Breakpoints							AQI	Category
O_3 (ppm) 8-hour	O_3 (ppm) 8-hour[1]	PM_{10} (μg/m³)	$PM_{2.5}$ (μg/m³)	CO (ppm)	SO_2 (ppm)	NO_2 (ppm)		
0-0.064	–	0-54	0-15.4	0-4.4	0-0.034	([2])	0-50	Good
0.065-0.084	–	55-154	15.5-40.4	4.5-9.4	0.035-0.144	([2])	51-100	Moderate
0.085-0.104	0.125-0.164	155-254	40.5-65.4	9.5-12.4	0.145-0.224	([2])	101-150	Unhealthy for sensitive groups
0.105-0.124	0.165-204	255-354	65.5-150.4	12.5-15.4	0.225-0.304	([2])	151-200	Unhealthy
0.125-0.374 (0.155-0.404)[4]	0.205-0.404	355-424	150.5-250.4	15.5-30.4	0.305-0.604	0.65-1.24	201-300	Very unhealthy
([3])	0.405-504	425-504	250.5-350.4	30.5-40.4	0.605-0.804	1.25-1.64	301-400	Hazardous
([3])	0.505-0.604	505-604	350.5-500.4	40.5-50.4	0.805-1.004	1.65-2.04	401-500	Hazardous

[1]Areas are required to report the AQI based on 8 hour ozone values. However, there are areas where an AQI based on 1-hour ozone values would be more protective. In these cases the index for both the 8-hour and the 1-hour ozone values may be calculated and the maximum AQI reported.
[2]NO_2 has no short term NAAQS and can generate an AQI only above a value of 200.
[3]8-hour O_3 values do not define higher AQI values (≥301). AQI values of 301 or higher are calculated with 1-hour O_3 concentration.
[4]The numbers in parentheses are associated 1 hour values to be used in this overlapping category only.

AQI Values	Level of Health Concern
0 – 50	Good
51 – 100	Moderate
101 – 150	Unhealthy for Sensitive Groups
151 – 200	Unhealthy
201 – 300	Very Unhealthy
301 – 400	Hazardous
401 – 500	Hazardous

Example AQI Calculation:

Suppose a $PM_{2.5}$ sensor records 30.1 $\mu g/m^3$ average pollution over a 24-hour period. This reading falls into the 12.1–35.4 range for $PM_{2.5}$, associated with the index range 51–100. (This is the "Moderate Quality" row of the table.) The AQI for this pollutant is computed as:

$$I_{PM_{2.5}} = \frac{(I_{\text{high}} - I_{\text{low}})}{(C_{\text{high}} - C_{\text{low}})} \times (C_P - C_{\text{low}}) + I_{\text{low}}$$
$$= \frac{(100 - 51)}{35.4 - 12.1} \times (30.1 - 12.1) + 51$$
$$= 88.85$$
$$\approx 89$$

$$I = \frac{I_{high} - I_{low}}{C_{high} - C_{low}} (C - C_{low}) + I_{low}$$

I = the resulting index value,
C= the pollutant concentration,
C_{low}= the concentration breakpoint below C,
C_{high}= the concentration breakpoint above C,
I_{low}= the index breakpoint corresponding to C_{low},
I_{high}= the index breakpoint corresponding to C_{high}.

Apparatus and Accessories:

- High Volume Sampler for PM_{10} (Respirable Dust Sampler, Envirotech APM 460 NL)
- High Volume Sampler for $PM_{2.5}$ (Fine Particulate Sampler, Envirotech APM550)
- Filter Papers:
  - Glass microfiber filter for PM_{10}
  - Teflon (PTFE) filter for $PM_{2.5}$
- Precision Weight Balances:
  - Precision of at least 0.001 grams for PM_{10}
  - Precision of at least 0.00001 grams for $PM_{2.5}$

PM_{10} Measurement Procedure:

1. Place the filter paper in a desiccator for 30 minutes for environmental conditioning, such as removing any moisture.
2. Using a balance with a precision of at least 0.001 grams, record the initial weight of the filter paper.
3. Position the glass microfiber filter paper on the Respirable Dust Sampler.
4. Check the initial position of the flow meter's water column with the pump off; it should be at zero. If it's below zero, adjust it by adding water to reach the zero level.
5. Turn on the sample pump and record the initial flow reading (unit: m^3/min). Note the pump start time. Continue to take flow readings at regular intervals, such as every 30 minutes.
6. Keep the pump running for the designated sampling duration, recommended for at least 3 hours.
7. At the end of the desired sampling duration, turn off the pump, remove the filter samples from the sampler, and place them back in the desiccator for 30 minutes of environmental conditioning.
8. After environmental conditioning, weigh the filter samples using the same precision balance used for the initial weight measurement.
9. Calculate the PM_{10} concentration by finding the difference between the final and initial weights, which represents the weight of particles collected on the filter paper. Divide this weight by the total volume of air sampled during the entire sampling duration. Report the concentration in micrograms per cubic meter ($\mu g/m^3$).

$PM_{2.5}$ Measurement Procedure:

1. Place the filter paper in a desiccator for 30 minutes to condition it and remove any moisture.
2. Using a balance with a precision of at least 0.00001 grams, record the initial weight of the filter paper.
3. Position the Teflon (PTFE) filter paper onto the Fine Particulate Sampler.
4. Turn on the power of the fine particulate sampler, which will turn on the pump, and note the initial air volume (in m^3) and time (in hours) as displayed on the sampler's designated panels.
5. Keep the pump running for the designated sampling duration, recommended for at least 3 hours for this exercise.
6. Upon completing the desired sampling duration, record the final air volume (in m^3) and time (in hours). The difference between the final and initial air volume readings represents the sampled air volume. You can determine the sampling duration by calculating the difference between the final and initial time readings.
7. Turn off the sampler's power, which will also turn off the pump, remove the filter samples, and place them back in the desiccator for another 30 minutes of environmental conditioning.
8. After environmental conditioning, use the same precision balance employed for the initial weight measurement to weigh the filter samples.
9. Calculate the $PM_{2.5}$ concentration by subtracting the final weight from the initial weight, which corresponds to the weight of collected particles on the filter paper. Divide this weight by the total volume of air sampled throughout the entire sampling duration. Report the concentration in micrograms per cubic meter ($\mu g/m^3$).

Experiment No. 16

Measurement of Ambient Noise Level

Introduction

This laboratory exercise is designed to equip you with hands-on experience in measuring ambient noise levels. By the end of this lab, you will have gained proficiency in operating a sound level meter, conducting measurements, and interpreting the significance of noise data in the realm of environmental engineering. Noise is defined as unwanted sound pollutant, which produces undesirable physiological and psychological effects in an individual, by interfering with one's social activities like work, rest, recreation, sleep, etc. It can be also defined as any undesirable human or machine created sound which disturbs the activity or balance of human or animal life.

Environmental Significance

Noise pollution is a pervasive environmental concern that affects the well-being of both human populations and ecosystems. Noise pollution presents a pervasive threat, causing annoyance, health ailments, and ecological imbalance. Physiological repercussions include anxiety, insomnia, and hearing impairment, with wildlife experiencing behavioral changes and habitat disruptions. Prolonged exposure can lead to more severe conditions like high blood pressure and long-term hearing loss. Ultimately, noise pollution emerges as a complex issue with wide-ranging impacts on both human health and the environment.

The ability to measure ambient noise levels is indispensable for comprehending the impact of noise pollution on communities and the natural world. Noise data plays a pivotal role in identifying noise pollution sources, assessing noise exposure, and formulating strategies for noise control and mitigation. Environmental engineers rely on noise measurements to ensure compliance with noise regulations and to design effective noise reduction measures, particularly in urban planning and infrastructure projects.

Noise Measurement

Sound, often depicted as pressure waves originating from vibrating objects, is characterized by its intensity or loudness, quantified in units known as decibels (dB). The dB scale is logarithmic, meaning that each 10 dB increment corresponds to a tenfold increase in sound intensity. Key reference levels include the threshold of hearing (0 dB) and the threshold of pain (approximately 120-130 dB).

A sound level meter, commonly referred to as a decibel meter, is the instrument of choice for measuring sound intensity. It consists of a microphone to capture sound, an amplifier for signal processing, and a display to present noise level readings. Microphone detects and reads minimal air pressure changes and converts them into electric signals. These signals are processed by electric circuits and converted into decibels. Sound level monitor must be calibrated before and after use. Every sound level meter has two modes and those are SLOW and FAST. It is the response rate which SLM averages before showing on screen. For workplace noise level monitoring it should be taken as SLOW. To maintain measurement accuracy, sound level meters must undergo calibration. Calibration entails adjusting the meter to a known reference level. Regular calibration is imperative to secure trustworthy noise measurements. In the lab the sound level meter is calibrated using a sound level meter calibrating device. The calibrating device can create sound of required known frequencies.

The following two concepts are used to measure the sound level.

- **L_N Concept:** Statistical measure of how frequently a particular sound level is exceeded.
- **L_{eq} Concept:** Statistical measure of sound pressure level that can be equated to any fluctuating noise level.

For this experiment, L_{eq} concept will be used.

Equivalent Continuous Sound Level (L_{eq})

The L_{eq} (Equivalent Continuous Sound Level) is a key metric in acoustics and noise studies, representing the average sound level over a designated period. The term 'equivalent' indicates that the combined energy from fluctuating sound levels during a timeframe is equivalent to the energy from a constant, non-varying sound level for that same duration. 'Continuous' implies that L_{eq} represents this sound as though it maintained a consistent level throughout the observed period. A graphical representation of the concept of Equivalent Continuous Noise Level (L_{eq}) is shown in Figure 16.1.

L_{eq} is the statistical value of sound pressure level that can be equated to any fluctuating noise. Thus, it is defined as the constant noise level over a given time that expends the same amount of energy as expended by the fluctuating levels over the same time. The value can be expressed as equation 16.1:

$$L_{eq} = 10log \sum_{i=1}^{i=n} (10)^{\frac{l_i}{10}} \times t_i \qquad (16.1)$$

Where, n = total number of sound samples

l_i = noise level of any i^{th} sample

t_i = Time duration of the i^{th} sample expressed as a fraction of total sample time

It is common practice to measure noise levels using the A-weighting setting built into all sound level meters. In which case the term is properly known as LA_{eq} and the results should say so - for example LA_{eq} = 73 dB or Leq = 73 dBA.

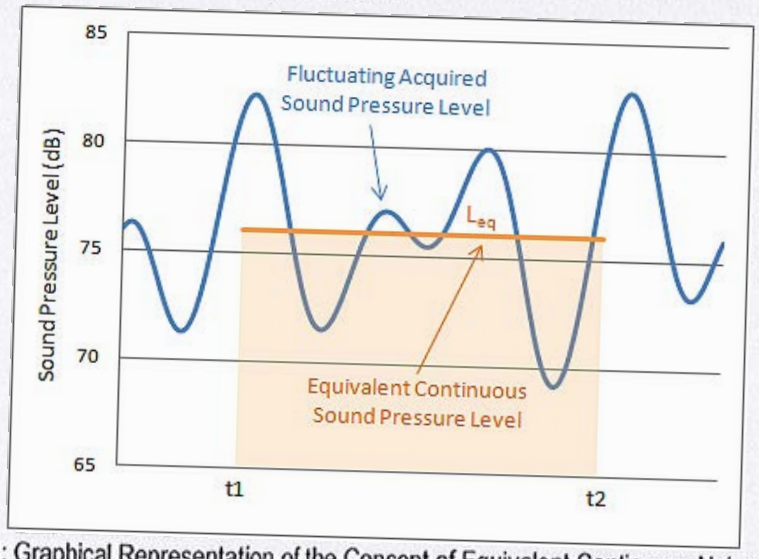


Figure 16.1: Graphical Representation of the Concept of Equivalent Continuous Noise Level (L_{eq})

L_{eq} is the constant noise level that would result in the same total sound energy being produced over a given period. It can be measured in A, B and C decibel weighing scales.

- A-weighting gives more value to frequencies in the middle of human hearing and less value to frequencies at the edges as compared to a flat audio decibel measurement. The A-weighting represents the way the human ear is more sensitive to mid-range frequencies and less sensitive to high and low frequencies. A-weighting is the standard for determining hearing damage and noise pollution.
- Unlike the A-weighting, which attenuates lower and higher frequencies to mimic the sensitivity of the human ear to mid-range frequencies, the B-weighting is less commonly used and provides less attenuation at low frequencies and more at higher frequencies. The B-weighting is sometimes used in sound level meters for specific applications where there may be a need to capture a wider range of frequencies, such as in certain industrial or environmental noise assessments. However, it is less commonly used than the A-weighting scale in most noise measurement standards and regulations.

- C-weighting can be used for Peak measurements and low frequency noise. It is often used in entertainment noise measurement, where high pressure low frequency noise is common. The C-weighting is also commonly used for sounds with impulsive characteristics such as fire-arms; shooting ranges; pile driving.

Noise Standards

The Bangladesh Noise Pollution Control Rules of 2006 establish maximum permissible L_{eq} values for various categories of areas, including silent zones, residential zones, mixed zones, commercial areas, and industrial zones, as detailed in Table 16.1. The Occupational Safety and Health Administration (OSHA) has set Occupational Noise Exposure levels, as outlined in Table 16.2.

Table 16.1: Bangladesh Standard for Ambient Noise Level (as per Noise Pollution Control Rules, 2006)

Sl. No.	Type of Area	Equivalent Noise Level, Leq dB(A)	
		Day	Night
1	Silent Area	50	40
2	Residential Area	55	45
3	Mixed Area	60	50
4	Commercial Area	70	60
5	Industrial Area	75	70

Note: a) Daytime: 6 AM to 9 PM; b) Nighttime: 9 PM to 6 AM

Table 16.2: Limits for permissible Noise Exposure according to OSHA

Exposure time	Noise Level
8 hours	90 dB
6 hours	92 dB
4 hours	95 dB
3 hours	100 dB
2 hours	102 dB
1 hours	105 dB
30 minutes	110 dB
15 minutes	115 dB

Apparatus

- Sound Level Meter (decibel meter)



Figure – 16.2: Sound Level Meter

Procedure

The experiment will be conducted in two steps. Firstly, collection of ambient noise data and followed by analysis of collected data.

Data Collection

1. Proceed to your designated location of interest, where you will be assigned to collect noise data (for example, inside a classroom, laboratory, near a roadside, etc.). Turn on the calibrated sound level meter and record the noise level displayed on the monitor.
2. While measuring noise levels it is held in an arm's length at the ear height. It does not matter whether microphone is point towards source.
3. Record readings at specific time intervals (e.g., every 1 minute) and collect at least 10 minutes of data at each location. Utilize the provided data sheet for recording measurements. Note the time, date, and any potential sources of noise.

Data Analysis

1. Calculate the Equivalent Noise Level (L_{eq}), Mean, Median, Standard Deviation, Maximum, and Minimum values of the collected noise data.
2. Share your observations with your fellow students/groups and prepare a summary of noise data (L_{eq}, Mean, Median, Standard Deviation, Maximum, and Minimum) by combining measurements from different groups.

APPENDIX A
WATER QUALITY STANDARDS – ECR'2023
(a) Standards and Guideline Values for Drinking Water

Sl. No.	Parameter	Unit	Bangladesh Standards (ECR 2023)	World Health Organization Guidelines(WHO, 2011)
1	Aluminum	mg/L	0.2	0.2**
2	Ammonia (NH3)	mg/L	0.5	1.5**
3	Arsenic	mg/L	0.05	0.01
4	Barium	mg/L	0.7	0.7
5	Benzene	mg/L	0.01	0.01
6	BOD5 (20°C)	mg/L	0.2	--
7	Boron	mg/L	1.0	2.4
8	Cadmium	mg/L	0.003	0.003
9	Calcium	mg/L	75	--
10	Chloride	mg/L	250*	250**
11	Chlorinated alkanes			
	Carbon tetrachloride	mg/L	0.005	0.004
	1,1- Dichloroethylene	mg/L	0.03	--
	1,2- Dichloroethylene	mg/L	0.03	0.05
	Tetrachloroethylene	mg/L	0.04	0.04
	trichloroethylene	mg/L	0.02	0.02
12	Chlorinated phenols			
	Pentrachlorophenol	mg/L	0.009	0.009
	2,4,6- Trichlorophenol	mg/L	0.2	0.2
13	Chlorine (residual)	mg/L	0.2	5
14	Chloroform	mg/L	0.09	0.3
15	Chromium (hexavalent)	mg/L	0.05	--
16	Chromium (total)	mg/L	0.05	0.05
17	COD	mg/L	4	--
18	Fecal Coliform	Nos/100 mL	0	0
19	Total Coliform	Nos/100 mL	0	0
20	Color	Pt-Co unit	15	15**
21	Copper	mg/L	1.5	2
22	Cyanide	mg/L	0.05	0.07**
23	Detergents	mg/L	0.2	--
24	Dissolved Oxygen	mg/L	6	--
25	Fluoride	mg/L	1	1.5
26	Hardness (CaCO3)	mg/L	500	500**

Sl. No.	Parameter	Unit	Bangladesh Standards (ECR 2023)	World Health Organization Guidelines (WHO, 2011)
27	Iron	mg/L	0.3-1.0	0.3**
28	Kjeldahl Nitrogen (total)	mg/L	1	--
29	Lead	mg/L	0.01	0.01
30	Magnesium	mg/L	30-35	--
31	Manganese	mg/L	0.4	0.1 (health based)**
32	Mercury	mg/L	0.001	0.006
33	Nickel	mg/L	0.05	0.07
34	Nitrate	mg/L	45	50 (as NO3-)
35	Nitrite	mg/L	1	3 (as NO2-)
36	Odor	mg/L	Odorless	--
37	Oil and grease	mg/L	0.01	--
38	pH	--	6.5-8.5	6.5-8.5**
39	Phenolic compounds	mg/L	0.002	--
40	Phosphate	mg/L	6	--
41	Phosphorous	mg/L	0	--
42	Potassium	mg/L	12	--
43	Radioactive materials (gross alpha activity)	Bq/L	0.1	--
44	Radioactive materials (gross beta activity)	Bq/L	1	--
45	Selenium	mg/L	0.01	0.04
46	Silver	mg/L	0.02	--
47	Sodium	mg/L	200	--
48	Sulfide	mg/L	0.05	--
49	Sulfate	mg/L	250	--
50	Total Dissolved Solids (TDS)	mg/L	1000	1000**
51	Total Suspended Solids (TSS)	mg/L	10	--
52	Temperature	℃	20-30	--
53	Tin	mg/L	2	--
54	Turbidity	NTU	5	5**
55	Zinc	mg/L	5	--

** In coastal areas, allowable chloride concentration is 1000 mg/L.*

*** Values are taken from WHO guideline value for drinking water 2004.*

Standards for Wastewater from Industrial Units or Projects Waste

Sl. No.	Water Quality Parameters	Unit	Bangladesh Standard for Industrial Effluent (ECR'23)		
			Discharge into Inland Water	In Sewer for Secondary Treatment	Discharge near Coastal Area
1	Ammonia Nitrogen(NH3 - N)	mg/L	50	50	50
2	Ammonia (free ammonia)	mg/L	5.0	5.0	5
3	Arsenic (As)	mg/L	0.2	0.2	0.2
4	Biochemical Oxygen Demand (BOD5) at 20°C	mg/L	30	250	100
5	Boron	mg/L	2	2	4
6	Cadmium, Cd	mg/L	2	1	2
7	Chloride, Cl	mg/L	600	600	-
8	Chromium, Cr	mg/L	0.5	1	1
9	Copper, Cu	mg/L	3	3.0	3.0
10	Chemical Oxygen Demand, COD ($K_2Cr_2O_7$ Value)	mg/L	200	400	250
11	Chromium(as hexavalent Cr)	mg/L	0.1	2.0	1.0
12	Dissolved Oxygen(DO)	mg/L	4.5–8	4.5 – 8	4.5 – 8
13	Electro-conductivity(EC)	mg/L	1200	1200	1200
14	Total Dissolved Solids (TDS)	mg/L	2,100	2,100	2,100
15	Fluoride(F)	mg/L	2	15	15
16	Sulfide(S2-)	mg/L	1	-	5
17	Iron(Fe)	mg/L	3	3	3
18	Total Kjeldahl Nitrogen(TKN)	mg/L	100	-	100
19	Lead(Pb)	mg/L	0.1	1.0	2.0
20	Manganese(Mn)	mg/L	2	2	2
21	Mercury(Hg)	mg/L	0.01	0.01	0.01
22	Nickel(Ni)	mg/L	1.0	2.0	5.0
23	Nitrate(elementary N)	mg/L	10.0	-	20.0
24	Oil and Grease	mg/L	10	20	20
25	Phenolic Compounds (C_6H_5OH)	mg/L	1	5	5
26	Dissolved Phosphorus (P)	mg/L	5	-	-
27	pH	mg/L	6 – 9	6 – 9	6 – 9
28	Selenium(Se)	mg/L	0.05	0.05	0.05
29	Zinc(Zn)	mg/L	5	15	15
31	Temperature (summer) (Winter)	°C	Not 5°C more than temp. of water body	-	Not 5°C more than temp. of water body
32	Total Suspended Solids(TSS)	mg/L	100	500	100
33	Cyanide(Cn)	mg/L	0.1	2.0	0.2

Standards for Sewage Discharge

Sl. No.	Parameter	Unit	Standard Limit
1	BOD	mg/L	40
2	Nitrate	mg/L	250
3	Phosphate	mg/L	35
4	Suspended Solids (SS)	mg/L	100
5	Temperature	^{0}C	30
6	Coliform number	no/100 mL	100

Notes:
(1) This limit shall be applicable to discharges into surface and inland water bodies.
(2) Sewage shall be chlorinated before final discharge.

APPENDIX B

Water Sample Preservation

Parameter	Matrix	Container	Minimum Sample Size	Preservative	Holding Time
Nitrogen, Nitrite	Water & Wastewater	Plastic or Glass	250 mL	Cool 6 C	48 Hours
	Solid	Plastic	50 mL	Cool 6 C	48 Hours
Nitrogen, Total	Water and Wastewater	Plastic or Glass	250 mL	H2SO4 to pH < 2, Cool 6C	
Nitrogen, Ammonia	Water & Wastewater	Plastic or Glass	500 mL	H2SO4 to pH < 2, Cool 6C	28 Days
	Solid	Plastic	50 mL	Cool 6 C	28 Days
Oil & Grease	Water & Wastewater	Amber Glass	1000 mL	H2SO4 to pH < 2 Cool 6 C	28 Days
Organic Carbon, Total	Water & Wastewater	Plastic or Glass	500 mL	H2SO4 to pH < 2, Cool 6C	28 Days
	Solid	Plastic	50 mL	Cool 6 C	28 Days
Oxygen, Dissolved	Water & Wastewater	Glass bottle w/ top, no headspace	500 mL	None	Analyze Immediately
pH	Water & Wastewater	Plastic or Glass	100 mL	None	Analyze in 15min
	Solid	Plastic	50 mL	None	Analyze in 15min
Phaeophytin	Water	Amber Glass	1000 mL	Cool 6 C	48 Hours
Phenolics, Total	Water & Wastewater	Amber, Plastic, or Glass	1000 mL	H2SO4 to pH <2, Cool 6C	28 Days
Phosphorus, Total	Water and Wastewater	Plastic or Glass	500 mL	H2SO4 to pH <2, Cool 6C	28 Days
	Solid	Plastic	50 mL	Cool 6 C	28 Days
Phosphorus, Ortho	Water & Wastewater	Plastic or Glass	500 mL	Filter, Cool 6 C	48 Hours, Filter within 15 minutes
	Solid	Plastic	50 mL	None	48 Hours
Salinity	Water & Wastewater	Glass	500 mL	None	Analyze Immediately
Settleable Matter	Water & Wastewater	Plastic or Glass	1000 mL	Cool 6 C	48 Hours
Silica	Water & Wastewater	Plastic	500 mL	Cool 6 C	28 Days
Solids, Total	Water & Wastewater	Plastic or Glass	500 mL	Cool 6 C	7 Days
	Solid	Plastic	50 mL	None	7 Days
Solids, Total Volatile	Water & Wastewater	Plastic or Glass	500 mL	Cool 6 C	7 Days
	Solid	Plastic	50 mL	None	7 Days
Solids, Total Suspended	Water & Wastewater	Plastic or Glass	500 mL	Cool 6 C	7 Days
Solids, Total Settleable	Water & Wastewater	Plastic or Glass	500 mL	Cool 6 C	7 Days
Solids, Total Dissolved	Water & Wastewater	Plastic or Glass	500 mL	Cool 6 C	7 Days
Solids, Suspended Volatile	Water & Wastewater	Plastic or Glass	500 mL	Cool 6 C	7 Days
Sulfate	Water & Wastewater	Plastic or Glass	500 mL	Cool 6 C	28 Days
	Solid	Plastic	50 mL	None	28 Days
Sulfide	Water & Wastewater	Amber Glass	500 mL	Cool 6 C 2 mL Zinc Acetate	7 Days
	Solid	Plastic	50 mL	None	7 Days
Sulfite	Water & Wastewater	Plastic or Glass	500 mL	None	Analyze Immediately
Surfactants	Water & Wastewater	Plastic or Glass	500 mL	Cool 6 C	48 Hours
Temperature	Water & Wastewater	Plastic or Glass	100 mL	None	Analyze Immediately
Turbidity	Water & Wastewater	Plastic or Glass	250 mL	Cool 6 C	48 Hours
Metals					
Metals, General	Water & Wastewater	Plastic or Glass, Rinsed w/ 1:1 HNO3	500 mL	HNO3 to pH < 2	6 Months
	Solid	Plastic	50 mL	Cool 6 C	6 Months
Mercury	Water & Wastewater	Plastic or Glass, Rinsed w/ 1:1 HNO3	500 mL	1:1 HNO3 to pH < 2	28 Days
	Solid	Plastic	50 mL	Cool 6 C	28 Days
Lead	Water & Wastewater	Plastic or Glass	1000 mL	HNO3 to pH <2	6 Months
	Drinking Water	Plastic or Glass	1000 mL	HNO3 to pH <2	6 Months

Parameter	Matrix	Container	Minimum Sample Size	Preservative	Holding Time
Organic Compounds					
GRO (Gasoline Range Organics), PVOC	Water	(4) 40 mL amber, glass bottle w/ 0.5mL 1:1 HCL, no headspace	40 mL	4 drops conc. HCL, Cool 6 C	14 Days
	Soil	(2) 60mL amber, glass bottle w/ 25mL MeOH	20- 35 grams	25 mL Methanol, Cool 6 C	21 Days
DRO (Diesel Range Organics)	Water	(2) 1 L Amber, glass bottle w/5.0 mL 1:1 HCL	1 L	5 mL 1:1 HCL, Cool 6 C	7 Days
	Soil	(2) 60 mL amber, glass bottle w/tare weight, no preservative	20- 35 grams	Cool 6 C	10 Days
BTEX	Water	(3) 40 mL glass vial w/ septa cap, zero headspace	40 mL	4 drops conc. HCL, Cool 6 C	14 Days
	Soil	(2) 4 oz Amber Glass w/ septa cap, zero headspace	25 grams	Cool 6 C	14 Days
VOCs:	Water & Wastewater	(4) 40 mL glass vial w/ septa cap, zero headspace	40 mL	0.5 mL 1:1 HCL	14 Days
	Soil	(2) 60 mL amber	20-35 g	25 mL MeOH	14 days
Pesticides, General	Water	(2) Amber Glass w/ TFE septa cap	1 Liter	Cool 6 C	7 Days
	Soil	(1) 4 oz Amber Glass	50 mL	Cool 6 C	14 Days
TCLP, Toxicity Characteristic Leaching	Water	1 L Amber Glass	1 Liter	Cool 6 C	14 Days
	Soil	(2) 4 oz Amber Glass	200 grams	Cool 6 C	14days

Solids & Semi-Solid - Microbiology	Matrix	Container	Minimum Sample Size	Preservative	Holding Time
Coliform, Fecal	Semi-Solid Solid	Sterile Container	100 mL	Cool 6 C	8 Hrs EPA compliance 24 Hours

Solids & Semi-Solid - Inorganics and Nutrients					
% Solids	Semi-Solid Solid	Plastic or Glass	100 grams	Cool 6 C	7 days

APPENDIX C
LAB REPORT FORMAT

CE 332 : ENVIRONMENTAL ENGINEERING SESSIONAL

Lab Experiment Report

Experiment No: - 1,2,3 &4

Name of the Experiment: Determination of pH, Color, Turbidity & Solids in Water

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Objective of the Tests:

Water Sampling Information (source, location, date and time of collection etc.):

Apparatus & Reagents used:

Lab Data Sheet

Experiment No. 1,2,3 & 4: Determination of pH, Color, Turbidity & Solids in Water

Sl No.	Water Quality Parameter	Unit	Concentration Present	Bangladesh Standard for Drinking Water (ECR'23)	WHO Guideline Values 2011 (or 2004)
1	pH				
2	Apparent Color				
3	True Color				
4	Turbidity				
5	Total Dissolved Solids (TDS)				
6	Total Suspended Solids (TSS)				
7	Total Solids (TS)				
8	Electric Conductivity (EC)				
9	Empirical Factor = TDS / EC				

Sample Calculation:

Q-3: How true color can be removed from water?.

Q-4: How the presence of turbidity causing elements interfere with water treatment processes?

Q-5: How TDS can be relate with conductivity property of water?

Q-6: A water sample is tested for determination of TDS in water and following are the obtained results:

Wt of beaker: 51.5694 gm
Wt of beaker + residue of filtered water: 51.9325 gm
test sample size = 300 mL
Empirical factor between EC and TDS = 0.75

Also comment on the obtained result.

Q-3: How true color can be removed from water?.

Q-4: How the presence of turbidity causing elements interfere with water treatment processes?

Q-5: How TDS can be relate with conductivity property of water?

Q-6: A water sample is tested for determination of TDS in water and following are the obtained results:

Wt of beaker: 51.5694 gm
Wt of beaker + residue of filtered water: 51.9325 gm
test sample size = 300 mL
Empirical factor between EC and TDS = 0.75

Also comment on the obtained result.

CE 332 : ENVIRONMENTAL ENGINEERING SESSIONAL
Lab Experiment Report

Experiment No: - 5,6 & 7

Determination of Carbon Dioxide, Alkalinity & Hardness in Water

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Objective of the Tests:

Water Sampling Information (source, location, date and time of collection etc.):

Apparatus & Reagents used:

Lab Data Sheet

Experiment No. 5,6 & 7: Determination of Carbon Dioxide, Alkalinity & Hardness in Water

Sl No.	Water Quality Parameter	Unit	Concentration Present	Bangladesh Standard for Drinking Water (ECR'23)	WHO Guideline Values 2011 / 2004
1	Carbon Dioxide (CO_2)				
2	CO_2 -Acidity				
3	P-Alkalinity				
4	Total Alkalinity				
5	Total Hardness				
6	CO_3 - Hardness				
7	Non-CO_3 Hardness				

Sample Calculation:

Discussion on obtained results:

Assignment Question & Answer:

Q-1: Why the test for carbon dioxide should be performed immediately after collection of water sample? Explain.

Q-2: If CO_2 –acidity of water sample are 16.8 mg/L as $CaCO_3$, calculate – amount of titrant in measuring CO_2 –acidity (when 200 mL sample titrated with titrant of N/40 normality)

Q-3: Calculate hydroxide, bicarbonate, and carbonate alkalinities of a water sample with a total alkalinity of 330 mg/L as $CaCO_3$ and a pH of 7.4. Which type of alkalinity dominates the total alkalinity? Also calculate the concentrations of carbonate (CO_3^{2-}) and bicarbonate (HCO_3) ions in water. [Hint: Use Eqs. 10 – 15].

Q-4: A water sample has the following analysis:

Na^+	: 40 mg/L	Cl^-	: 40 mg/L
K^+	: 31 mg/L	HCO_3^-	: 167 mg/L
Ca^{2+}	: 84 mg/L	CO_3^{2-}	: 0.85 mg/L
Mg^{2+}	: 39 mg/L	SO_4^{2-}	: 5 mg/L
Sr^{2+}	: 2 mg/L	NO_3^-	: 10 mg/L

Calculate total hardness, carbonate hardness, and non-carbonate hardness. Assume alkalinity of the water sample is 250 mg/L as $CaCO_3$.

Discussion on obtained results:

Assignment Question & Answer:

Q-1: Write down some merit demerits of ETAAS method and FAAS method.

Q-2: In determination of arsenic (following the methods described here), why the water samples are acidified and often pre-concentrated before actual determination of arsenic?

Question & Answer:

Q-1: In determination of chloride, why an indicator "blank" or "error" is subtracted from the amount of silver nitrate used in titration? Explain.

Q-2: Write down some important environmental significance of chloride in water.

Q-3: Why hydrochloric acid and potassium permanganate are added to the sample in the determination of iron?

Q-4: If 0.75 mL standard iron solution [strength of the standard iron solution = 100 mg/L] is needed to match with the color of a 50 mL sample, what is the iron concentration of the test sample in mg/L?

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Lab Experiment Report

Experiment No: - 8 & 9

Determination of Chloride & Total Iron Concentration in Water

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Objective of the Tests:

Water Sampling Information (source, location, date and time of collection etc.):

Apparatus & Reagents used:

Lab Data Sheet

Experiment No. 8 & 9: Determination of Chloride & Total Iron Concentration in Water

Sl No.	Water Quality Parameter	Unit	Concentration Present	Bangladesh Standard for Drinking Water (ECR'23)	WHO Guideline Values 2011 (or 2004)

Sample Calculation:

Discussion on obtained results:

Sample Calculation:

Discussion on obtained results:

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Lab Experiment Report

Experiment No: - 10

Determination of Arsenic in Water

Objectives of the Tests:

Source of sample:

Apparatus & Reagents used:

Lab Data Sheet

Experiment No. 10: Determination of Arsenic Concentration in Water

Sl No.	Water Quality Parameter	Unit	Concentration Present	Bangladesh Standard for Drinking Water (ECR'23)	WHO Guideline Values 2011 (or 2004)

Sample Calculation:

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Lab Experiment Report

Experiment No: - 12

Name of the Experiment: Estimation of Organic Pollution Load through determination of Biological Oxygen Demand (BOD) & Chemical Oxygen Demand (COD)

Objectives of the Tests:

Water Sampling Information (source, location, date and time of collection etc.):

Apparatus & Reagents used:

CE 332: ENVIRONMENTAL ENGINEERING SESSIONAL
Lab Experiment Report

Experiment No: - 11

Microbiological Quality of Water: Determination of Total Coliform and Fecal Coliform

Objective of the Tests:

Water Sampling Information (source, location, date and time of collection etc.):

Apparatus & Reagents used:

Lab Data Sheet

Experiment No. 11: Microbiological Quality of Water: Determination of Total Coliform and Fecal Coliform

Sl No.	Water Quality Parameter	Unit	Concentration Present	Bangladesh Standard for Drinking Water (ECR'23)	WHO Guideline Values 2011 (or 2004)

Discussion on obtained results:

Assignment Question & Answer:

Q-1: What is the significance of conducting total coliform test particularly for treated/ chlorinated water supplies?

Q-2: What are the major advantages of "membrane filtration method" over "multiple tube method"?

Q-3: A water sample is tested for bacteriological analysis and the number of blue colonies and red colonies (with golden shin) are found to be 2 and 7 respectively. Test samples are prepared by mixing 50 ml original sample with1liter dilution water. If 200 mL sample is filtrated for each test, calculate value of total coliform and fecal coliform of original sample.

Q-4: In determination of COD of wastewater, compare the suitability of using $KMnO_4$ and $K_2Cr_2O_7$ as oxidizing agent.

Q-5: Write down some advantages of COD test.

Lab Data Sheet

Experiment No. 12: Name of the Experiment: Estimation of Organic Pollution Load through determination of Biological Oxygen Demand (BOD) & Chemical Oxygen Demand (COD)

Sl No.	Water Quality Parameter		Unit	Concentration Present	ECR 2023 Standards		
					Discharge in Inland Water	Discharge into Public Sewer	Discharge on Irrigated Land
1	BOD (non-aerated sample)						
2	BOD (aerated sample)						
3	BOD loading rate (discharged into river)						
4	COD (non-aerated sample)	$KMnO_4$					
		$K_2Cr_2O_7$					
5	COD (aerated sample)	$KMnO_4$					
		$K_2Cr_2O_7$					

Sample Calculation:

Lab Data Sheet

Experiment No. 13: Chemical Coagulation of Water - Alum Coagulation

Initial pH of sample:

Initial Turbidity (NTU):

Alum stock Solution concentration (mg/L):

Sample Size (mL):

Beaker No.	Alum dose added (mg/L)	Amount of Alum Stock Solution added (mL)	pH of treated sample	Turbidity of treated sample (NTU)	Optimum Alum Dose (mg/L)

Sample Calculation:

Discussion on obtained results and graphs:

Assignment Question & Answer:

Q-1: A test bottle containing just seeded dilution water has its DO level drop by 0.6 mg/L in a 5-day test. A 300 mL BOD bottle filled with 30 mL of wastewater and the rest with seeded dilution water experiences a drop of 6.5 mg/L in the same period (5-day). Calculate the BOD5 of the wastewater

Q-2: A textile dyeing industry uses vegetable dyes in their textile dyeing process. The wastewater is partially treated before being discharged in to nearby water body. A lab analysis of the effluent shows that the BOD_5 = 115 mg/L. If the wastewater discharge rate is 120 m^3/hr and the operating hour of the industry is 8 hrs per day, calculate the BOD loading rate of the wastewater. Assume BOD_5 = 0.7 UBOD.

Q-3: Why seeding is necessary for BOD test?

Assignment Question & Answer:

Q-1: briefly discuss the following mechanisms of coagulation process- i. Sweep floc ii) Double layer compression.

Q-2: What is charge reversal? When and why it happens?

Q-3: In a laboratory alum coagulation test is carried out in a series of beakers each containing 300 mL of water sample. Alum is added to the beakers using a sock solution having 10 gm alum per liter. Optimum alum dose is obtained for the beaker to which 6 mL stock solution was added. Determine:

a) Optimum alum dose in mg/L &

b) If this alum dose is applied in a water treatment plant with a capacity of treating 900 m3 of water per day, how much alum (kg) would be required at the treatment plan per day?

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Lab Experiment Report

Experiment No: - 14

Name of the Experiment: Determination of Residual Chlorine and Chlorine Demand: Break Point Chlorination

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Objective of the Tests:

Water Sampling Information (source, location, date and time of collection etc.):

Apparatus & Reagents used:

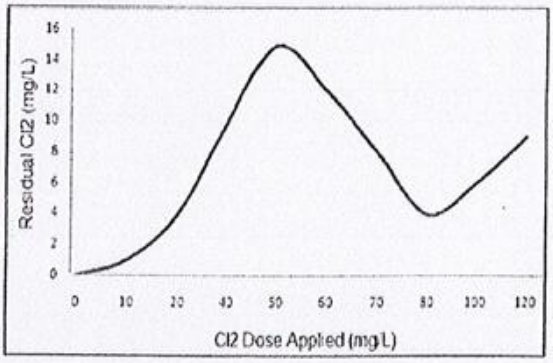
Discussion of obtained result(s) and graph:

Assignment Question & Answer:

Q-1: What are the major disadvantages of chlorination? Name some of the alternate disinfectants.

Q-2: You would like to perform chlorination to a water sample with pH 7.5. At this pH, what would be the relative proportions of HOCl and OCl^- (see Fig.- 1). What kind of change in pH would you propose in order to increase the relative proportion of HOCl, which is a better disinfectant?

Q-3: In a lab test of break point chlorination the following curve is obtained using 10 water samples (sample size = 250 mL). Determine:



i. Chlorine demand for a Cl_2 dose of 95 mg/L.
ii. Free residual chlorine available at a Cl_2 dose of 110 mg/L.
iii. How much chlorine is exhausted during reaction with reducing agents present in water?
iv. If chlorine concentration in the stock solution is 2 g/L, what is the amount of stock solution (in mL) is need to be added to the sample to maintain a chlorine dose of 112 mg/L?

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Lab Experiment Report

Experiment No: - 15

Name of the Experiment: Measurement of Ambient Particulate Air Pollution Concentration: $PM_{2.5}$ and PM_{10}

Objective of the Tests:

Data Collection Details:

Measurement location:

Measurement height from ground:

Name and model of sampler:

PM_{10} sampler: $PM_{2.5}$ sampler:

Measurement date: Measurement start time:

Ambient Temperature: Relative Humidity (RH):

Apparatus used:

Lab Data Sheet

Experiment No. 15: Measurement of Ambient Particulate Air Pollution Concentration: $PM_{2.5}$ and PM_{10}

Filter Paper Weight:

Parameter	Filter paper type	Size of filter paper	Initial Weight (g)	Final Weight (g)	Weight Difference (final – Initial) (g)
PM_{10}					
$PM_{2.5}$					

Sampled Air Volume:

PM_{10}

Sl.	Time	Flow reading (m^3/min)
1		
2		
3		
4		
5		
6		
	Avg. flow	

Total Time = min
Avg. flow rate = m^3/min
Total air volume sampled = m^3

$PM_{2.5}$

	Initial Reading	Final Reading
Time		
Air Volume		

Total Time = h
Total Air Volume = m^3

Concentration Calculation:

	PM mass collected on filter (µg)	Total air volume sampled (m^3)	Concentration (µg /m^3)
PM_{10}			
$PM_{2.5}$			

AQI Calculation

	Measured Concentration, C_P (µg /m^3)	From breakpoint table				AQI	Comment on Air Quality
		C_{high}	C_{low}	I_{high}	I_{low}		
PM_{10}							
$PM_{2.5}$							

Sample Calculation:

Discussion on obtained results:

Assignment Question & Answer:

Q-1: Given the following measured air quality data for a specific location:

$PM_{2.5}$ (24 h)	PM_{10} (24 h)	Ozone (8 h)	NO_2 (1 h)	CO (8 h)	SO_2 (1 h)
45 µg/m³	70 µg/m³	35 ppb	20 ppb	5 ppm	10 ppb

Calculate the Air Quality Index (AQI) for this location based on the respective AQI breakpoints and formula for each pollutant. Provide the final AQI value and its corresponding air quality category.

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Lab Experiment Report

Experiment No: - 16
Measurement of Ambient Noise Level

Objective of the Tests:

Data Collection Details:

Measurement Location:

Measurement height from ground:

Name and model of sound level meter:

Measurement date:

Measurement start time:

Observations on potential sources of noise during the data collection (if any):

Apparatus used:

Lab Data Sheet

Experiment No. 16: Measurement of Ambient Noise Level

Noise Readings

Sl.	Time	Noise Level (dB)
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		

Sl.	Time	Noise Level (dB)
11		
12		
12		
14		
15		
16		
17		
18		
19		
20		

Calculation of summary statistics

L_{eq}		Mean	
		Median	
		SD	
		Max	
		Min	

A summary of noise level measured in different microenvironments.

Gr. No	Location	Time	L_{eq}	Mean	Median	Standard Deviation	Max	Min
			dBA	dBA	dBA	dBA	dBA	dBA

Sample Calculation:

Discussion on obtained results:

Assignment Question & Answer:

Q-1: What is L_{eq}, and what is the significance of L_{eq}?

Q-2: A sound of 80 dB lasting for 10 minutes, followed by a sound of 60 dB lasting for the next 80 minutes, and then followed by a sound of 100 dB for the next 5 minutes. Calculate L_{eq} for the noise levels over 95 minutes.