CE 332
Environmental Engineering- Lab I
(Lab Manual)

Department of Civil Engineering
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Preface

Environmental engineering is the application of science and engineering principles to protect and utilize natural resources, control environmental pollution, improve environmental quality to enable healthy ecosystems and comfortable habitation of humans. It is based on multiple disciplines including geology, hydrology, biology, chemistry, physics, medicine, engineering, management, economics, law, etc. Environmental engineering involves water supply, pollution control, recycling, waste (solid and liquid) disposal, radiation protection, industrial hygiene, environmental sustainability, and public health. This manual mainly deals with the determination of physio-chemical and bacteriological properties of water. This will also aid to determine optimum dosing for common physiochemical treatments. The manual contains relevant fundamental chemistry and biology concepts/theories and their applications in environmental engineering. The key tests include Physical, chemical and bacteriological tests of water and waste water. Sampling and laboratory analysis of air and solid waste are also discussed in this manual.

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", 1997.

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Water Sampling Methods for Lab Analysis

Meaning of Water Quality Analysis:

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 standard.

Requirement of Water Quality Analysis:

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 whether up gradation / 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.

Sampling of Water for Analysis:

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.

The objective of sampling is to collect a portion of material small enough in volume to be transported conveniently and yet large enough for analytical purposes while still accurately representing the material being sampled.

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, electric conductivity, etc. More complex measurements are often made in a laboratory requiring a water sample to be collected, preserved, transported, and analyzed at another location.

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.

Sample Collection bottles, Size and Materials:

The methods that will be followed will determine the type of bottles used. For example, samples for metals' analyses are usually collected in plastic bottles, while analyses for volatile organics and pesticides are collected in glass containers. Bottles used to collect samples for bacteria should be sterilized. Certain analysis like volatile organics and radon require vials that are to be filled leaving no head space, which keeps these analytes dissolved in the water, preventing them from escaping into the air. Additionally, some analyses require samples to be collected in amber colored bottles. These darker bottles are for analytes that break-down in sunlight, which helps keep these contaminants from breaking down while in transit to the laboratory for analysis. The size of the container is important to ensure enough sample to run the analysis needed.

Water Sampling Techniques:

Water sampling can be done in any of the following three methods depending on test requirements:

- Grab sampling
- Composite sampling
- Integrated sampling

Grab sampling:

Grab Samples are samples collected at a particular time and space. They represent the composition at that time and place. When a source is known to vary in time e.g.in case of waste effluents, grab samples collected at suitable time intervals and analyzed separately.

Composite sampling:

Composite samples are a mixture of grab samples collected at one sampling point at different times. The composite samples are useful for observing values. Individual samples are collected in wide mouth bottles every hour and mixed in volume proportional to the flow or by using specially designed automatic sampling devices.

Advantages of Composite Samples:

- reduced costs of analyzing a large number of samples.
- more representative samples of varied conditions.
- larger sample sizes when amounts of test samples are limited.

Disadvantages of Composite Samples

- loss of analytic relationships in individual samples.
- potential dilution of any parameter below detection levels.
- increased potential analytical interferences.
- increased possibility of analytic interactions.

Integrated sampling:

Integrated samples are a mixture of grab samples collected from different points simultaneously and mixed in equal volumes.

Surface Water Sampling Techniques:

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, 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.

When the water source is inaccessible

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

Ground Water (from well) Sampling Techniques

A bailer in 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

- Carefully clean and disinfect the inside and outside of the tap.
- Open the tap and let water flow for 2-3 minutes or until the water temperature has stabilized.
- 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.
- Take care not to allow the container to touch the tap.
- Take a water sample with the sample container.

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.

Assignment:

Collect your water sample from the location as instructed by the course teacher in the class. Follow the standard procedure during sample collection and preservation. Prepare a sample collection report (on A4 offset paper) describing details of sampling method (e.g. location, weather condition, details of surrounding features etc.) and also provide some pictures taken at the collection site.

Experiment 1
Determination of pH in water

Introduction:

The term pH refers to the measure of hydrogen ion concentration in a solution and defined as the negative log of H^+ ions concentration in water and wastewater

$$\mathbf{pH} = -\log\{H^+\} \qquad \mathbf{1.1}$$

Where $\mathbf{\{H^+\}}$ is the concentration (or activity) of hydrogen ion (or photon) in moles per liter (M).

Water dissociates to form hydrogen ion **(H+)** and hydroxyl ion **(OH-)** according to the following equation:

$$\{\mathbf{H_2O}\} = \{H^+\} + \{OH^-\} \qquad \mathbf{1.2}$$

At equilibrium, we can write,

$$K_W = \frac{\{H^+\}\{OH^-\}}{\{H_2O\}} \qquad \mathbf{1.3}$$

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

$$\mathbf{K_W} = \{H^+\}\{OH^-\} \qquad \mathbf{1.4}$$

Where, $\mathbf{K_w}$ = Equilibrium Constant

For pure water at 25 °C, $\mathbf{K_W = 10^{-7} \times 10^{-7} = 10^{-14}}$. This is known as the ion product of water or ionization constant for water. In other words, water (de-ionized or distilled water) at **25°C** dissociates to yield $\mathbf{10^{-7}}$**mol/L** of hydrogen ion $\mathbf{(H^+)}$ and $\mathbf{10^{-7}}$**mol/L** of hydroxyl ion $\mathbf{(OH^-)}$. Hence, according to Equation (1) pH of deionized water is equal to 7.

The values of **pH,**0 to a little less than 7 are termed as acidic and the values of **pH** a little above **7** to **14** are termed as basic. When the concentration of $\mathbf{H^+}$ and $\mathbf{OH^-}$ ions are equal then it is termed as neutral **pH**.

Environmental significance:

Determination of **pH** is one of the important objectives in biological treatment of the wastewater. In anaerobic treatment, if the **pH** goes below **5** due to excess accumulation of acids, the process is severely affected. Shifting of pH beyond **5** to **10** upsets the aerobic treatment of the wastewater. In these circumstances, the **pH** is generally adjusted by addition of suitable acid or alkali to optimize the treatment of the wastewater. **pH** value or range is of immense importance for any chemical reaction. A chemical shall be highly effective at a particular **pH**. Chemical coagulation, disinfection, water softening and corrosion control are governed by **pH** adjustment.

Lower value of **pH** below **4** will produce sour taste and higher value above **8.5** a bitter taste. Higher values of **pH** hasten the scale formation in water heating apparatus and also reduce the germicidal potential of chlorine. High **pH** induces the formation of tri-halomethanes, which are causing cancer in human beings.

Guideline:

According to **Bangladesh Environment Conservation Rules (1997)**, drinking water standard for **pH** ranges from **6.5** to **8.5**.

Principle:

The **pH** electrode used in the **pH** measurement is a combined glass electrode. It consists of sensing half-cell and reference half-cell, together form an electrode system. The sensing half-cell is a thin **pH** sensitive semi permeable membrane, separating two solutions, viz., the outer solution, the sample to be analyzed and the internal solution enclosed inside the glass membrane and has a known **pH** value. An electrical potential is developed inside and another electrical potential is developed outside, the difference in the potential is measured and is given as the **pH** of the sample.

Apparatus:

1. **pH** meter
2. Beaker

Reagent:

1. Buffers Solutions of known pH value

Sample handling and preservation

Preservation of sample is not practical. Because biological activity will continue after a sample has been taken, changes may occur during handling and storage. The characteristics of the water sample may change. To reduce the change in samples taken for the determination of **pH**, keep samples at **4°C**. Do not allow the samples to freeze. Analysis should begin as soon as possible.

Precautions

The following precautions should be observed while performing the experiment:

1. Temperature affects the measurement of **pH** at two points. The first is caused by the change in electrode output at different temperatures. This interference can be controlled by the instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second is the change of **pH** inherent in the sample at different temperatures. This type of error is sample dependent and cannot be controlled; hence both the **pH** and temperature at the time of analysis should be noted.
2. In general, the glass electrode is not subject to solution interferences like color, high salinity, colloidal matter, oxidants, turbidity or reductants.
3. Oil and grease, if present in the electrode layer, should be removed by gentle wiping or detergent washing, followed by rinsing with distilled water, because it could impair the electrode response.
4. Before using, allow the electrode to stand in dilute hydrochloric acid solution for at least 2 hours.
5. Electrodes used in the pH meter are highly fragile, hence handle it carefully.

Procedure:

Three major steps are involved in the experiment. They are

1. Preparation of Reagents
2. Calibrating the Instrument
3. Testing of Sample

Steps:

- Perform calibration of the **pH** meter using standard **pH** solutions. The calibration procedure would depend on the **pH** range of interest.
- In a clean dry **100 mL** beaker take the water sample and place it in a magnetic stirrer, insert the teflon coated stirring bar and stir well.
- Now place the electrode in the beaker containing the water sample and check for the reading in the **pH** meter. Wait until you get a stable reading.
- Take the electrode from the water sample, wash it with distilled water and then wipe gently with soft tissue.

Assignment

1. pH is one of the most important controlling factors for treatment and chemical analysis of water and wastewater — explain.
2. Define pH in terms of hydrogen-ion (H^+) concentration and hydroxyl-ion (OH^-) concentration. An increase in pH of one unit represents how much decrease in hydrogen ion concentration?

DATA SHEET

Experiment Name :
Experiment Date :

Student's Name :
Student's ID :
Year/ Semester :
Section :
Group :

Table

Sample No	Source of Sample	Temperature of Sample (°C)	pH

Signature of the course teacher

Experiment 2
Determination of Color in 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. The color value of water is extremely pH-dependent and invariably increases as the pH of the water is raised. For this reason recording pH along with color is advised.

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 tri-halomethanes including chloroform and a range of other chlorinated organics leading to problems which is a major concern in water treatment. So it is important to limit the color of water for domestic supplies.

Guideline:

According to Bangladesh Environment Conservation Rules (1997), drinking water guideline value for color is 15 Pt-Co Unit.

Theory on experimental method:

Available methods for determining color of water:

1. Standard Color Solutions Method
2. Dilution Multiple Method
3. Spectrophotometric method

1. Standard color solution method

Waters containing natural color are yellow-brownish in appearance.

Standard Color Solution: Solutions of potassium chloroplatinate (K_2PtCl_6) tinted with small amounts of cobalt chloride yield colors that are very much like the natural colors. In this method, the color produced by 1 mg/l of platinum (as K_2PtCl_6) and 0.5mg/l of cobalt (as $CoCl_2 \cdot 6H_2O$) is taken as the standard one unit of color.

Usually, a stock solution stock solution of K_2PtCl_6 that contains 500mg/l of platinum is prepared, which has a color of 500 units. Then, a series of working standards may be prepared from it by dilution.

Color -comparison tubes are usually used to contain the standards. A series ranging from 0 to 70 color units is employed and samples with color less than 70 units are tested by direct comparison with the prepared standards. For samples with a color greater than 70 units, a dilution is made with distilled water distilled water to bring the resulting color within the range of the standards. In this case, the final result should be corrected using a dilution factor.

2. Dilution multiple method

Color of most domestic and industrial waste waters are not yellow-brownish hue.

Other systems of measurement have to be used to measure and describe colors that do not fall into this classification.

For dilution multiple methods, color is measured by successive dilutions of the sample with color -free water until the color is no longer detectable comparing with distilled water. The total dilution multiple is calculated and used to express the color degree.

3. Spectrophotometric method

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 nm). 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, 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.

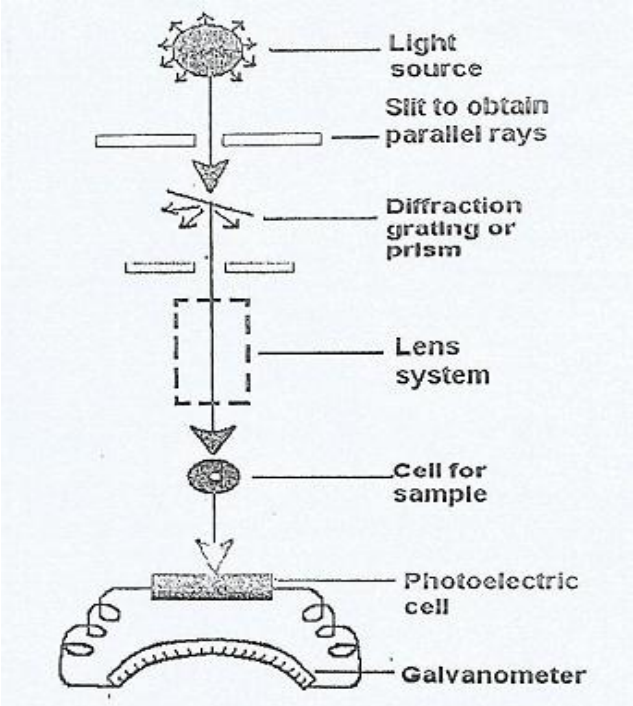
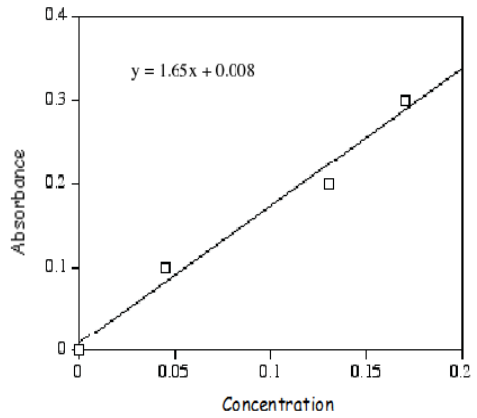


Figure 2.1: Optical property used in measurement of color in water using spectrophotometer



Slope of the best straight line through the data points in the calibration plot is 1.65. Plot intercept is 0.008.

$$\text{Slope} = \frac{\Delta(\text{absorbance})}{\Delta(\text{concentration})} = 1.65$$

Equation of straight line:

Absorbance = 1.65 (Concentration) + 0.008

To find an unknown concentration for a sample, subtract the intercept from the absorbance reading and divide the result by the slope. Here the equation would be

$$\text{Concentration} = \frac{\text{Absorbance} - 0.008}{1.65}$$

Figure 2.2: Calibration Curve of spectrophotometer

Apparatus:

1. Color Disk
2. Filtration system including filter paper, funnel, holder, beaker etc.

Reagent:

1. Standard potassium chloro-platinate solution

Procedure:

1. Prepare standard samples having color within a specific range by mixing different concentration of standard potassium chloroplatinate solution with distilled water. Using these samples to prepare a color calibration curve (absorbance vs. color concentration) for the spectrophotometer.
2. Take 50-mL of filtered test sample in a beaker. Take 50-mL distilled water in another beaker. Use this sample as blank.
3. Set the spectrophotometer to determine color concentration of the sample.
4. Put the blank sample inside the spectrophotometer cell and set the reading "zero".
5. Bring out the blank sample and place the test sample inside the spectrophotometer
6. After a While the display will show the color concentration of the sample.

Assignment:

1. Define true and apparent color. Why true color is more important than apparent color?
2. Discuss the optical mechanism used in color measurement using spectrophotometer.

DATA SHEET

Experiment Name :
Experiment Date :

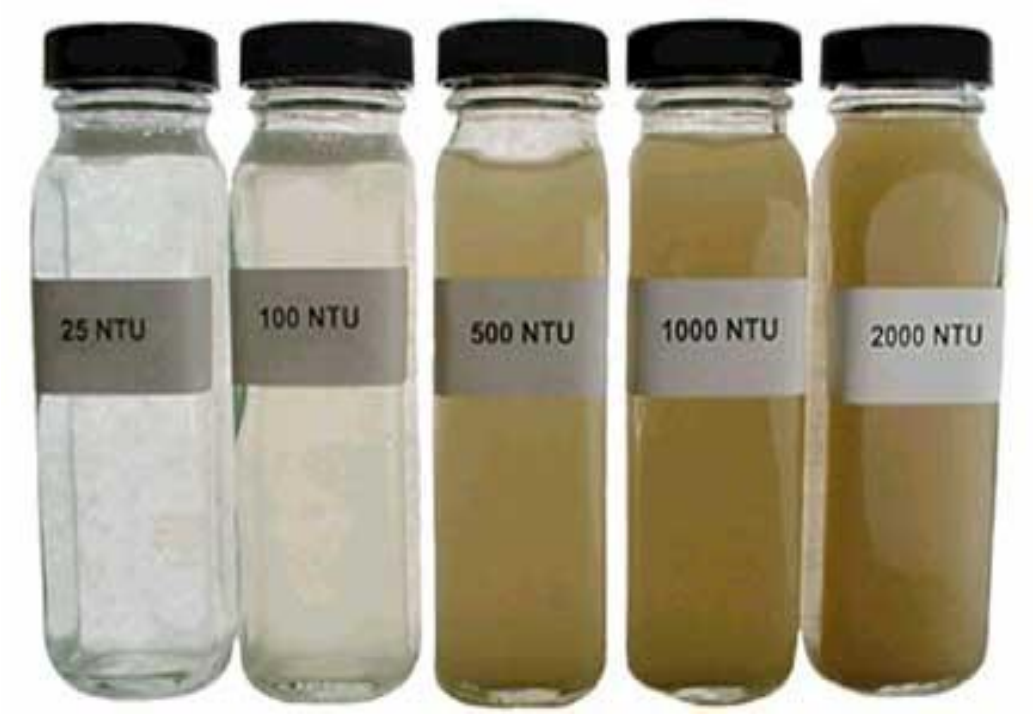
Student's Name :
Student's ID :
Year/ Semester :
Section :
Group :

Table

Sample No	Source of Sample	Temperature of Sample (°C)	Color (Pt-co)

Signature of the course teacher

Experiment 3
Determination of Turbidity of Water



Introduction:

Turbidity is the technical term referring to the cloudiness of a solution and it is a qualitative characteristic which is imparted by solid particles obstructing the transmittance of light through a water sample. Turbidity often indicates the presence of dispersed and suspended solids like clay, organic matter, silt, algae and other microorganisms. So in short turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in straight lines through the sample.

Environmental significance:

When the turbid water in a small, transparent container such as drinking glass is held up to the light, an aesthetically displeasing opaqueness or milky coloration is apparent. The colloidal material which exerts turbidity provides adsorption sites for chemicals and for biological organism that may not be harmful. They may be harmful or cause undesirable tastes and odours. Disinfection of turbid water is difficult because of the adsorptive characteristics of some colloids and because the solids may partially shield organisms from disinfectant. In natural water bodies, turbidity may impart a brown or other color to water and may interfere with light penetration and photosynthetic reaction in streams and lakes. Turbidity increases the load on slow sand filters.

The filter may go out of operation, if excess turbidity exists. Knowledge of the turbidity variation in raw water supplies is useful to determine whether a supply requires special treatment by chemical coagulation and filtration before it may be used for a public water supply. Turbidity measurements are used to determine the effectiveness of treatment produced with different chemicals and the dosages needed. Turbidity measurements help to gauge the amount of chemicals needed from day-to-day operation of water treatment works. Measurement of turbidity in settled water prior to filtration is useful in controlling chemical dosages so as to prevent excessive loading of rapid sand filters. Turbidity measurements of the filtered water are needed to check on faulty filter operation. Turbidity measurements are useful to determine the optimum dosage of coagulants to treat domestic and industrial wastewaters. Turbidity determination is used to evaluate the performance of water treatment plants.

Turbidity in water may be caused by a wide variety of suspended matter suspended matter, such as clay, silt, finely divided organic and inorganic matter, soluble colored organic compounds, and other organisms. Under flood conditions, great amounts of topsoil are washed to receiving streams. As the rivers pass through urban areas, the domestic and industrial wastewaters may be added.

Guideline:

According to WHO standard 5 NTU is suggested as the turbidity limit for drinking water, while 1 NTU is recommended to achieve the adequate disinfecting safety.
According to Bangladesh Environment Conservation Rules (1997), drinking Water standard for Turbidity is 10 NTU (Nephelometric turbidity unit).

Principle:

Turbidity is based on the comparison of the intensity of light scattered by the sample under defined conditions with the intensity of the light scattered by a standard reference suspension under the same conditions. The turbidity of the sample is thus measured from the amount of light scattered by the sample taking a reference with standard turbidity suspension. The

higher the intensity of scattered light the higher is the turbidity. Formazin polymer is used as the primary standard reference suspension.

Because of the wide variety of materials that cause turbidity in natural waters, it has been necessary to use an arbitrary standard. The original standard chosen was; **1 mg SiO_2/L =1 unit of turbidity.** The silica used had to meet certain specifications as to particle size. The Jackson candle turbidimeter has been replaced by more reliable, sensitive, and easier to use instruments that depend upon the principle of nephelometry. As a standard reference material, Silica has been replaced by formazin polymer. The formazin suspensions were first calibrated against the Jackson candle turbidimeter. The standard nephelometry procedure is now reported in nephelometric turbidity units (NTU). Because the basic principles difference for Jackson candle turbidimeter method and nephelometric method, results got from the two methods can vary widely. In order to avoid any confusion this may cause, turbidity measurements by the standard nephelometry procedure are now reported in nephelometric turbidity units (NTU), and the other one is reported in Jackson candle turbidimeter units (JTU).

40 NTU are about equivalent to 40 JTU.

The applicable range of this method is 0-40 nephelometric turbidity units (NTU). Higher values may be obtained with dilution of the sample.

Sample handling and preservation:

Water samples should be collected in plastic cans or glass bottles. All bottles must be cleaned thoroughly and should be rinsed with turbidity free water. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis (if required), and minimize waste disposal. No chemical preservation is required. Keep the samples at 4°C. Do not allow samples to freeze. Analysis should begin as soon as possible after the collection. If storage is required, samples maintained at 4°C may be held for up to 48 hours.

Precautions:

The following precautions should be observed while performing the experiment:

- The presence of coloured solutes causes measured turbidity values to be low. Precipitation of dissolved constituents (for example, Fe) causes measured turbidity values to be high.
- Light absorbing materials such as activated carbon in significant concentrations can cause low readings.
- The presence of floating debris and coarse sediments which settle out rapidly will give low readings. Finely divided air bubbles can cause high readings.

Apparatus:

1. Turbidity Meter

Chemicals

1. Formazin Polymer standards

Procedure:

1. For testing the given water sample first the reagents are to be prepared. Then the turbidity meter is required to be calibrated.
2. To the sample cells, add sample water up to the horizontal mark, wipe gently with soft tissue and place it in the turbidity meter. Cover the sample cell with the light shield.
3. Check for the reading in the turbidity meter. Wait until you get a stable reading.

Assignment

1. Why turbidity is important in "filtration" and "disinfection" processes?

DATA SHEET

Experiment Name :
Experiment Date :

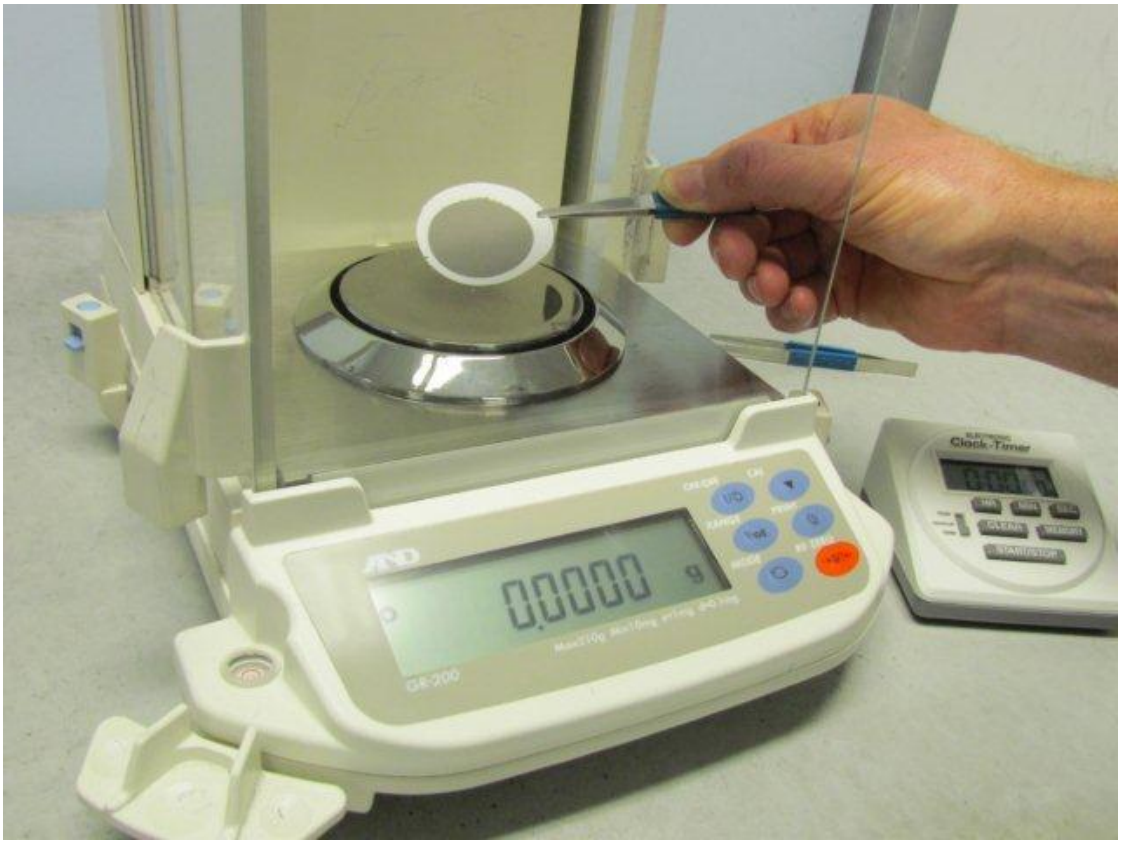
Student's Name :
Student's ID :
Year/ Semester :
Section :
Group :

Table

Sample No	Source of Sample	Temperature of Sample (°C)	Turbidity (NTU)

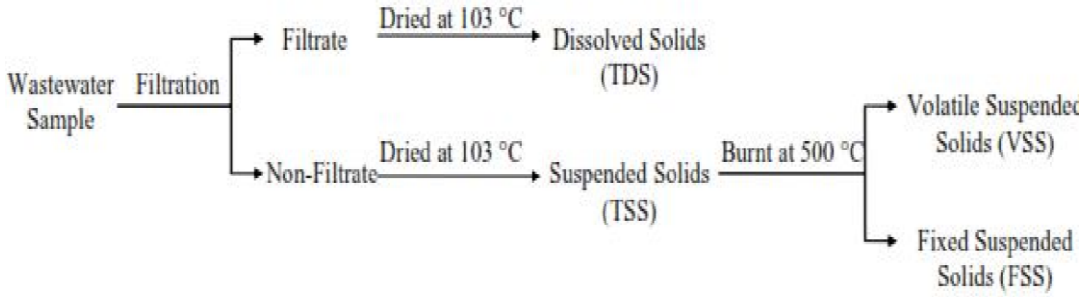
Signature of the course teacher

Experiment 4
Determination of Total Solids, Dissolved Solids and Suspended Solids in Water



Introduction:

Environmental engineering is concerned with the solid material in a wide range of natural waters and wastewaters. The usual definition of solids (referred to as "total solids") is the matter that remains as residue upon evaporation at 103~105°C. The various components of "total solids" can be simplified as follows



Total Solids (TS) are the total of all solids in a water sample. They include the total suspended solids and total dissolved solids. Total Suspended Solids (TSS) is the amount of filterable solids in a water sample. Samples are filtered through a glass fiber filter. The filters are dried and weighed to determine the amount of total suspended solids in mg/l of sample. Total Dissolved Solids (TDS) are those solids that pass through a filter with a pore size of 2.0 micron (1/1000000th of a meter, Also known as a Micrometer) or smaller. They are said to be non-filterable. After filtration the filtrate (liquid) is dried and the remaining residue is weighed and calculated as mg/l of Total Dissolved Solids.

Environmental significance:

Total solids measurements can be useful as an indicator of the effects of runoff from construction, agricultural practices, logging activities, sewage treatment plant discharges, and other sources. Total solids also affect water clarity. Higher solids decrease the passage of light through water, thereby slowing more rapidly and hold more heat; this, in turn, might adversely affect photosynthesis by aquatic plants. Water will heat up affect aquatic life that has adapted to a lower temperature regime. As with turbidity, concentrations often increase sharply during rainfall, especially in developed watersheds. They can also rise sharply during dry weather if earth-disturbing activities are occurring in or near the stream without erosion control practices in place. Regular monitoring of total solids can help detect trends that might indicate increasing erosion in developing watersheds. Total solids are related closely to stream flow and velocity and should be correlated with these factors. Any change in total solids over time should be measured at the same site at the same flow. Water with total solids generally is of inferior palatability and may induce an unfavorable physiological reaction. It may be esthetically unsatisfactory for purposes such as bathing. Total solids will be higher in highly mineralized waters, which result in unsuitability for many industrial applications. It indicates effectiveness of sedimentation process and it affects effectiveness of disinfection process in killing microorganisms. It is used to assess the suitability of potential supply of water for various uses. In the case of water softening, amount of total solids determine the type of softening procedure. Corrosion control is frequently accomplished by the production of stabilized waters through pH adjustment. The pH stabilization depends to some extent upon the total solids present as well as alkalinity and temperature.

Solids analyses are important in the control of biological and physical wastewater treatment processes and for assessing compliance with regulatory agency wastewater effluent limitations

Although the waste water or sewage normally contains 99.9 percent of water and only 0.1 percent of solids, but it is the solids that have the nuisance value. The amount of solids in wastewater is frequently used to describe the strength of the water. The more solids present in a particular wastewater, the stronger that wastewater will be. The environmental impacts of solids in all forms have detrimental effects on quality since they cause putrefaction problems. If the solids in wastewater are mostly organic, the impact on a treatment plant is greater than if the solids are mostly inorganic.

In the realm of municipal wastewater, suspended solids analysis is by far the most important gravimetric method. It is used to evaluate the strength of the raw wastewater as well as the overall efficiency of treatment. Furthermore, most waste water treatment plants (WWTP's) have effluent standards of 10 to 30 mg/L suspended solids which may be legally enforceable. As was the case with municipal wastewater, suspended solids analysis is useful as a means of assessing the strength of industrial wastewaters and the efficiency of industrial wastewater treatment.

Some Typical Solids Concentrations

Source		Concentration (mg/L)		
		Low	Avg	High
Fresh Natural Water	TDS	20	120	1,000
Raw Domestic Wastewater	TDS	350	600	900
	TSS	100	200	350
Storm Water	TSS	5	300	3,000

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. Water with higher solids content often has a laxative and sometimes the reverse effect upon people whose bodies are not adjusted to them. Estimation of total dissolved solids is useful to determine whether the water is suitable for drinking purpose, agriculture and industrial purpose. Suspended material is aesthetically displeasing and provides adsorption sites for chemical and biological agents. Suspended organic solids which are degraded anaerobically may release obnoxious odors. Biologically active suspended solids may include disease causing organisms as well as organisms such as toxic producing strains of algae. The suspended solids parameter is used to measure the quality of wastewater influent and effluent. Suspended solids determination is extremely valuable in the analysis of polluted waters. Suspended solids exclude light, thus reducing the growth of oxygen producing plants. High concentration of dissolved solids about 3000 mg/L may also produce distress in livestock. In industries, the use of water with high amount of dissolved solids may lead to scaling in boilers, corrosion and degraded quality of the product.

Guideline:
According to Bangladesh Environment Conservation Rules (1997), potable water should not contain more than 1000 mg/l of total dissolved solids (TDS).

Principle:
The measurement of solids is by means of the gravimetric procedure. The various forms of solids are determined by weighing after the appropriate handling procedures. The total solids concentration of a sample can be found directly by weighing the sample before and after drying at 103°C. However, the remaining forms, TDS and TSS require filtration of the sample. For liquid samples, all these solids levels are reported in mg/L.

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 electric 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.

Sample handling and preservation:
Preservation of sample is not practical. Because biological activity will continue after a sample has been taken, changes may occur during handling and storage. Both the characteristics and the amount of solids may change. To reduce this change in samples taken for solids determinations, keep all samples at 4° C. Do not allow samples to freeze. Analysis should begin as soon as possible.

Precautions:
The following precautions should be observed while performing the experiment:

- Water or Wastewater samples which contain high concentrations of calcium, chloride, magnesium or sulphate can rapidly absorb moisture from the air. Such samples may need to be dried for a longer period of time, cooled under proper desiccation and weighed rapidly in order to achieve a reasonable constant weight. We should be aware prolonged drying may result in loss of constituents, particularly nitrates and chlorides.
- Non-representative particulates such as leaves, sticks, fish and lumps of fecal matter should be excluded from the sample if it is determined that their inclusion is not desired in the final result.
- Floating oil and grease, if present, should be included in the sample and dispersed by a blender device before sub-sampling.
- Volume of sample should be adjusted to have residue left after drying as 100 to 200mg. It is mainly to prevent large amount of residue in entrapping water during evaporation.
- Highly mineralized water containing significant concentration of calcium, magnesium, chloride, and/or sulphate may be hygroscopic. Hence prolonged drying, desiccation and rapid weighing.
- We should be aware prolonged drying may result in loss of constituents, particularly nitrates and chlorides.

Apparatus:

1. Balance
2. Beaker
3. Measuring Cylinder
4. Filter paper
5. Funnel
6. Dropper

Procedure:

Measurement of Total Solids (TS)

(1) Take a clear dry glass beaker (which was kept at 103°C in an oven for 1 hour) of 150ml. capacity and put appropriate identification mark on it. Weight the beaker and note the weight.
(2) Pour 100ml. of the thoroughly mixed sample, measured by the measuring cylinder, in the beaker.
(3) Place the beaker in an oven maintained at 103°C for 24hours. After 24 hours, cool the beaker and weight. Find out the weight of solids in the beaker by subtracting the weight of the clean beaker determined in step (1)
(4) Calculate total solids (TS) as follows:

Measurement of Total Dissolved Solids (TDS)

(1) Same as above (step 1 of total solids).
(2) Take a 100 ml. of sample and filter it through a double layered filter paper and collect the filtrate in a beaker.
(3) The repeat the same procedure as in steps (3) and (4) of the total solids determination and determine the dissolved solids contents as follows:

Calculation:

Total solids, TS (mg/l) = mg of solids in the beaker x 1000 / (volume of sample)

Total Dissolved Solids, TDS (mg/l) =mg of solids in the beaker x1000 /(volume of sample)

Total Suspended Solids, TSS (mg/l) = TS (mg/l) – TDS (mg/l)

Assignment:

1. Discuss possible sources of solids in ground water and surface water.
2. “Groundwater usually has higher dissolved solids and surface water usually has higher suspended solids”- Explain.
3. Why water is evaporated at 103°C rather than 100 °C in assessment of solid of water?

DATA SHEET

Experiment Name :
Experiment Date :

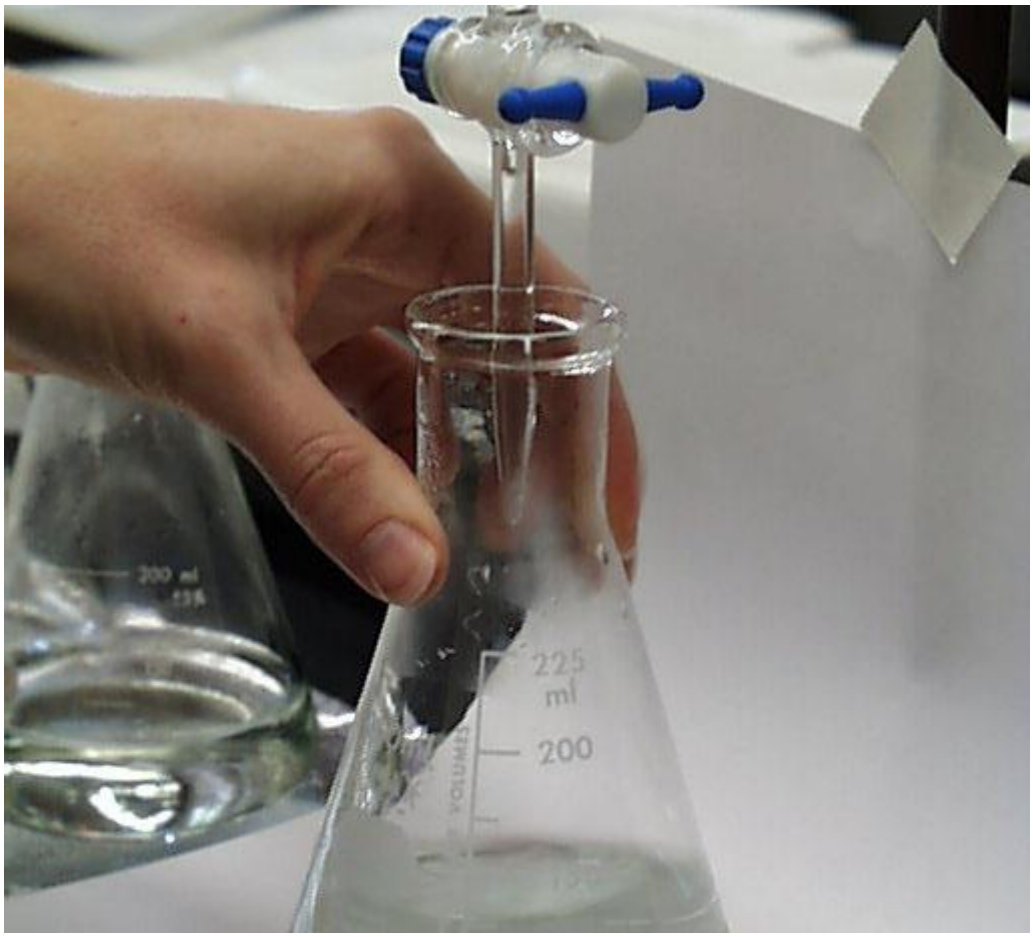
Student's Name :
Student's ID :
Year/ Semester :
Section :
Group :

Table

Sample No	Source of Sample	TS (mg/L)	TDS (mg/L)	TSS (mg/L)

Signature of the course teacher

Experiment 5
Determination of CO_2 in water



Introduction:

Carbon Dioxide is present in water in the form of a dissolved gas. Surface waters normally contain less than 10 ppm free carbon dioxide, while some ground waters may easily exceed that concentration. Carbon dioxide is readily soluble in water. Over the ordinary temperature range (0-30°C) the solubility is about 200 times that of oxygen. Calcium and magnesium combine with carbon dioxide to form carbonates and bicarbonates.

Carbon dioxide does dissolve in water; however the system is somewhat complex. First the CO_2 dissolves according to:

CO_2 (g) = CO_2 (l) **5.1**

At room temperature, the solubility of carbon dioxide is about 90 cm^3 of CO_2 per 100 ml water (c_l/c_g = 0.8). Any water-soluble gas becomes more soluble as the temperature decreases, due to the thermodynamics of the reaction:

GAS (l) = GAS (g) **5.2**

This effect is particularly large for gases like CO_2 that undergo specific reactions with water. Equilibrium is established between the dissolved CO_2 and H_2CO_3, carbonic acid.

CO_2 (l) + H_2O (l) = H_2CO_3 (l) **5.3**

Carbonic acid is a weak acid that dissociates in two steps.

$$H_2CO_3 + H_2O \Leftrightarrow H_3O^+ + HCO_3^-$$
$$HCO_3^- + H_2O \Leftrightarrow H_3O^+ + CO_3^{2-}$$

The test for determination of free carbon dioxide in water is based on the titration of water sample with standard Sodium Hydroxide solution in the presence of phenolphthalein indicator. The CO_2 reacts with NaOH to form Sodium Bicarbonate with a consequent increase in pH. The reaction of NaOH with free CO_2 reach a completion at a pH of 8.3.

$$CO_2 + H_2O = H_2CO_3$$
$$H_2CO_3 + NaOH = HCO_3^- + Na^+ + H_2O$$
$$HCO_3^- + NaOH = CO_3^{2-} + Na^+ + H_2O$$

Environmental significance:

Aquatic plant life depends upon carbon dioxide and bicarbonates in water for growth. Microscopic plant life suspended in the water, phytoplankton, as well as large rooted plants, utilize carbon dioxide in the photosynthesis of plant materials; starches, sugars, oils, proteins. The carbon in all these materials comes from the carbon dioxide in water.

When the oxygen concentration in waters containing organic matter is reduced, the carbon dioxide concentration rises. The rise in carbon dioxide makes it more difficult for fish to use the limited amount of oxygen present. To take on fresh oxygen, fish must first discharge the carbon dioxide in their blood streams and this is a much slower process when there are high concentrations of carbon dioxide in the water itself.

Corrosion is the principal difficulty caused by carbon dioxide. This gas on solution in water produces carbonic acid resulting in lowering of pH. With a decrease in pH corrosive characteristics is induced in water resulting severe corrosion of heat exchanger, pipes, valves etc. Corrosion in boiler system takes place due to the presence of carbonate and bicarbonate although Carbon dioxide is not present in this case.

Guidelines:

Bangladesh Environment Conservation Rules (1997) does not set any limits for the presence of CO_2 in water.

Precaution:

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 ground water samples which usually contain high CO_2. 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 CO_2 to the atmosphere during the titration process. To check this, it is advisable to secure a second sample. Upon complete determination of CO_2 on the first sample, then take the second sample (100ml) and immediately add the full amount of N/44 sodium hydroxide solution used in the titration of the first sample. Add 10 drops of phenolphthalein indicator and if the sample remain colourless, add additional N/44 sodium hydroxide to the end point (till the slight pink colour appears) and accepting this second test as more accurate titration.

Apparatus:

1. Beaker
2. Measuring cylinder
3. Dropper
4. Stirrer
5. Burette

Reagent:

1. Standard N/44 Sodium Hydroxide
2. Phenolphthalein Indicator **

**An indicator is a substance that undergoes a change in color when the end-point of a titration is reached. Acid-base indicators are used to signal the end of acid-base titrations. An acid-base indicator is itself a weak acid (or its conjugate base).

Phenolphthalein is a commonly used indicator for titrations, and is a weak acid. The weak acid is colorless and its ion is bright pink. Adding extra hydrogen ions shifts the position of equilibrium to the left, and turns the indicator colorless. Adding hydroxide ions removes the hydrogen ions from the equilibrium which tips to the right to replace them - turning the indicator pink. The half-way stage happens at pH 8.3. Since a mixture of pink and colorless is simply a paler pink, this is difficult to detect with any accuracy.

Procedure:

1. Take a 100 ml of sample in a beaker and add 10 drops of Phenolphthalein indicator. If a pink color develops, no carbon dioxide is present in the water sample.
2. Add N/44 sodium Hydroxide solution from a burette to the sample and stir gently until a slight permanent pink color appears as compared with distilled water. Record ml of sodium hydroxide used. Since excess CO_2, if present easily escapes to atmosphere, so tests should be performed immediately after collection of water sample. If this is not possible sample bottle should be completely filled and stoppered and be kept at a temperature lower than that at which it was collected.

Calculation:

Carbon dioxide (mg/L) =mL of N/44 NaOH used x Multiplying Factor (M.F.)

Where, **M.F.** = $$\frac{\text{Normality of NaOH x equivalent wt.of CO2 x 1000}}{\text{mL of sample taken}}$$

Determination of CO_2 acidity:

Phenolphthalein acidity (often called CO_2 acidity) of water is defined as the amount of standard base (usually 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:

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

Assignment:

1. Discuss changes in the form of Carbon in solution at different pH.
2. Why does groundwater contain higher carbon dioxide then surface water?
3. Why test for Carbon dioxide should be performed immediately after collection of water sample?

DATA SHEET

Experiment Name :
Experiment Date :

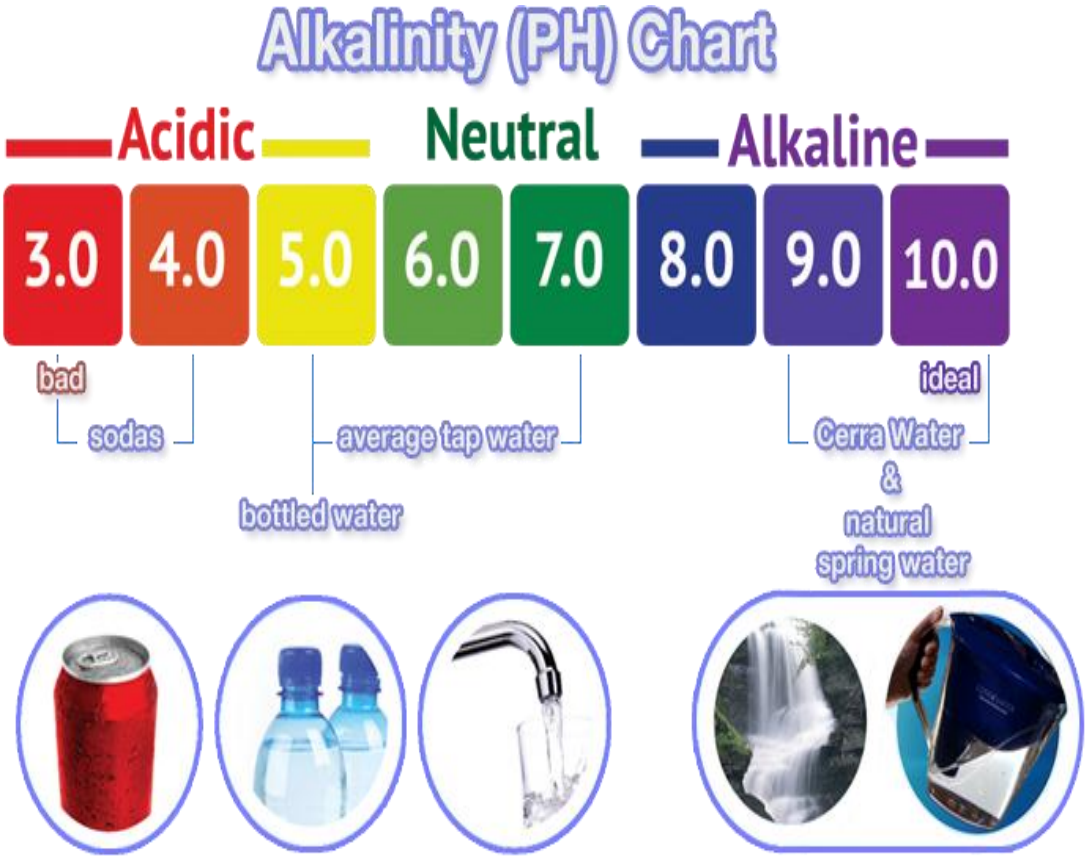
Student's Name :
Student's ID :
Year/ Semester :
Section :
Group :

Table

Sample No and source	Temperature of Sample (°C)	Volume of Sample (mL)	Burette Reading (mL)		Volume of NaOH (mL)	CO_2 Concentration (mg/L)	CO_2 acidity (mg/L as CaCO3)
			Initial	Final			

Signature of the course teacher

Experiment 6
Determination of Alkalinity in Water



Introduction:
Alkalinity is primarily a way of measuring the acid neutralizing capacity of water. In other words, its ability to maintain a relatively constant pH. The possibility to maintain constant pH is due to the hydroxyl, carbonate and bicarbonate ions present in water. The ability of natural water to act as a buffer is controlled in part by the amount of calcium and carbonate ions in solution.
Carbonate ion and calcium ion both come from calcium carbonate or limestone. So water that comes in contact with limestone will contain high levels of both Ca^{++} and CO_3^{2-} ions and have elevated hardness and alkalinity.

Environmental significance:
Alkalinity is important for fish and aquatic life because it protects or buffers against rapid pH changes. Higher alkalinity levels in surface waters will buffer acid rain and other acid wastes and prevent pH changes that are harmful to aquatic life. Large amount of alkalinity imparts bitter taste in water. The principal objection of alkaline water is the reactions that can occur between alkalinity and certain actions in waters. The resultant precipitate can corrode pipes and other accessories of water distribution systems.

Wastewaters containing excess caustic (hydroxide) alkalinity are not to be discharged into natural water bodies or sewers. Alkalinity as carbonate and bicarbonate of saline water is very important in tertiary recovery processes for recovering petroleum. Alkaline water offers better wetting to the formation rock and improve oil release. As an additional benefit, ions that provide alkalinity absorb on rock surfaces occupying adsorption sites and decrease the loss of recovery chemical by adsorption. The alkalinity value is necessary in the calculation of carbonate scaling tendencies of saline waters.

The alkalinity acts as a pH buffer in coagulation and lime-soda softening of water. In wastewater treatment, alkalinity is an important parameter in determining the amenability of wastes to the treatment process and control of processes such as anaerobic digestion, where bicarbonate alkalinity, total alkalinity, and any fraction contributed by volatile acid salts become considerations.

Principle:
The alkalinity of water can be determined by titrating the water sample with sulphuric acid of known values of pH, volume and concentrations. Based on stoichiometry of the reaction and number of moles of sulphuric acid needed to reach the end point, the concentration of alkalinity in water is calculated. When a water sample that has a pH of greater than 4.5 is titrated with acid to a pH 4.5 end point, all OH^-, CO_3^{2-}, and HCO_3^- will be neutralized.

For the pH more than 8.3, add phenolphthalein indicator, the colour changes to pink colour. This pink colour is due to presence of hydroxyl ions. If sulphuric acid is added to it, the pink colour disappears i.e. OH^- ions are neutralized. Then add methyl orange indicator, the presence of CO_3^{2-} and HCO_3^- ions in the solution changes the colour to yellow. While adding sulphuric acid, the color changes to slight orange ting, this color change indicates that all the CO_3^{2-} and HCO_3^- ions has been neutralized. This is the end point.

Apparatus:

1. Burette with Burette stand and porcelain title
2. Pipettes with elongated tips
3. Conical flask
4. Measuring cylinders
5. Beakers
6. Dropper
7. Stirrer

Chemicals

1. Standard0.02N sulphuric acid
2. Phenolphthalein indicator
3. Methyl orange indicator

Sample handling and preservation:

Preservation of sample is not practical. Because biological activity will continue after a sample has been taken, changes may occur during handling and storage. To reduce the change in samples, keep all samples at 4°C. Do not allow samples to freeze. Analysis should begin as soon as possible. Do not open sample bottle before analysis.

Procedure:

1. Measure 50 ml or 100 ml of your sample into a 250 mL beaker or erlenmyer flask. Place your sample onto a stir plate (make sure to put a bar magnet in the flask).
2. Measure initial pH of your sample. If the sample pH is below 8.3 (if above 8.3, do step **3** first), add several drops of methyl orange indicator. If the color of the solution turned yellow, titrate your sample with 0.02 N H_2SO_4 (you may need to dilute the acid provided in the lab) until the color changes to slightly orange ting (pH 4.5). Record the total volume of acid used for the titration.
3. Measure initial pH of your sample. If the sample pH is above 8.3, add several drops of phenolphthalein indicator. If the color of the solution turned pink, titrate your sample with 0.02 N H_2SO_4 or HCl (you may need to dilute the acid provided in the lab) until color changes from pink to clear (pH 8.3). Record the volume of acid used for the titration. Then, proceed with step **2**.
4. Calculate both Phenolphthalein Alkalinity and Total Alkalinity using the formula provided above.

Calculation:

Phenolphthalein Alkalinity (mg/L as $CaCO_3$)

= Multiplying Factor (MF) x milliliter of 0.02N H_2SO_4*(added up to pH 8.3)*

Total Alkalinity (mg/L as $CaCO_3$)

=Multiplying Factor (MF) x milliliter of 0.02N H_2SO_4*(added up to pH approx. 4.5)*

Where, **M.F.** = $$\frac{\text{Normality of } H_2SO_4 \text{ x equivalent wt.of } CaCO_3 \text{ x } 1000}{\text{mL of sample taken}}$$

Assignment:

1. Discuss the importance of alkalinity in water for different treatment processes.
2. Define total alkalinity, phenolphthalein alkalinity, and methyl orange alkalinity. Discuss their dominant pH range.

DATA SHEET

Experiment Name :
Experiment Date :

Student's Name :
Student's ID :
Year/ Semester :
Section :
Group :

Table -1: Determination of Phenolphthalein Alkalinity:

Sample No and source	Volume of Sample (mL)	Burette Reading (mL)		Volume of Sulphuric acid (mL)	Phenolphthalein Alkalinity (mg/L as CaCO3)
		Initial	Final		

Table – 2: Determination of Total Alkalinity:

Sample No and source	Volume of Sample (mL)	Burette Reading (mL)		Volume of Sulphuric acid (mL)	Total Alkalinity (mg/L as CaCO3)
		Initial	Final		

Signature of the course teacher

Experiment 7
Determination of Hardness in Water

Introduction:

Hard waters are generally considered to be those waters that require considerable amounts of soap to produce foam or leather and that also produce scale in hot-water pipes, 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 ion and manganaous ions. These cations and the important anions with they are associated with are shown in the following table 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-7.1: 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+}	NO3-
Mn^{2+}	SiO_3^{2-}

Hardness caused by each cation can be calculated as follows:

$$\text{Hardness (mg/l as } CaCO_3) = \frac{M^{2+}(mg/l)\times 50}{Equivalent\ weight\ of\ M^{2+}} \qquad \textbf{7.1}$$

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

$$\text{Total hardness} = \sum\left[\frac{50}{20.04}Ca^{2+} + \frac{50}{12.15}Mg^{2+} + \frac{50}{27.92}Fe^{2+} + \frac{50}{27.46}Mn^{2+}\right] \qquad \textbf{7.2}$$

$$= \sum\left[2.49\,Ca^{2+} + 4.12\,Mg^{2+} + 1.79\,Fe^{2+} + \frac{50}{27.46}Mn^{2+}\right] \qquad \textbf{7.3}$$

Environmental significance:

Hard water is as satisfactory for human consumption as soft waters. Because of their 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 developments 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. Change 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 follows:

Table 7.2: 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 the complete cation analysis (Equation 7.1 – 7.3). This method is preferred where complete analysis are available.

However, complete analysis of metal ions is 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 methods of choice in most laboratories.

The EDTA titrimetric method involves the use of solutions of ethylene-diamine-tetra-acidic acid (EDTA) or its sodium salt as the titrating agents. 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 hardens, as shown in the following equation.

$$M^{2+} + EDTA = [M.EDTA]_{complex} \quad 7.4$$

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 Erichome Black T dye serves as an excellent indicator to show when all the hardness ions having form complex with EDTA. When small amount of Erichrome Black T 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 produce wine red color in the solution.

$$M^{2+} + \text{Erichrome Black T} = (\text{M. Erichrome Black T})_{complex} \quad 7.5$$

During titration with EDTA, all free hardness ions are complexed according to euqtion7.2. Finally, the EDTA disrupts the weak wine red complex compounds (M. Erichrome Black T) and forms more stable complex with the divalent ions. The action frees the Erichrome Black T indicator, and the wine red colour changes to a distinct blue colour.

$$(\text{M. Erichrome Black T})_{complex} + EDTA = (M.EDTA)_{complex} + \text{Erichrome Black T} \quad 7.6$$
(blue color)

Reagents:

1. Buffer solution (for attaining a pH close to 10.0)
2. Erichrome Black T dye
3. Standard EDTA solution

Apparatus:

1. Beaker
2. Measuring cylinder
3. Dropper
4. Stirrer
5. Auto-titration device

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.
3. Add one packet of Erichome 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 (supplied by 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 colour of the sample changes to blue. Take final reading of the counter.

Calculation:

Total hardness (mg/L as $CaCO_3$)

= Multiplying Factor (MF) x (initial counter reading – final counter reading)

Where, **M.F.** = $$\frac{\text{Normality of EDTA x equivalent wt.of } CaCO_3 \text{ x } 1000}{\text{mL of sample taken}}$$

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

Therefore for 100ml sample,
Total hardness (mg/L as $CaCO_3$) = final counter reading – initial counter reading

And for 50ml sample,
Total hardness (mg/L as $CaCO_3$) = 2× (final counter reading – initial counter reading)

DATA SHEET

Experiment Name :
Experiment Date :

Student's Name :
Student's ID :
Year/ Semester :
Section :
Group :

Table

Sample No and source	Temperature of Sample (°C)	Volume of Sample (mL)	Burette Reading (mL)		Volume of EDTA solution (mL)	Hardness Concentration (mg/L as CaCO3)
			Initial	Final		

Signature of the course teacher

Experiment 8
Determination of Chloride in water

Introduction:

Chlorides occur in all natural waters in widely varying concentration, 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 urine, contain chloride in an amount about equal to the chlorides consumed with food and water. This amount average about 6 gm of chlorides per person per day and increases the amount of CC 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. For this reason, chlorides are generally limited to 250 mg/L in supplies intended for public use. In many areas of the world where water supplies are scarce, source be 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.

Guideline:

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.

Principle: (Mohr's Method)

This method determines the chloride ion concentration of a solution by titration with silver nitrate. As the silver nitrate solution is slowly added, a precipitate of silver chloride forms.

$$Ag^{+}_{(aq)} + Cl^{-}_{(aq)} \rightarrow AgCl_{(s)} \qquad \textbf{8.1}$$

The end point of the titration occurs when all the chloride ions are precipitated. Then additional silver ions react with the chromate ions of the indicator, potassium chromate, to form a red-brown precipitate of silver chromate.

$$2Ag^{+}_{(aq)} + CrO_4^{2-}{}_{(aq)} \rightarrow Ag_2CrO_{4(s)} \qquad \textbf{8.2}$$

This method can be used to determine the chloride ion concentration of water samples from many sources such as seawater, stream water, river water and estuary water. The pH of the sample solutions should be between 6.5 and 10. If the solutions are acidic, the gravimetric method or Volhard's method should be used.

***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 CI^- ions becomes exhausted, the silver ion concentration increases and a reddish brown precipitate of silver chromate is formed.

$2Ag^+ + CrO_4^{2-} = Ag_2CrO_4$ **(reddish brown precipitate)** **8.3**

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.
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.

Precautions:

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.
4. The chromate solution needs to be prepared and used with care as chromate is a known carcinogen.
5. Silver nitrate solution causes staining of skin and fabric (chemical burns). Any spills should be rinsed with water immediately.

Apparatus:

1. Burette
2. Measuring cylinder
3. Beaker
4. Dropper
5. Stirrer

Reagents:

1. Potassium chromate indicator
2. Silver nitrate solution (0.0141 N)

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" or "blank") x Multiplying Factor (M.F.)

Where, **M.F.** = $$\frac{\text{Normality of } AgNO_3 \text{ x equivalent wt.of } Cl^- \text{ x } 1000}{\text{mL of sample taken}}$$

Assignment

1. "Chlorides can introduce into natural waters in many ways" --explain.
2. Why it is necessary to dilute sample if end point does not obtained even after adding more than 7 or 8 mL silver nitrate to the original sample?
3. In determination of chloride, why an indicator "blank" or "error" is subtracted from the amount of silver nitrate used in titration? Explain.

DATA SHEET

Experiment Name :
Experiment Date :

Student's Name :
Student's ID :
Year/ Semester :
Section :
Group :

Table

Sample No and source	Temperature of Sample (°C)	Volume of Sample (mL)	Burette Reading (mL)		Volume of $AgNO_3$ solution (mL)	Chloride Concentration (mg/L)
			Initial	Final		

Signature of the course teacher

Experiment 9
Determination of Total Iron in Water

Introduction:

Iron, as well as manganese, creates serious problems in public water supplies. The problems are most critical for groundwater. 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.9.1.

$$FeCO_3 + CO_2 + H_2O = Fe^{2+} + 2HCO_3^- \qquad \textbf{9.1}$$

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. There seems to be enough evidence to suggest that development of reducing (anaerobic) condition is essential for appreciable amount of iron (as well as manganese) to gain entrance into water.

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 Mn4+ 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.

Guideline:

According to Bangladesh Environment Conservation Rules (1997), drinking water standard for iron is 0.3 - 1.0 mg/l.

Theory on Experimental Method:

Iron may be present in two forms, namely the reduced form (ferrous, Fe^2+) and the fully oxidized form (ferric, Fe^3+). 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.

$$5Fe^{2+} + MnO_4^- + 8H^+ = 5\,Fe^{3+} + Mn^{2+} + 4H_2O \qquad \textbf{9.2}$$

Ferric iron is determined by producing a red-colored iron compound, ferric thiocyanate, by the addition of potassium thiocyanate (Eq.-9.3).

$$Fe^{3+} + 3\ KCNS = \underset{(red)}{Fe(CNS)_3} + 3\ K^{+} \qquad \textbf{9.3}$$

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

Apparatus:

1. Nessler tube
2. Measuring cylinder
3. Dropper

Reagents:

1. Hydrochloric acid
2. Potassium permanganate solution
3. Potassium thiocyanate solution
4. Standard iron solution

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) =

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

DATA SHEET

Experiment Name :
Experiment Date :

Student's Name :
Student's ID :
Year/ Semester :
Section :
Group :

Table

Sample No	Source of Sample	Temperature of Sample (°C)	Total iron concentration (mg/l)

Signature of the course teacher

Experiment 10
Determination of Arsenic in Water

Introduction:

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. In Bangladesh, the arsenic in groundwater is of geologic origin and is probably only apparent now because it is only the last 20 - 30 years that groundwater has been extensively used for drinking in rural areas.

Weathering of arsenic-rich base metal sulphides in the upstream of the Ganges basin appears to be a major source of arsenic-rich iron oxyhydroxides in the sediments of Bangladesh. 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.

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 1.

Table 1: Arsenic compounds and species and their environmental and toxicological importance in water

Compounds	Example	Environmental Significance/ Dominant pH region	Toxicity
Arsine	As3.	Minor importance	Most toxic As
Elemental Arsenic	As	Minor importance	Least toxic As
Trivalent Arsenic	As(III)	Anaerobic	10 more As(V)
Arsenite, Inorganic	H_3As0_3 $H_2As0_3^{1-}$, $HAs0_3^{2-}$., $As0_3^{3-}$.	pH = 0-9 pH = 10-12 pH = 13 pH = 14	
Methylated As(III) Organo-As(III)		Minor importance	Less toxic than As(III)
Pentavalent arsenic	As(V)	Aerobic	10 times less As(III).
Arsenate, Inorganic	H_3As0_4, H_2As0^{1-}, $HAs0_4^{2-}$., $As0_4^{3-}$.	pH = 0-2 pH = 3-6 pH = 7-11 pH = 12-14	
Methylated As(V) Organo-As(V)		Minor importance	Less than As(V)

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. In groundwater environment where the conditions are mostly reducing, a

significant part of the arsenic exists as As(III).

Environmental significance:

Arsenic is a major **environmental** pollutant and exposure occurs through **environmental**, occupational and medicinal sources. Airborne exposure is small except in polluted locations. Food exposure can be significant but, particularly in fish and shellfish, it is mostly in organic forms that are relatively nontoxic. Drinking water remains the most significant source worldwide, and large numbers of people are subject to serious exposure from this source. Toxicity consists mostly of neuropathy, skin lesions, vascular damage, and carcinogenesis. Vascular lesions are the result of endarteritis (blackfoot disease). This appears to be more prevalent in developing rather than developed countries and may be related to nutritional deficiencies. Skin cancer is the most clearly associated malignancy related to arsenic exposure from drinking water; however, bladder, lung, liver, and kidney tumors also appear to be related. There is no particular remedial action for chronic **arsenic** poisoning. Low socioeconomic status and malnutrition may increase the risk of chronic toxicity.

Guideline:

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 10µg/Land the USEPA is also planning to revise its standard from50 µg/Lto10µg/L.

Analytical Methods for Measuring Arsenic:

The most commonly used method for detection of arsenic concentration water may be categorized as follows:

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 (SDOC) 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.

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

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.

Hydride generation-sera per-spectrophotometric (SDDC) method

Minimum detectable quantity for this method is 1 micro gram As. This method essentially involves: conversion (reduction) of all arsenic in water into As(III) and generation of arsine gas in the form of arsenic hydride (AsH3). Absorbance of red-coloured complex produced by passing of arsine gas through a solution of silver diethyl-dithiocarbamate (SODC) is

measured in a spectrophotometer at 535 nm wavelength.

Hydride generation-scraper-indicator paper-field kit

A simple and reasonably accurate method for arsenic measurement Similar to SOOC method all arsenic in water is converted to As(III) and generates arsine gas which is then passes through a filter paper soaked in mercuric bromide.

Laboratory Measurement of Arsenic by Arsenic Tool kit Method:

In the class arsenic measurement by arsenic tool kits will be conducted. The kit involves the generation of arsine (AsH_3) from inorganic arsenic species by reduction with Zn and HCl. The arsine then reacts with a test strip containing $HgBr_2$ to produce a color that is compared with a color scale for quantitation.

Apparatus:

1. Arsenic toolkits

Procedure:

1. Add reagent to Reaction Bottle and shake vigorously.
2. Insert the strip into the turret and close it. Let it sit 10 minutes.
3. Select the As concentration on the chart that matched the color of the test strip most closely. The reference chart provided with the kit displays the yellow to brown range of colors expected for As concentrations of 0, 10, 25, 50, 100, 200, 300, 500, and 1000 µg/L.

Assignment:

1. Discuss possible sources of arsenic in ground water.
2. Does the toxicity of arsenic contaminated groundwater depend on arsenic speciation?

DATA SHEET

Experiment Name :
Experiment Date :

Student's Name :
Student's ID :
Year/ Semester :
Section :
Group :

Table

Sample No	Source of Sample	Temperature of Sample (°C)	Arsenic concentration (µg/l)

Signature of the course teacher

Experiment 11
Determination of Biochemical oxygen demand

Introduction:

The biochemical oxygen demand determination is a chemical procedure for determining the amount of dissolved oxygen needed by aerobic organisms in a water body to break the organic materials present in the given water sample at certain temperature over a specific period of time.
BOD of water or polluted water is the amount of oxygen required for the biological decomposition of dissolved organic matter to occur under standard condition at a standardized time and temperature. Usually, the time is taken as 5 days and the temperature is 20°C.
The test measures the molecular oxygen utilized during a specified incubation period for the biochemical degradation of organic material (carbonaceous demand) and the oxygen used to oxidize inorganic material such as sulfides and ferrous ion. It also may measure the amount of oxygen used to oxidize reduced forms of nitrogen (nitrogenous demand).

Environmental significance:

BOD is the principle test to give an idea of the biodegradability of any sample and strength of the waste. Hence the amount of pollution can be easily measured by it. Efficiency of any treatment plant can be judged by considering influent BOD and the effluent BOD and so also the organic loading on the unit.

Application of the test to organic waste discharges allows calculation of the effect of the discharges on the oxygen resources of the receiving water. Data from BOD tests are used for the development of engineering criteria for the design of wastewater treatment plants. Ordinary domestic sewage may have a BOD of 200 mg/L. Any effluent to be discharged into natural bodies of water should have BOD less than 30 mg/L. This is important parameter to assess the pollution of surface waters and ground waters where contamination occurred due to disposal of domestic and industrial effluents. Drinking water usually has a BOD of less than 1 mg/L. But, when BOD value reaches 5 mg/L, the water is doubtful in purity. The determination of BOD is used in studies to measure the self-purification capacity of streams and serves regulatory authorities as a means of checking on the quality of effluents discharged to stream waters.
The determination of the BOD of wastes is useful in the design of treatment facilities. It is the only parameter, to give an idea of the biodegradability of any sample and self-purification capacity of rivers and streams. The BOD test is among the most important method in sanitary analysis to determine the polluting power, or strength of sewage, industrial wastes or polluted water. It serves as a measure of the amount of clean diluting water required for the successful disposal of sewage by dilution.

Guideline:

According to Bangladesh Environment Conservation Rules (1997), drinking water standard for biochemical oxygen demand (BOD) is 0.2 mg/L (at 20°C). For wastewater effluent allowable concentration of BOD varies from 50- 250 mg/L depending on discharge point of the effluent (e.g., inland water, irrigation land, public sewer etc.)

Principle:

The sample is filled in an airtight bottle and incubated at specific temperature for 5 days. The dissolved oxygen (DO) content of the sample is determined before and after five days of incubation at 20°C and the BOD is calculated from the difference between initial and final DO. The initial DO is determined shortly after the dilution is made; all oxygen uptake occurring after this measurement is included in the BOD measurement.

Since the oxygen demand of typical waste is sever hundred milligrams per liter, and since the saturated value of DO for water at 20^{u}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,

$$\mathbf{BOD_5} = [DO_i - DO_f] \times D.F. \qquad \mathbf{11.1}$$

Here,

$$\textbf{Dilution factor (D.F.)} = \frac{(\text{Volume of waste water} + \text{Volume of dilution water})}{\text{Volume of waste water}}$$

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 wader. The changes in DO in both are measured. The oxygen demand of waste water (BOD_w) is then determined from the following relationship:

$$\mathbf{BOD_m \times V_m} = BOD_w \times V_w + BOD_d \times V_d \qquad \mathbf{11.2}$$

Where, BOD_m, is the BOD of the mixture of wastewater and dilution water and BOD_d is the BOD of the dilution water alone; V_w and V_d are the volumes of wastewater and dilution water respectively in the mixture and $V_m = V_w + V_d$.

Sample handling and preservation:

Preservation of sample is not practical. Because biological activity will continue after a sample has been taken, changes may occur during handling and storage.

If Analysis is to be carried out within two hours of collection, cool storage is not necessary. If analysis cannot be started with in the two hours of sample collection to reduce the change in sample, keep all samples at 4° C.

Do not allow samples to freeze. Do not open sample bottle before analysis. Begin analysis within six hours of sample collection.

Apparatus:

1. BOD bottle
2. Beaker (250 ml)
3. Measuring cylinder
4. Dropper
5. Stirrer

Reagents:

1. Manganous sulfate solution
2. Alkaline potassium iodide solution
3. 0.025N sodium thiosulfate
4. Starch solution (indicator)
5. Concentrated sulfuric acid.

Procedure:

Fill two BOD bottles with sample (or diluted sample); the bottles should be completely filled. Determine initial DO (DO_i) in one bottle immediately after filling with sample (or diluted sample). Keep the other bottle in dark at 20°C and after particular days (usually 5-days) determine DO (DO_f) in the sample (or diluted sample). 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 Multiplying Factor (M.F.)

Calculate BOD of the sample according to Eq. – 11.1 or Eq. – 11.2.

Assignment:

1. In a BOD test on a diluted wastewater sample (1:20 dilution, but not seeded), the initial DO is 8.2 mg/L and final DO after 5 days is 3.2 mg/L. If the reaction rate constant is 0,2/day, calculate: (a) 5-day BOD of the wastewater, (b) Ultimate carbonaceous BOD of the wastewater, (c) Remaining Oxygen demand after 5-days.
2. 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 40 mL of wastewater and the rest with seeded dilution water experiences a drop of 7.1 mg/L in the same period (5-day), Calculate the BOD_5 of the wastewater.

DATA SHEET

Experiment Name :
Experiment Date :

Student's Name :
Student's ID :
Year/ Semester :
Section :
Group :

Table

Sample No	Source of Sample	Temperature of Sample (°C)	BOD (mg/L)

Signature of the course teacher

Experiment 12
Determination of Chemical oxygen demand

Introduction:

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.

Organic matter + Oxidizing agent = $CO_2 + H_2O$ 12.1

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 matter is 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 BOO. 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.

Guideline:

According to Bangladesh Environment Conservation Rules (1997), drinking water standard for chemical oxygen demand (COD) is 4.0 mg/L. For wastewater effluent allowable concentration of CBOD varies from 200- 400 mg/L depending on discharge point of the effluent (e.g., inland water, irrigation land, public sewer etc.)

Principle:

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.

Apparatus:

1. Beaker (250 mL)
2. Dropper
3. Stirrer

Reagent:

1. Diluted sulfuric acid solution
2. Standard potassium permanganate solution
3. Standard Ammonium Oxalate solution

Procedure:

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 $KMn0_4$ 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 $[(NH_4)_2C_20_4]$ 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 $[(NH_4)_2C_20_4]$ now remaining in the flask is just equivalent to the amount of the oxidizing agent ($KMn0_4$) 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.

Calculation:

$$\textbf{COD (mg/L)} = \frac{\text{mL of } KMnO_4 \text{ used in step 6} \times 100}{\text{mL of sample used}}$$

Assignment:

1. What are the principal advantages and disadvantages of the COD test over the BOD test?
2. Explain why COD values are greater than BOD values.

DATA SHEET

Experiment Name :
Experiment Date :

Student's Name :
Student's ID :
Year/ Semester :
Section :
Group :

Table

Sample No	Source of Sample	Temperature of Sample (°C)	COD (mg/L)

Signature of the course teacher

Experiment 13
Chemical Coagulation of water:
Alum Coagulation

Introduction:

Chemical coagulation is a treatment method widely used for removal of small sized and colloidal impurities from water. Surface water generally contains a wide variety of colloidal impurities that may cause the water to appear turbid and may impart color to the water. Colloidal particles that cause color and turbidity 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. In order to be removed, the individual colloids must aggregate and grow in size so that they can settle by gravity. Chemical agents are used to promote colloid aggregation by destroying the forces that stabilize colloidal particles.

The process of destroying the stabilizing forces and causing aggregation of colloids is referred to as chemical coagulation. Coagulation involves reduction of electrical forces of repulsion and promotion of "chemical type" interaction between colloids, which eventually results in settling of the colloids and accomplishes removal of turbidity and color. 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.

Many authors use the term "coagulation" to describe the process by which the charge on particles is destroyed, and the term "flocculation" to describe the aggregation of particles into larger units. 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.

The common metal salt alum (aluminum sulfate) is a good coagulant for water containing appreciable organic matter. The chemical formula used for commercial alum is $Al_2(SO_4)_3.14H_20$. Once dissolved in water, aluminum forms hydroxo-complexes and solids [e.g., $Al(OH)_3(s)$, $Al(OH)^{2+}$, $Al(OH)_2^+$, $Al(OH)_4^-$; and as a result pH of water is lowered, especially if alkalinity of water is low,. Theoretically, each mg/L of alum consume 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 polyelectrolytes are used to provide the necessary alkalinity. Iron coagulants 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.

$Al_2(SO_4)_3.14H_2O$ (alum) = 2 Al^{3+} + 3 $SO4^{2-}$ **13.1**

$Al^{3+} + 3H_2O = Al(OH)_3$ (s) + 3 H^+ **13.2**

$A1^{3+} + H_2O = Al(OH)^{2+} + H^+$ **13.3**

$Al^{3+} + H_3O = Al(OH)_2^+ + 2H^+$ **13.4**

$A1^{3+} + H_2O = Al(OH)_4^+ + H^+$ **13.5**

Environmental Significance:

Besides efficient removal of turbidity and color, coagulation with alum and ferric chloride or ferric sulfate 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 / sulfate is being successfully used for removal of arsenic from water.

Principle:

Treatment of water by coagulation involves -

(1) Determination of optimum dose of coagulant by jar test.
(2) Determination of power input for the flocculator.

In the class jar test to determine optimum coagulant dose will be carried out, it is important to determine the optimum dose to avoid charge reversal and resuspension colloids. Optimum coagulant dose is considered as the amount of coagulant which produces water with lowest turbidity.

Apparatus:

1. Coagulation (stirring) device
2. pH meter
3. Turbidity meter
4. Glass beakers (1000 mL)

Reagent:

1. Standard Alum solution.

Procedure:

1. Determine pH and turbidity of the water to be treated. You may be instructed to determine color and arsenic concentration of the water to be treated (if removal efficiencies of these parameters are to be determined).
2. Fill six 1000 mL beakers each with 500 mL water to be treated,
3. Add required (as instructed by teacher) coagulant (standard alum solution) to each beaker.
4. Mix the samples in the beaker with the help of the stirring device. Subject the samples to one minute of rapid mixing followed by 15 minutes of slow mixing (about 40 rpm).
5. Allow the flocs to settle down for about 15 minutes. Observe the characteristics of the flocs and the settling rates.
6. Collect the supernatant from each beaker and measure pH and turbidity of each. You may be instructed to measure color and arsenic concentration of the supernatant samples (if removal efficiencies of these parameters are to be determined).
7. Plot pH versus alum dose in a graph paper and observe effect of alum dose on pH.
8. Plot turbidity (NTU) versus the coagulant (alum) dose (mg/L) in a graph paper. Determine optimum dose of alum from this plot.

Assignment:

1. What is charge reversal? When and why it happens?
2. Why addition of alum may result in a drop in pH value. Discuss the effect of alum dose on pH from your experimental results.

DATA SHEET

Experiment Name :
Experiment Date :

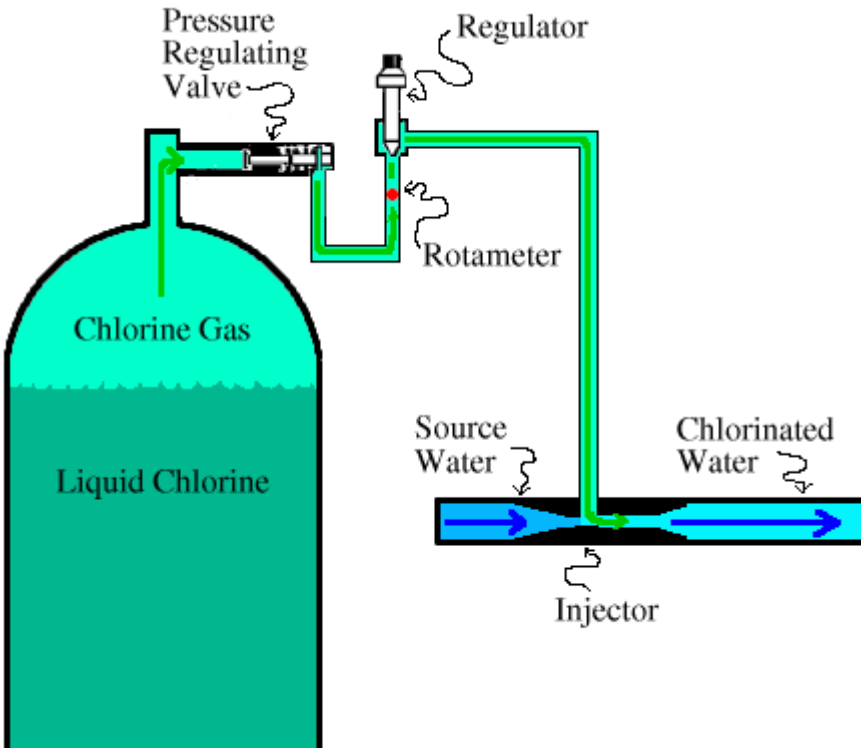
Student's Name :
Student's ID :
Year/ Semester :
Section :
Group :

Table

Observation No.	pH (initial)	Initial Turbidity (NTU)	Alum Dose (mg/L)	pH (final)	Final Turbidity (NTU)
1					
2					
3					
4					
5					
6					

Signature of the course teacher

Experiment 14
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 practiced. 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:

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 $Ca(OC1)_2$]. 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.

Chlorine gas rapidly hydrolyzes to hypochlorous acid according to:

$$Cl_2 + H_2O = HOCl + H^+ + Cl^- \quad \textbf{14.1}$$

Aqueous solutions of sodium or calcium hypochlorite hydrolyze too:

$$Ca(OCl)_2 + 2H_2O = Ca^{2+} + 2HOCl + 2OH^- \quad \textbf{14.2}$$

$$NaOCl + H_2O = Na^+ + HOCl + OH^- \quad \textbf{14.3}$$

Hypochlorous acid is a weak acid and will disassociate according to:

$$HOCl \Leftrightarrow H^+ + OCl^- \quad \textbf{14.4}$$

The two chemical species formed by chlorine in water, hypochlorous acid (HOCl) and hypochlorite ion (OCl^-), are commonly referred to as "free" or "available" chlorine.

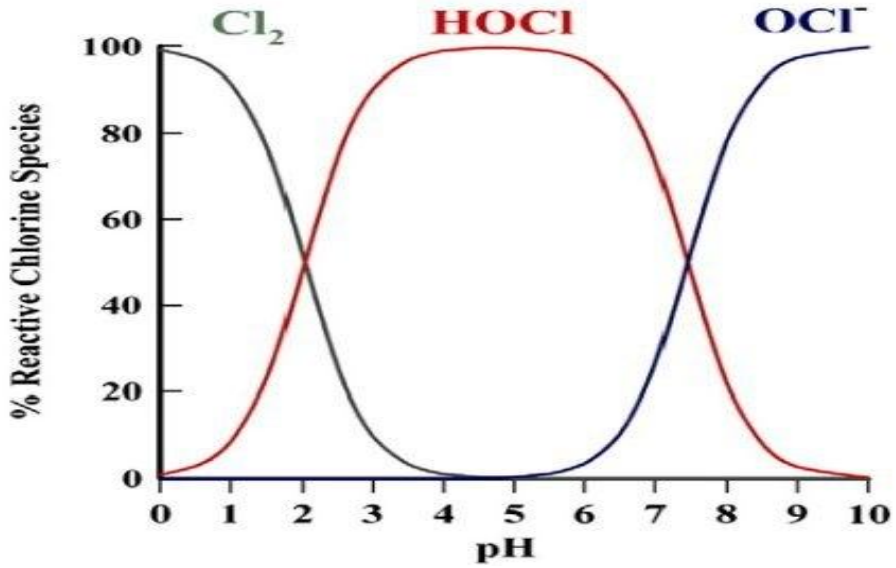


Figure 14.1: Distribution of Chlorine species at 25^0C

Figure 13.1 shows that Cl_2 can be significantly 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 decrease, while the corresponding fraction of OCl^- increases. In waters with pH between 6.5~8.5, the reaction is incomplete and both species (HOCl and OCl^-) will be present. Hypochlorous acid is the more germicidal of the two, especially at short contact time. 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.

Reactions of Chorine 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_2CI), dichloramine ($NHCl_2$) and trichloramine or nitrogen trichloride (NCI_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.

$NH_3 + HOCI = NH_2C1 + H_2O$; pH 4.5 to 8 **14.5**
$NH_2CI + HOCI = NHCl_2 + H_2O$; pH 4.5 to 8 **14.6**
$NHCl_2\ HOCI = NCI_3 + H_2O$; pH < 4.5 **14.7**

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

$3\ Cl_2 + 2\ NH_3 = N_2\ (g) + 6H^+ + 6\ CI^-$ **14.8**
$4C1_2 + NH_3 + 3H_2O = 8C1^- + NO_3^- + 9H^+$ **14.9**

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 organi$_c$ 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 mono-, di-, or trichlorophenols, which can impart tastes and odors to waters, Chlorine also reacts with humic substances present in water to form trihalomethanes (THMs, e.g., chloroform, brornoform, 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 following figure. The different segment of the curve is described as follows :

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" (see Fig.).
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.

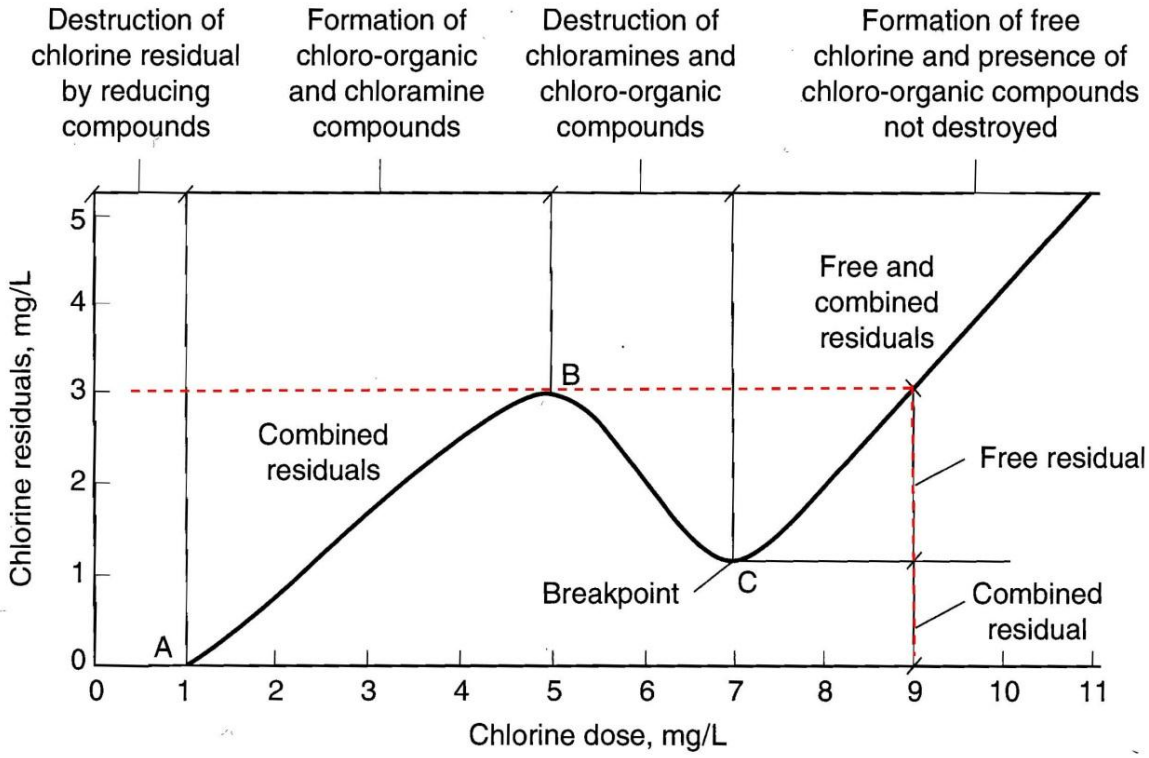


Figure 14.2: Generalized curve obtained during breakpoint chlorination of water sample containing ammonia

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., $C1_2$: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 figure (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^-. 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., $C1_2$:NH_3-N = 1.5:1]. This is represented by the portion of the curve from B to C.
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. Addition of chlorine beyond point C would produce free chlorine residuals and is referredto 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".*

Environmental Significance:

Breakpoint chlorination is required to obtain a free chlorine residual for better disinfection if ammonia is present in a water supply. While free chlorine residuals have good disinfecting powers, they are usually dissipated quickly in the distribution system. For this reason, final treatment with ammonia if often practiced to convert free chlorine residuals to longer-lasting combined chlorine residuals. 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'*.

Apparatus:

1. Erlemeyer flask (250 mL)
2. Bottle
3. Beaker (250 mL)
4. Measuring cylinder
5. Dropper
6. Stirrer

Reagents:

1. Starch Indicator
2. Standard 0.025 N Sodium thiosulfate
3. Potassium Iodine crystal
4. Concentrated Acetic Acid
5. 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 to stand for 30 minutes.
4. Determine residual chlorine of water from each flask by the starch-iodine method as described below:

Starch-Iodine 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 quantity of iodine 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, $I_2 + 2S_2O_3^{2-} = S_4O_6^{2-} + 2I^-$
I_2 + starch = 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 sulphate.

Procedure for determination of residual chlorine concentration:

1. Place 200 mL of the sample in an Erlenmeyer flask.
2. Add 'about 1g of potassium iodide (estimated on a spatula) and 2 mL of concentrated Acetic acid to the water.
3. Add 0.025 N sodium thiosulfate drop by drop from a burette until the yellow color almost disappears.
4. Add 1 mL of starch solution to the water.
5. Continue addition of standard sodium thiosulfate ($Na_2S_2O_3$) solution until the blue color just disappears.
6. Record the quantity (in mL) of sodium thiosulfate ($Na_2S_2O_3$) solution used.

Calculation:

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

$$\textbf{M.F.} = \frac{\text{Normality of } N_2S_2O_3 \text{ X Equivalent wt.of } Cl_2 \text{ X } 1000}{\text{mL of sample taken}}$$

Assignment:

1. What are the major disadvantages 'of chlorination? Name some of the alternate disinfectants.
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^-? What kind of change in pH would you propose in order to increase the relative proportion of HOCL, which is a better disinfectant?
3. Schematically draw “chlorine residual" versus "chlorine dose" curve for a water sample with no ammonia or organic matter.

DATA SHEET

Experiment Name :
Experiment Date :

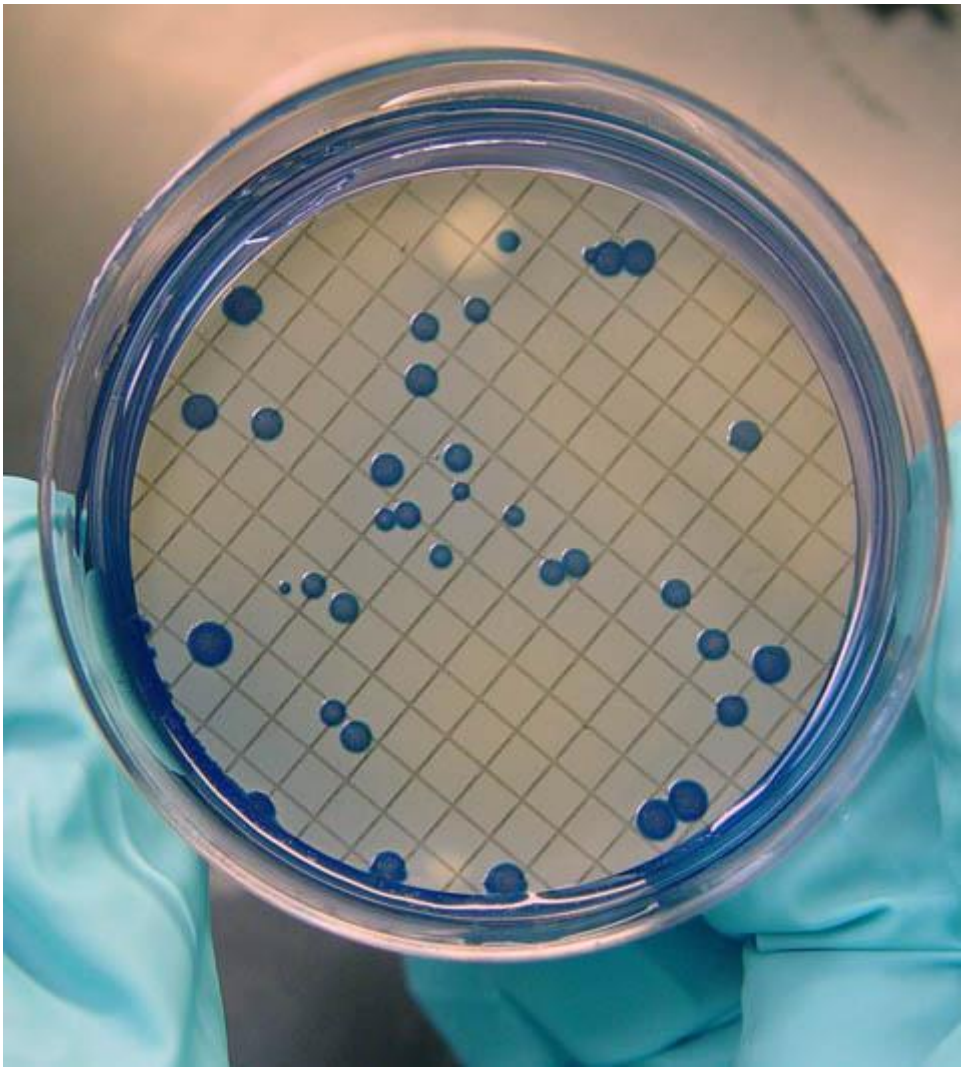
Student's Name :
Student's ID :
Year/ Semester :
Section :
Group :

Table

Observation No.	pH (initial)	Chlorine Dose (mg/L)	Residual Chlorine (mg/L)
1			
2			
3			
4			
5			
6			

Signature of the course teacher

Experiment 15
Determination of Total and Fecal Coliform in water



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. The ideal indicator organism 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 humans

No organisms or group of organisms meet all of these criteria; but the *coliform* bacteria fulfill most of them, and this group is most common indicator used in microbial examination of water. Total coliforms are grouped into two categories (*1) Fecal coliform* (thermo-tolerant coliform^{-}) and (*2) 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 coliform* are a subgroup of total coliforms, which live in the warm blooded animals and have the same properties as the*totalcoliform* but tolerate and grow at 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 arc 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 coliforms as an indicator may therefore be of little value in assessing the fecal contamination of surface water, unprotected shallow wells etc. where contamination by coliforms of non-fecal origin can occur. The measurement of total coliforms is of particular relevance for treated and / or chlorinated water supplies; in this case the absence of total coliforms would normally indicate that the water has been sufficiently

treated / disinfected to destroy various pathogens. Measurement of focal coliforms is a better indicator of general contamination by material of fecal origin. The predominant species of fecal coliform group is *Escherichia coil*(E. coil), which is exclusively of fecal origin, but strains of *Klebsella pneumonia* 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, which can biochemically mimic the coliform group
- False negative result can be obtained when conforms 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 coliforms 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.

Apparatus:

1. Petri Dish
2. Incubator
3. Measuring Cylinder, beaker, dropper etc.

Reagents:

1. Appropriate culture medium (broth)
2. Distilled water

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.

(a) Multiple Tube/ Most Probable Number (MPN) method:

MPN is a procedure to estimate the population density of viable microorganisms in a test sample. It's based upon the application of the theory of probability to the numbers of observed positive growth responses to a standard dilution series of sample inoculums placed into a set number of culture media tubes. Positive growth response after incubation may be indicated by such observations as gas production in fermentation tubes or visible turbidity in broth tubes, depending upon the type of media employed.

(b) Membrane Filter Method:

In contrast to the multiple-tube (MT) method, the membrane filter (IVIF) 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 0.45 µm; 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 determination of coliforms:
- Waters containing particular toxic substances which may be absorbed by the membranes, can affect the growth of the coliforms.

Test Procedure (For MF method):

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.
3. 'With a sterile pipette add 2 mL of selective broth (culture) medium to saturate the pad.
4. Assemble the filtration unit by placing sterile membrane filter on the porous support, using forceps sterilized earlier by flaming.
5. 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.
6. Pour tide 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.
7. 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.
8. 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.
9. Invert the Petri-dish for incubation.
10. 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:

$$\textbf{Total coliform (CFU/ 100 mL)} = \frac{\text{No.of coliform colonies counted x 100}}{\text{mL of sample filtered}}$$

$$\textbf{Fecal coliform (CFU/ 100 mL)} = \frac{\text{No.of coliform colonies counted x 100}}{\text{mL of sample filtered}}$$

Assignment:

1. What do you understand by "indicator organisms"? Why water samples are usually tested for indicator organisms instead of specific pathogenic organisms?
2. Define and differentiate between total coliform (TC) and fecal coliform (FC)?
3. What are the major advantages of "membrane filtration method" over "multiple tube method"?

DATA SHEET

Experiment Name :
Experiment Date :

Student's Name :
Student's ID :
Year/ Semester :
Section :
Group :

Table

Observation No.	Total Coliform Unit per 100 ml	Fecal Coliform Unit per 100 ml
1		
2		
3		
4		
5		
6		

Signature of the course teacher

Sampling and Laboratory Analysis of Air

Ambient Air Sampling Guideline:

It includes-

- Survey for preliminary information
- Selection of sampling procedure
- Sampling locations
- Period of sampling, frequency and duration

Survey for preliminary information:

During ambient air pollutants sampling, it is also necessary to collect information on qualitative and quantitative data on the local sources of air pollution, topography, population distribution, land use pattern, climatology, etc, depending upon the objectives of the survey or measurement campaign. For example, an area map to locate pollution sources and monitoring locations, sources of pollution situated at far distances, etc. and other relevant data that describe the behaviour of atmosphere for a specific pollutant to be sampled may also be required. It includes-

a) Selection of sampling procedures including procedures of analysis of samples
b) Sampling locations
c) Period of sampling, frequency of sampling and duration
d) Auxiliary measurements (including meteorological parameters)
e) Processing of data

Selection of sampling procedure:

There are two types of sampling – continuous and time averaged in–situ samplings. Continuous sampling is carried out by automatic sensors, optical or electrochemical, and spectroscopic methods which produce continuous records of concentration values. The specific time-averaged concentration data can then be obtained from continuous records. Time-averaged data can also be obtained by sampling for a short time – i.e. by sampling a known volume of air for the required averaging time. Samples are then analyzed by established physical, chemical, and biological methods for the concentration values which are the effective average over the period of sampling.

Sampling locations:

Sampling locations are in general governed by factors like objectives, method of sampling and resources available. If the objective is to study health hazards and material damages, then locations should be kept close to the objects where the effects are being studied and should be kept at breathing level in the population centres, hospitals, schools, etc. For vegetation, it should be at foliage level. For background concentration, sampling location should be away from the sources of pollution. It can also be done by gridding the entire area to get statistically recommended values. The number of locations however depends upon the variability of concentration over the area under survey. A spot checking may be done to decide the location besides considering practical factors.

Period of sampling, frequency and duration:

Period, frequency and duration of sampling should be appropriate to the objectives of the study. It should be such that the measurable quantities are trapped in the sample at the end of the sampling. It is preferable to observe sampling period consistent with the averaging times for which air quality standards of the given pollutants are specified.

Ambient air sampling and analysis:

The pollutants for which sampling and analytical techniques discussed are SO_2, NO_X, O_3, CO and Particulate matters.

Sulphur dioxide (SO_2):

Title:
Sampling of sulphur dioxide in ambient air and the determination of its concentration

Method of measurement:
Volumetric - sampling a volume of air through a collecting medium at a known flow rate for a specified time.

Instrument:
Gas sampler of midget impingers or high volume sampler with a gas kit attachment for $S0_2$.

Auxiliaries:
Standard glass bubblers, airflow rotameter for measuring flow rate

Chemicals:
0.1 N Sodium tetra-chloromercurate, an absorbing reagent.

Sampling location guidelines:
Sampling station should be located depending upon the objective of measurement campaign and be kept at an altitude depending upon the type of study region (roadways, industrial area, disposal sites, residential tract, etc). Generally it is kept at a height of about 3 to 10 m from the ground level and sufficiently away from the disturbance or direct obstacle from the source under consideration.

Sampling frequency guidelines:
Sampling is carried out for various purposes. The regular monitoring campaign of national ambient air quality includes measurement of $S0_2$ typically for 24 hours at least twice a week making about 104 samples a year.

Steps for sampling:

- Prepare absorbing reagent (sodium tetra-chloromercurate) by dissolving 27.2 g mercuric chloride and 11.7 g sodium chloride in 1 lit of water
- Prepare a sampling train of at least 2 gas bubblers (for average reading) properly washed with distilled water and air dried.
- Place bubblers in the sampling system securely connecting to the manifold. Check the connections of bubblers with the manifold and the inlet and outlet
- Fill the bubblers with an absorbing reagent with an amount sufficient to last for 24 hours (approximately 15 ml for 8 hours sampling to 50 ml for 24 hours sampling)
- To eliminate interferences of trace metals, if any, 1 drop of 0.01% EDTA solution may be added to the reagent prior to sampling; similarly, effect of oxides of nitrogen may also be eliminated by adding 1 ml of 0.06% sulphamic acid to the reagent at site
- Start the sampler and adjust flow rate to 2 lit/min.
- Note the flow rate at the end of the desired sampling period and stop the sampler
- Transit the sampling train to environmental laboratory carefully with scientific precautions and preserve the sample tubes in a controlled environmental conditions.

Laboratory analysis:
Method:
Colorimetric - by estimating absorbance of $S0_2$ from the exposed absorbing reagent at 540 nm using spectrophotometer

Chemicals:

- Pararosaniline hydrochloride
- Acid bleached
- Formaldehyde (0.2%)
- Sulphamic acid by dissolving 0.8 g in 100 ml distilled water
- Standard sulphur dioxide solution by dissolving *004*g sodium metabisulphite in 250 ml distilled water

Steps of analysis:

Calibration curve:

- Prepare a standard solution of $S0_2$ concentrations ranging from 0 to 25 micro gram $S0_2$ by taking definite amounts of std. sulphur dioxide solution in a 25 ml volumetric flasks
- Add 14 ml of absorbing reagent and 1 ml of pararosaniline hydrochloride to each of the flasks making a total volume of 20 ml.
- Measure absorbance for each flasks by spectrophotometer at 540 nm wavelength
- Plot graph of absorbance vi concentration

Absorbance in *samples:*

- Transfer samples to a 25 ml flask and develop colour as done in calibration curve
- Measure absorbance at 540 nm
- Find out the concentration (micro gram $S0_2$) corresponding to the measured absorbance from the calibration curve

Calculations:

1. Average flow rate (if there is a significant difference in initial and final flow rates)
2. Total volume of air sampled (TVA) in m^3 = Avg. flow rate (lit)" *10-3* (me/lit) * time (hr) * 60 (rnin/hr)
3. Micro gram $S0_2$ /TVA

Oxide of Nitrogen:

Title:

Sampling of oxides of nitrogen In ambient air and the determination of its concentration

Method of measurement:

Volumetric - sampling a volume of air through a collecting medium at a known flow rate for a specified time.

Instrument:

Gas sampler of midget impingers or high volume sampler with a gas kit attachment for NO_x

Auxiliaries:

Standard glass bubblers, airflow rotameter for measuring flow rate

Chemicals:

Solution of sodium hydroxide and sodium arsenite, an absorbing reagent

Sampling location guidelines:

Sampling station should be located depending upon the objective of measurement campaign and be kept at an altitude depending upon the type of study region (roadways, industrial area, disposal sites, residential tract, etc). Generally it is kept at a height of about 3 to 10 m from the ground level and sufficiently away from the disturbance or direct obstacle from the source under consideration.

Sampling frequency guidelines:

Sampling is carried out for various purposes. The regular monitoring campaign of national ambient air quality includes measurement of NO_x typically for 24 hours at least twice a week making about 104 samples a year.

Steps for sampling:

- Prepare absorbing reagent (a solution of sodium hydroxide and arsenite) by dissolving 4 g sodium hydroxide and 1 g of sodium arsenite in 1 lit of distilled water
- Prepare a sampling train of at least 2 gas bubblers (for average reading) properly washed with distilled water and air dried.
- Place bubblers in the sampling system securely connecting to the manifold. Check the connections of bubblers with the manifold and the inlet and outlet
- Fill the bubblers with an absorbing reagent with an amount sufficient to last for 24 hours (approximately 15 ml for 8 hours sampling to 50 ml for 24 hours sampling)
- To eliminate interferences of sulphur dioxide, drop of hydrogen peroxide may be added to the reagent to convert sulphur dioxide into sulphate during analysis
- Start the sampler and adjust flow rate to about 0.2 lit/rnin for 24 hours sampling
- Note the flow rate at the end of the desired sampling period and stop the sampler
- Transit the sampling train to environmental laboratory carefully with scientific precautions and preserve the sample tubes in a controlled environmental conditions

Laboratory analysis:

Method:

Colorimetric - by reacting the nitrite ions with phosphorus acid, sulphanilamide, and NEDA solution by measuring absorbance of NO_x from the exposed absorbing reagent at 540 nm using spectrophotometer.

Chemicals:

- Hydrogen peroxide solution
- N-(I-Napthyl)-ethylenediamine Di-hydrochloride (NEDA)
- Sodium nitrite - assay of $NaN0_2$ (=> 97 %)
- Sodium nitrite stock solution (1000 µg N02/ml)
- Sodium nitrite solution (10 µg $N0_2$/ml)
- Sodium nitrite solution (1 µg $N0_2$/ml)
- Sulphanilamide solution
- Phosphoric acid

Steps of analysis:

Calibration curve:

- Prepare a standard solution of NOx concentrations ranging from 0 to 25 µg $S0_2$ by taking definite amounts of std. sulphur dioxide solution in a 25 ml volumetric flasks
- Add 14 ml of absorbing reagent and 1 ml of pararosaniline hydrochloride to each of the flasks making a total volume of 20 ml.
- Measure absorbance for each flasks by spectrophotometer at 540 nm wavelength
- Plot graph of absorbance via concentration

Absorbance in *exposed samples:*

- Transfer samples to a 25 ml flask and develop colour as done in calibration curve
- Measure absorbance at 540 nm

Calculations:

1. Average flow rate (if there is a significant difference in initial and final flow rates)
   Average flow rate (initial and final flow rates) in *lit/min.* = (Initial flow rate + final flow rate/2
2. Total volume of air sampled (TVA) in m^3 = = Avg. flow rate (*lit/min)** 10-^{3}*(m^3/lit)* * sampling time *(hr)* * 60 *(min/hr)*

Particulate matter:

Title:

Sampling of suspended particulate matter in ambient air and the determination of its concentration

Method of measurement:

Volumetric-filtration - sampling a volume of air through a filter medium at a known flow rate for a specified time.

Instrument:

High volume sampler with SPM filter manifold

Auxiliaries:

High volume sampler with airflow manometer for measuring flow rate, microfiber filter (8" x 10" size)

Sampling location guidelines:

Sampling station should be located depending upon the objective of measurement campaign and be kept at an altitude depending upon the type of study region (roadways, industrial area, disposal sites, residential tract, etc). Generally it is kept at a height of about 3 to 10 m from the ground level and sufficiently away from the disturbance

Sampling frequency guidelines:

Sampling is carried out for various purposes. The regular monitoring campaign of national ambient air quality includes measurement of SPM typically for 24 hours at least twice a week making about 104 samples a year.

Steps for sampling:

- Condition a filter paper in oven
- Prepare a sampling assembly by uncorking screws of the bracket.
- Take the tare (initial) weight of filter paper *(Wi, mg)*
- Place the filter in the sampling system securely and tighten the screws of the bracket.
- Set the timer for the period of sampling
- Start the sampler and adjust flow rate to about 2 lit/rnin for 24 hours sampling.
- Note the flow rate at the end of the desired sampling period and stop the sampler
- Transit the sampling train to environmental laboratory carefully with scientific precautions
- Condition the filter paper again for the same period as was done prior to sampling

Laboratory analysis:

Method:

Gravimetric - by weighing the mass of particles

Steps of analysis:

Weighing of exposed samples:

- Take final weight of the exposed filter with a standard balance *(WI, mg)*

Calculations:

1. Average flow rate (initial and final flow rates) in *lit/min.* = (Initial flow rate + final flow rate/2
2. Total volume of air sampled (TVA) in m^3 = Avg. flow rate *(lit/min)** 10-^3*(m^3/lit)* * sampling time *(hr)* * 60 *(min/hr)*
3. Concentration of SPM in $\mu g/m^3$ = $(w_f - w_i)$ (mg)/ TVA (m^3)*10^6 g/m^3

Sampling and Laboratory Analysis of Solid Waste

Guidance for carrying out waste sampling and analysis

Depending on the needs of the client analysis of waste may have different objectives. For instance, waste producers want to know what kind of recovery/disposal is possible, managers of waste treatment plants need to know if they can accept and will be able to treat the waste and authorities are interested in the environmental effects related to a particular waste. The different needs of the concerned actors lead to different testing programmes in which sample taking is required. The strategy of sampling and analysis has to be planned in advance very carefully in order to avoid useless efforts and unnecessary costs. The correct procedure of sampling is very important to get a representative sample of the specific waste subject to testing. A representative sample of a specific waste is important to ensure the reliability of analysis results obtained, which is the decision basis for the subsequent choice of waste management operations and handling.

Common properties for evaluation of Solid Waste

A. Physical properties
B. Chemical properties

A. Physical properties of solid waste

This includes the determination of percent contents of various ingredients of the solid waste.

1. Specific Weight (Density)

Specific weight is defined as the weight of a material per unit volume (e.g. kg/m^3, lb/ft^3) . Usually it refers to uncompacted waste.

1. Moisture Content

The moisture *(water content can change chemical and physical properties)* in a sample is expressed as percentage of the wet weight of the MSW material. The wet-weight method is most commonly used in the field of solid waste management.

2. Field Capacity

The total amount of moisture that can be retained in a waste sample subjected to the downward pull of gravity *(under free drainage conditions).* It is expressed as the fraction of water retained by a waste sample based on the dry weight of the sample.

B. Chemical Characteristics of Solid waste

Chemical properties of MSW are very important in evaluating the alternative treatment, processing and recovery options. Used primarily for combustion and waste to energy (WTE) calculations but can also be used to estimate biological and chemical behaviors.

1. Proximate Analysis

This analysis includes for **the combustible components** of MSW includes the following tests:

i. Loss of moisture: after heated at105^0C temperature for 1 hr
ii. Volatile Combustible Matter (VCM) :
    - the components, which are liberated at high temp *(except moisture)* in absence of air.
    - additional loss of weight of dry sample after ignition at temp 950^0C for 7 min in closed crucible (*in absence of oxygen*).
iii. Fixed Carbon
    - combustible residue after the expulsion of VCM.
    - higher the % of fixed carbon, greater it is calorific value.
iv. Ash: residue after combustion at temp 950^0 C in open crucible.

2. Fusing Point of Ash

Fusing point of ash is the temperature at which the ash resulting from the burning of waste will form a solid (clinker) by fusion and agglomeration.

3. Ultimate Analysis

Molecular composition (C, H, N, O, P, etc.) to characterize the chemical composition of the organic matter in MSW.

4. Energy Content

- Energy content can be determined by
  - by using a full scale boiler as calorimeter
  - by using a laboratory bomb calorimeter
  - by calculations
- Can be estimated by modified Dulong formula also.
  Energy Content (KJ/Kg) =338.2 C +1430 (H-O/8) +95.4 S

Appendix 1
The Environment Conservation Rules, 1997

(A) Standards for inland surface water

Best Practice based classification	Parameter			
	pH	BOD mg/l	DO mg/l	Total Coliform number/100
a. Source of drinking water for supply only after disinfecting:	6.5-8.5	2 or less	6 or above	50 or less
b. Water usable for recreational activity :	6.5 – 8.5	3 or less	5 of more	200 or less
c. Source of drinking water for supply after conventional treatment :	6.5 – 8.5	6 of less	6 or more	5000 or less
d. Water usable by fisheries:	6.5 – 8.5	6 of less	5 or more	---
e. Water usable by various process and cooling industries :	6.5 – 8.5	10 or less	5 or more	5000 or less
f. Water usable for irrigation:	6.5 – 8.5	10 or less	5 or more	1000 or less

Notes:

1. In water used for pisiculture, maximum limit of presence of ammonia as Nitrogen is 1.2 mg/l.
2. Electrical conductivity for irrigation water – 2250 µmhoms/cm (at a temperature of 25°C); Sodium less than 26%; boron less than 0.2%.

(B) Standards for drinking water

Sl. No.	Parameter	Unit	Standards
1	2	3	4
1.	Aluminum	mg/l	0.2
2.	Ammonia (NH_3)	,,	0.5
3.	Arsenic	,,	0.05
4.	Balium	,,	0.01
5.	Benzene	,,	0.01

1	2	3	4
6.	BOD_5 20°C	,,	0.2
7.	Boron	,,	1.0
8.	Cadmium	,,	0.005
9.	Calcium	,,	75
10.	Chloride	,,	150 – 600*
11.	Chlorinated alkanes		
	carbontetrachloride	,,	0.01
	1.1 dichloroethylene	,,	0.001
	1.2 dichloroethylene	,,	0.03
	tetrachloroethylene	,,	0.03
	trichloroethylene	,,	0.09
12.	Chlorinated phenols		
	- pentachlorophenol	mg/l	0.03
	- 2.4.6 trichlorophenol	,,	0.03
13.	Chlorine (residual)	,,	0.2
14.	Chloroform	,,	0.09
15.	Chromium (hexavalent)	,,	0.05
16.	Chromium (total)	,,	0.05
17.	COD	,,	4
18.	Coliform (fecal)	n/100 ml	0
19.	Coliform (total)	n/100 ml	0
20.	Color	Hazen unit	15
21.	Copper	mg/l	1
22.	Cyanide	,,	0.1
23.	Detergents	,,	0.2
24.	DO	,,	6
25.	Fluoride	,,	1
26.	Hardness (as $CaCO_3$)	,,	200 – 500
27.	Iron	,,	0.3 – 1.0
28.	Kjeldhl Nitrogen (total)	,,	1
29.	Lead	,,	0.05

1	2	3	4
30.	Magnesium	,,	30 – 35
31.	Manganese	,,	0.1
32.	Mercury	,,	0.001
33.	Nickel	,,	0.1
34.	Nitrate	,,	10
35.	Nitrite	,,	<1
36.	Odor	,,	Odorless
37.	Oil and grease	,,	0.01
38.	pH	,,	6.5 – 8.5
39.	Phenolic compounds	,,	0.002
40.	Phosphate	,,	6
41.	Phosphorus	,,	0
42.	Potassium	,,	12
43.	Radioactive materials (gross alpha activity)	Bq/l	0.01
44.	Radioactive materials (gross beta activity)	Bq/l	0.1
45.	Selenium	mg/l	0.01
46.	Silver	,,	0.02
47.	Sodium	,,	200
48.	Suspended particulate matters	,,	10
49.	Sufide	,,	0
50.	Sulfate	,,	400
51.	Total dissolved solids	,,	1000
52.	Temperature	°C	20-30
53.	Tin	mg/l	2
54.	Turbidity	JTU	10
55.	Zinc	mg/l	5

SCHEDULE – 2

Standards for Air

[See Rule 12]

Density in microgram per cusec meter

Sl. No.	Categories of Area	Suspended Particulate Maters (SPM)	Sulphur-dioxide	Carbon Monoxide	Oxides Nitrogen
a.	Industrial and mixed	500	120	5000	100
b.	Commercial and mixed	400	100	5000	100
c.	Residential and rural	200	80	2000	80
d.	Sensitive	100	30	1000	30

Notes:

1. At national level, sensitive area includes monuments, health center, hospital, archeological site, educational institution, and government designated areas (if any).
2. Industrial units located in areas not designated as industrial areas shall not discharge pollutants which may contribute to exceeding the standard for air surrounding the areas specified at Sl. nos. c and d above.
3. Suspended Particulate Matter means airborne particles of adiameter of 10 micron or less.

SCHEDULE – 9

Standards for Sewage Discharge

[See Rule 12]

Parameter	Unit	Standard Limit
BOD	miligram/l	40
Nitrate	,,	250
Phosphate	,,	35
Suspended Solids (SS)	,,	100
Temperature	Degree Centigrade	30
Coliform	number per 100 ml	1000

Notes:

1. This limit shall be applicable to discharges into surface and inland waters bodies.
2. Sewage shall be chlorinated before final discharge.

SCHEDULE – 10

Standards for Waste From Industrial Units or Projects Waste

[See Rule 13]

Sl. No.	Parameter	Unit	Places for determination of standards		
			Inland Surface Water	Public Sewerage system connected to treatment at second stage	Irrigated Land
1	2	3	4	5	6
1	Ammonical Nitrogen (as elementary N)	mg/l	50	75	75
2	Ammonia (as free ammonia)	,,	5	5	15
3	Arsenic (as)	,,	0.2	0.05	0.2
4	BOD_5 at 20^oC	,,	50	250	100
5	Boron	,,	2	2	2

1	2	3	4	5	6
6	Cadmium (as CD)	,,	0.50	0.05	0.05
7	Chloride	,,	600	600	600
8	Chromium (as total Cr)	,,	0.5	1.0	1.0
9	COD	,,	200	400	400
10	Chromium (as hexavalent Cr)	,,	0.1	1.0	1.0
11	Copper (as Cu)	,,	0.5	3.0	3.0
12	Dissolved Oxygen (DO)	,,	4.5 – 8	4.5 – 8	4.5 – 8
13	Electro-conductivity (EC)	micro mho/ cm	1200	1200	1200
14	Total Dissolved Solids	,,	2,100	2,100	2,100
15	Fluoride (as F)	,,	2	15	10
16	Sulfide (as S)	,,	1	2	2
17	Iran (as Fe)	,,	2	2	2
18	Total Kjeldahl Nitrogen (as N)	,,	100	100	100
19	Lead (as Pb)	,,	0.1	1.0	0.1
20	Manganese (as Mn)	,,	5	5	5
21	Mercury (as Hg)	,,	0.01	0.01	0.01
22	Nickel (as Ni)	,,	1.0	2.0	1.0
23	Nitrate (as elementary N)	mg/l	10.0	Not yet Fixed	10
24	Oil and Grease	,,	10	20	10
25	Phenolic Compounds (as C_6H_5OH)	,,	1.0	5	1
26	Dissolved Phosphorus (as P)	,,	8	8	15
27	Radioactive substance	To be specified by Bangladesh Atomic Energy Commission			
28	pH		6 – 9	6 – 9	6 – 9
29	Selenium (as Se)	mg/l	0.05	0.05	0.05
30	Zinc (as Zn)	Degree	5	10	10

1	2	3	4	5	6
31	Total Dissolved Solids	,,	2,100	2,100	2,100
32	Temperature	Centig rade	40	40	40-Summer
			45	45	45-Winter
33	Suspended Solids (SS)	mg/l	150	500	200
34	Cyanide (as Cn)	,,	0.1	2.0	0.2

Notes:

1. These standards shall be applicable to all industries or projects other than those specified under the heading "Standards for sector wise industrial effluent or emission."
2. Compliance with these standards shall be ensured from the moment an industrial unit starts trial production, and in other cases, from the project starts operation.
3. These standards shall be inviolable even in case of any sample collected instantly at any point of time. These standards may be enforced in a more stringent manner if considered necessary in view of the environmental conditions of a particular situation.
4. Inland Surface Water means drains/ponds/tanks/water bodies/ditches, canals, rivers, springs and estuaries.
5. Public sewerage system means treatment facilities of the first and second stage and also the combined and complete treatment facilities.
6. Irrigable land means such land area which is sufficiently irrigated by waste water taking into consideration the quantity and quality of such water for cultivation of selected crops on that land.
7. Inland Surface Water Standards shall apply to any discharge to a public sewerage system or to land if the discharge does not meet the requirements of the definitions in notes 5 and 6 above.

Appendix 2
Lab Report Format

1. All students must have a same colored printed **cover page**. The design of cover page is provided with the lab manual. Students have to compose only the course teacher's name and designation ant their information.

2. An **index** is provided. It should be printed and set after the cover page. Table may be fill up by pen during each submission after test.

3. Each report must have a common printed **top page**. Only the experiment name and no. and the date may be filled up by pen. A top page design is provided.

4. **A4 papers** have to be used for preparing the lab report. Writing should be done with **pen**. Pencil may be used for any kind of sketch.

5. In each experiment of the lab report the following points must have to be present: **Objective, Equipment, Procedure, Data Table (signed), Sample Calculation, Result** and **Discussion**.

CE 332
Environmental Engineering- Lab I
(Lab Manual)

Prepared For
Name of Course Teacher
Designation of Course Teacher
&
Name of Course Teacher
Designation of Course Teacher

Prepared By
Name of Student
Student's ID
Year/ Semester
Group

INDEX

Sl. No.	Name of The Experiment/ Assignment	Date of Performance	Date of Submission	Signature	Comments	Page no.

CE 332
Environmental Engineering- Lab I
(Lab Report)

Experiment / Assignment No. :
Experiment/Assignmrnt Name:

Date of Performance:
Date of Submission:

Prepared For
Name of Course Teacher
Designation of Course Teacher
&
Name of Course Teacher
Designation of Course Teacher

Prepared By
Name of Student
Student's ID
Year/ Semester
Group

Appendix 3
Lab Instructions

1. All students must have to be present at laboratory just in time.
2. All students must have to submit the lab report just after the entrance and before the class start.
3. Lab reports have to be submitted serially according to Student's ID.
4. Students have to complete full report in class and take sign from the course teacher. (In some experiment which require more times, data sheet and discussion should be completed as possible in class time.)
5. Students should be very careful about any test. They should conduct the tests by taking maximum care of the equipment during test.

References

1. “Standard Methods for the Examination of Water and Waste Water”, American Public Health Association, APHA, 20th Edition, 1995.
2. “The Environment Conservation Rules", 1997.
3. “The Environmental Conservation Act”, 1995.