JP College of Engineering

Run by DMI Sisters

Affiliated to Anna University, Chennai & Approved by AICTE, New Delhi,
ISO 9001:2015 Certified

Ayikudy-627852, Tenkasi (Dist), Tamil Nadu, India.

Department of Civil Engineering

Academic Year 2022-2023

Odd Semester

CE3311 Water and Wastewater Analysis Laboratory

Lab Manual

Regulation 2021

Mr.G.Baskar Singh

Department of Civil Engineering

Department Vision & Mission Statement

Vision:

To build young Technocrats by imparting their technical knowledge in the field of Civil Engineering, by laying the foundation for future engineers, who can meet the demands of industry and community effectively in all part of civil works and to make significant contribution in the economic development of the state, region and nation.

Mission:

M1: To adopt valuable teaching methods and implement high quality education to maximize Engineering knowledge for students.

M2: To promote innovative and original thoughts in the minds of civil engineers.

M3: To provide facilities to the students and faculty members to enhance the understanding and implementation of recent trends in the Civil Engineering field.

M4: To produce Civil Engineering graduates with good ethical skills and managerial skills to become as successful professionals and entrepreneurs.

M5: To promote advanced technology, Industry Institute interaction, research and consultancy in Civil Engineering department with global linkages.

	LIST OF EXPERIMENTS
1.	STUDY OF SAMPLING AND PRESERVATION METHODS ANTI SIGNIFICANCE OF CHARACTERISATION OF WATER AND WASTE WATER
2.	EXPERIMENT ON DETERMINATION OF pH
3.	EXPERIMENT ON DETERMINATION OF TURBIDITY
4.	EXPERIMENT ON DETERMINATION OF TOTAL HARDNESS
5.	EXPERIMENT ON DETERMINATION OF CALCIUM HARDNESS
6.	EXPERIMENT ON DETERMINATION OF RESIDUAL CHLORINE
7.	EXPERIMENT ON DETERMINATION OF ALKALINITY OF WATER
8.	EXPERIMENT ON DETERMINATION OF CHLORIDES
9.	EXPERIMENT ON DETERMINATION OF SULPHATES
10.	EXPERIMENT ON DETERMINATION OF OPTIMUM COAGULANT DOSAGE
11.	EXPERIMENT ON DETERMINATION OF DISSOLVED OXYGEN
12.	EXPERIMENT ON DETERMINATION OF TOTAL ORGANIC AND INORGANIC SOLIDS IN WATER
13.	EXPERIMENT ON DETERMINATION OF TOTAL DISSOLVED AND SUSPENDED SOLIDS IN WATER
14.	EXPERIMENT ON DETERMINATION OF CHEMICAL OXYGEN DEMAND
15.	EXPERIMENT ON DETERMINATION OF FLUORIDE USING ION SELECTIVE ELECTRODE
16.	DETERMINATION OF AMMONIACAL NITROGEN
17.	EXPERIMENT ON DETERMINATION OF BIOCHEMICAL OXYGEN DEMAND

STUDY OF SAMPLING AND PRESERVATION METHODS ANTI SIGNIFICANCE OF CHARACTERISATION OF WATER AND WASTE WATER

AIM

To study the sampling and preservation methods in water and waste water characterization and to learn the significance of characterisation of water and waste water

SAMPLING PROGRAMME AND PROCEDURES

The collection of a representative sample is the most important function of an environmentalist. The interpretation of results and recommendation for prevention and corrective treatment are all based on the analysis report. Scrupulous care in the collection of samples is therefore necessary to ensure that the sample is representative of the body of water under examination and to avoid spoilage and accidental contamination of the sample during collection and transport.

METHODS OF SAMPLING

Three types of samples are often collected depending on situations

a. Grab Samples

Grab samples are samples collected at a designated place at a particular time. They represent the composition at the time and space. When a source is known to vary in time, as in the case of waste effluents, grab samples collected at various time intervals and analysed separately can be of greater value.

b. Composite samples

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

c. Integrated samples

Integrated samples are a mixture of grab samples collected from different points simultaneously and mixed in equal volumes. Individual samples are collected from both banks of a river and at varying depths to represent available situations.

SAMPLING AND PRESERVATION REQUIREMENTS
1. Physical and Chemical Requirements:

For general physical and chenica1 examination, the sample should be collected in a chemically clean bottle made of good quality glass fitted with a ground glass stopper or a chemically inert polyethylene container. The volume of sample to be collected would depend on the selection of tests; however, for general examination 3.0 litre sample would be sufficient,

The following precautions must be taken while collecting the sample

i) The sampling location is representative of the water body

ii) The place is devoid of floating material

Where ever possible the sample should be collected 15cm, below the surface or as the situation warrants

No physical activity is permitted upstream of sampling point Shorter the time between collection and examination, the reliable will be the analytical results. For certain constituents and physical values, immediate analysis in the field is required, because, the composition of water may change before it arrives at the laboratory.

The maximum limits of storage are:

Unpolluted water: 72 hours Slightly polluted. : 48 hours Grossly polluted: 1 2hours

Some determinations are more likely to be affected by storage than others. Temperature may change, pH may change significantly, and dissolved gases may be evolved and lost (O2, CO2. and H2S)

FREQUENCY OF SAMPLING

Frequency depends on objectives. Yet, collection of samples of both raw and treated waters should be carried out as frequently as possible and at least once in every three months. Some waters undergo more pronounced seasonal variation and therefore require more frequent testing. Samples from treatment units should be collected and analyses frequently, at least one from each unit daily.

dd

2. BACTERIOLOGICAL REQUIREMENTS

The samples for bacteriological examination are collected in sterilized. neutral glass, glass-stopper 80z, and 300 ml bottles. The stopper and the neck should be protected by paper or parchment cover. If the sample is likely to contain traces of residual chlorine, an amount equal to 3.0 mg of sodium thiosul1ite (Na2s203, 51120) to neutralize chlorine is added to the bottle before sterilization. The sterilization is done at 15 psi (121°C) for 20-30 minutes in an autoclave.

The sterilized sample bottle should be kept unopened until the time of collection. The stopper should be removed with care to eliminate chances of spoiling and contamination and should never the rinsed. After filling, the stopper should be replaced immediately. The place of collection should be predetermined and procedure of collection conditioned depending on the source.

The standard procedure in sampling from a water faucet or tap is as follows:

a) Flame the tap briefly to kill clinging bacteria. This can be done with a piece of burning paper.

b) Turn on the water and allow it to run for 1 mm.

c) Remove the stopper from the bottle, being careful not to touch the inner portions o the stopper or bottle neck.

d) Fill bottle carefully, allowing no water to enter that has come in contact with hands. It is sometimes necessary to collect a sample from a reservoir or basin. If the water can be reached, remove the stopper, plunge the bottle below the surface and move the bottle while it is filling, so that no water will enter that has been in contact with hand. If the water is out of reach, as in a dug well, the bottle can be lowered with a cord.

The sample after collection should be examined immediately, preferably within one hour. If the conditions do not permit immediate examination, the sample should be stored at low temperatures. This period should in case be more than 24 hours. If storage or transportation is necessary, they should be got at a temperature between 0°C and 10°C.

FREQUENCY OF SAMPLING:

The frequency of sampling should be fixed depending on the magnitude of the problem involved. The number of samples to be examined from drinking water supply distribution system is normally decided on the basis of population served as given in the tabulation:

Population	Treated / untreated water entering distribution system	
	Max.interval between successive sampling	Max.no.of samples to be examined.
Upto 20,000	1 month	One sample for every 5000 population
20,001 – 50000	15 days	
50,001 – 1,00,000	4 days	
More than 100,000	1 day	One sample for every 10,000 population

The raw water should be examined as frequently as the situation demands. The frequency is also determined based on objectives of study.

3. BIOLOGICAL REQUIREMENTS

In general the samples for biological examination are collected in wide mouth, clean glass bottles of 2.0 litre capacity. They are never filled completely. This method is employed when total microscopic count is the aim. In some specific cases the concentrate of a sample may be collected through plankton nets made of bolting silk cloth, or the. sample filtered through Sedge wick Rafter funnels.

In general the sample must be examined microscopically within one hour of collections. If the facilities do not permit an immediate examination, it should be preserved after collection by addition of 2 ml neutralized (pH 7.0) formaline to each 100 ml of the sample.

There is no practice about the frequency of sampling but the examination should be made regularly, or else as the situation demands. Benthos study is complex, Collection through cages placed at proper preselected sites for a defined period of time is

recommended.

PRESERVATION METHODS FOR CHARACTERISATION OF SAMPLES:

Determination	Containers	Minimum sample size ml	Preservation	Max.storage recommended
pH	P,G	-	Analyse immediately preferably in field	0.5 h
Solids	P,G	300	Refrigeration	7 d
Sulfates	P,G	100	Refrigerate	28 d
D.O	G,BOD Bottle	300	Titration may be delayed after fixation (1 ml Alk.KI and 1 ml MnSO4) and acidification	8 h
Turbidity	P,G	-	Analyse same day store in dark, refrigerate	24 h
Hardness	P,G	100	Add HNO3 to pH<2	6 months
Fluoride	P	300	None	28 d
God	P,G	100	Analyse as soon as possible or add H2SO4 to pH<2	7 d

B.O.D	**P,G**	**1000**	**Refrigerate**	**6h**
Chlorine residuals	**P,G**	**500**	**Analyse immediately**	**0.5 h**
Ammoniacal Nitrogen	**P,G**	**500**	**Analyse as soon as possible or add H_2SO_4 refrigerate**	**6 months**

Note: **Refrigerate – Storage at 40C; P=plastic (Polyethylene or equivalent), G=Glass, neutral.**

SIGNIFICANCE OF CHARACTERISATION OF VARIOUS PARAMETERS

Natural waters are never completely pure. During their precipitation and passage over or through ground they require a wide variety of dissolved and suspended impurities. The concentrations of these impurities are seldom large in ordinary chemical sense but they modify the chemical behaviour of water or its usefulness.

The waste waters generally have values far higher than in waters.
Some of these impurities are toxic, some may affect health, and some affect the portability while others indicate pollution. A list of such impurities is given below:

Toxic substances	**Max. allowable limit (W.H.O. Standards) mg/L**
Lead	**0.05**
Arsenic	**0.05**
Selenium	**0.01**
Chromium	**0.05**

Cyanide	**0.2**
Cadmium	**0.01**
Radio active compounds (gross beta activity)	**1000**mm **c/L**
Components hazardous to health	
Fluorides	**1.5**
Nitrates as NO3	**45**
Compounds affecting the portability	
TDS	**1500**
Iron	**5.0**
Managese	**5.0**

Copper	**1.5**
Zinc	**1.5**
Magnesium plus – sodium sulfate	**1000**
Surfactants (ABS)	**0.5**
Chemical indicators of population	
BOD	**60**
COD	**10.0**
Total Nitrogen exclusive of NO3	**1.0**

Ammonia cal Nitrogen as NH3	**0.5**
Carbon chloroform extract (CCE)	**0.5**
Oil and Grease	**1.0**
D.O	**40% saturation**

HARDNESS

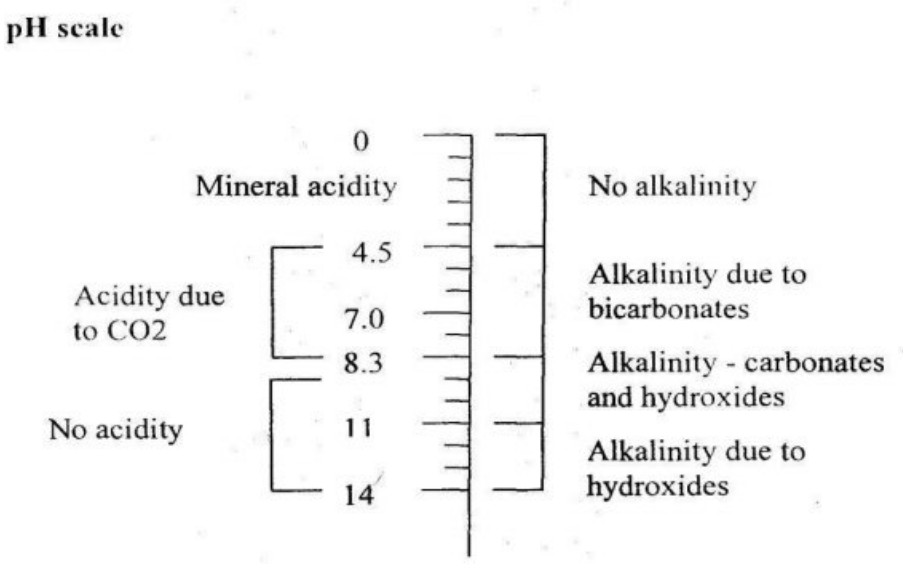
The study of hardness is important from the point of view of industrial utilization of water especially in boilers, where scales are formed. Hardness in municipal supplies increases the consumption of soap, fuel, tea leaves etc. in the household and renders it unsuitable for use in air-conditioning.

TURBIDITY

It is a measure of degree of opaqueness of water and interference presented by suspended matter to the passage of light. The turbidity is due to clay, silt, finely divided organic matter and microscopic organisms. Turbidity tests are important from aesthetic consideration and from the point of economics of treatment. The most important health significance of turbidity is that may, harbour pathogenic organisms.

pH

Determinations of pH, alkalinity and its forms, along with acidity are of interest in coagulation, softening and corrosion control



RESIDUE OR SOLID MATTER

The test for residue is of very great importance in sewage treatment processes to indicate the physical state of the principal constituents. The ratio of the weight of suspended solids to turbidity often referred as coefficient of fineness. The solids present in dissolved form are related to the electrical conductivity. The fixed solids indicate the mineral level while volatile solids are related to organic matter.

CHLORIDE

Concentration of chlorides in municipal sewage is often significantly (15-50 mg/L) higher than those in its water supply. For this reason, a change in its concentration may be indicative of sewage pollution, in waters of low chloride concentration. Chlorides occur in an all natural waters in widely varying amounts. Mountain streams are normally low in chloride values. Chloride gain access to water either because of excellent solvent properties or through human excreta or industrial pollutants. Chlorides were for several years used as an indicator of pollution by municipal wastes in rivers, streams, wells and lakes.

DISSOLVED OXYGEN

In raw water and domestic wastes, dissolved oxygen is a factor which determines whether the biological processes undergoing a change are aerobic or anaerobic. It is very desirable that aerobic conditions are maintained. It is a Single test which will immediately indicate the sanitary status of a stream. Low values of dissolved oxygen adversely affect the portability of water and may cause fish kill.

ORGANIC MATTER

The tests of organic matter indicate type and extent of pollution, which has its origin in plant or animal matter. Tests are mostly restricted to the study of nitrogen in various forms and oxygen requirements in biodegradation of puterscible carbonaceous organic matter (BOD). A measure of the demand is also indicated in terms of demand through strong chemical oxidants (COD)

BOD.

The BOD is the amount of oxygen required by bacteria while stabilizing decomposable organic matter under aerobic conditions. Polluted water does not contain sufficient oxygen in solution to maintain aerobic condition during decomposition. The quantity of oxygen required for complete stabilization is taken as a measure of its organic content.

COD

The COD test is based on the concept that a large majority of organic compounds can be completely oxidized by the action of strong oxidizing agents in acidic medium. The

quantity of oxygen required is proportional to organic matter, regardless of the biological assimilability of the substance.

NITROGEN

Nitrogen is estimated as organic nitrogen, ammonical nitrogen, nitrite nitrogen and nitrate nitrogen throw light on the pollutional history of the carrying water.

BACTERIOLOGICAL TESTS

The routine bacteriological tests are aimed at enumerating the members of coliform group, which are considered indicators of pollution. The natural habitat of these bacteria is the intestinal tract of man and other warm blooded animals. They are present wherever the pathogens are present and by their absence exclude the probability of the presence of pathogens. They share the fate of the most significant pathogenic enteric bacteria outside the human and animal body both in the rate of death and in the rate of removal when water is purified.

Another test of bacteria is aimed at detecting chemo-synthetic heterotrophic heterogeneous group developing under conditions of cultivation and is referred as Total Plate Count. This test is not differential and indicates a total picture of bacteria associated with organic matter.

Biological Examination

The biological examination (microscopic) provides useful information for the control of water quality and treatment. It serves for one or several of the following purposes:

i) To explain the cause of color or an odor in water
ii) To aid in the interpretation of various chemical analysis reports
iii) Permitting identification of specific water when it is mixed with another
iv) To explain clogging of pipes/screens/filters
v) Rapidly detect organic pollution and contamination with toxic substances
vi) To indicate the progress of self purification streams.

RESULT

Thus the sampling and preservation methods in water and waste water characterization and to learn the significance of characterisation of water and waste water are studies

ExNo.1 EXPERIMENT ON DETERMINATION OF pH

PREAMBLE:

"**How to determine pH in Water and Wastewater**".

Test procedure is in accordance to IS: 3025 (Part 11) - Reaffirmed 2002.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

(1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 4500-H^+ B.

(2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 150.1.

AIM

To determine the PH of the given water sample with the stipulations as per **IS: 3025 (Part 11) - Reaffirmed 2002**

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. 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 H^+ and 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.

Dewatering of sludges, oxidation of cyanides and reduction of hexavalent chromium into trivalent chromium also need a favorable pH range. It is used in the calculation of

carbonate, bicarbonate, CO_2 corrosion, stability index and acid base equilibrium.

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 trihalomethanes, which are causing cancer in human beings.

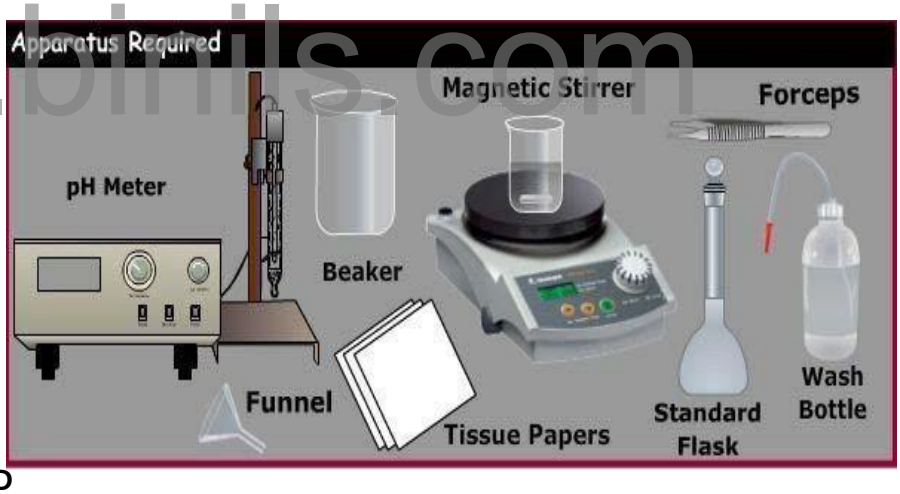
PRINCIPLE

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.

MATERIALS REQUIRED

APPARATUS REQUIRED

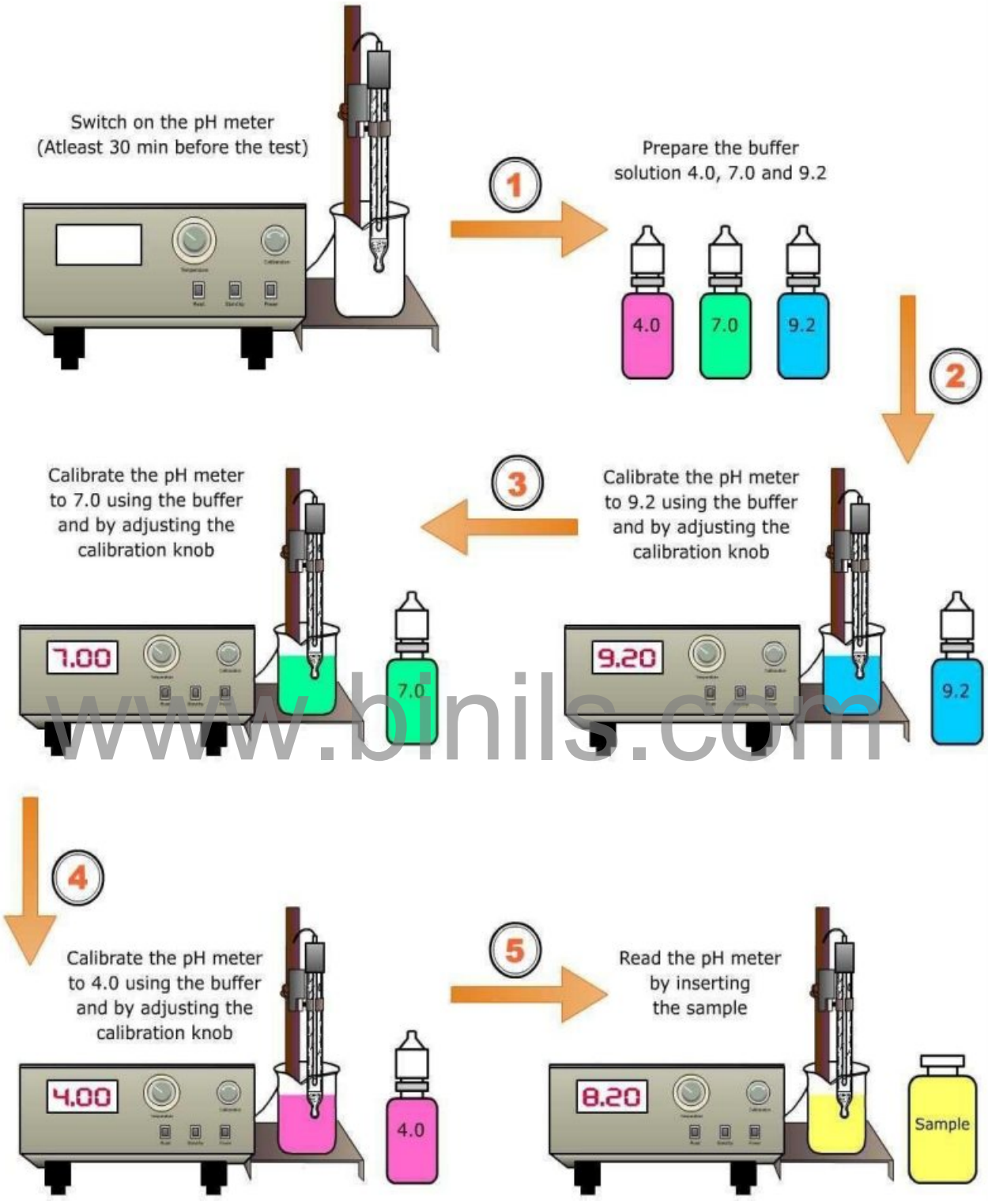
1. pH meter
2. Standard flasks
3. Magnetic Stirrer
4. Funnel
5. Beaker
6. Wash Bottle
7. Tissue Paper
8. Forceps



CHEMICALS REQUIRED

1. Buffers Solutions of pH 4.01, 7.0 and 9.2
2. Potassium Chloride
3. Distilled Water

PROCEDURE CHART



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 4^0 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:

i. 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.

ii. In general, the glass electrode is not subject to solution interferences like color, high salinity, colloidal matter, oxidants, turbidity or reductants.

iii. 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.

iv. Before using, allow the electrode to stand in dilute hydrochloric acid solution for at least 2 hours.

v. 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

PREPARATION OF REAGENTS

1. Buffer Solution of pH 4.0

- Take 100 mL standard measuring flask and place a funnel over it.
- Using the forceps carefully transfer one buffer tablet of pH 4.0 to the funnel.
- Add little amount of distilled water, crush the tablet and dissolved it.
- Make up the volume to 100 mL using distilled water.

2. Buffer Solution of pH 7.0

- Take 100 mL standard measuring flask and place a funnel over it.
- Using the forceps carefully transfer one buffer tablet of pH 7.0 to the funnel.
- Add little amount of distilled water, crush the tablet and dissolved it.
- Make up the volume to 100 mL using distilled water.

3. Buffer Solution of pH 9.2

- Take 100 mL standard measuring flask and place a funnel over it.
- Using the forceps carefully transfer one Buffer tablet of pH 9.2 to the funnel.
- Add little amount of distilled water, crush the tablet and dissolved it.
- Make up the volume to 100 mL using distilled water.

CALIBRATING THE INSTRUMENT

Using the buffer solutions calibrate the instrument.

Step 1

In a 100 mL beaker take pH 9.2 buffer solution 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 stirred buffer and check for the reading in the pH meter.

If the instrument is not showing pH value of 9.2, using the calibration knob adjust the reading to 9.2.

Take the electrode from the buffer, wash it with distilled water and then wipe gently with soft tissue.

Step 2

In a 100 mL beaker take pH 7.0 buffer solution 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 stirred buffer and check for the reading in the pH meter.

If the instrument is not showing pH value of 7.0, using the calibration knob adjust the reading to 7.0.

Take the electrode from the buffer, wash it with distilled water and then wipe gently with soft tissue.

Step 3

In a 100 mL beaker take pH 4.0 buffer solution 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 stirred buffer and check for the reading in the pH meter.

If the instrument is not showing pH value of 4.0, using the calibration knob adjust the reading to 4.0.

Take the electrode from the buffer, wash it with distilled water and then wipe gently with soft tissue.

Now the instrument is calibrated.

TESTING OF SAMPLE

- 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.
- The pH of the given water sample is 8.84
- Take the electrode from the water sample, wash it with distilled water and then wipe gently with soft tissue.

CALCULATION

To determine the value of pH of the given water sample the readings obtained are required to be tabulated

TABLE

Sample No	Temperature of Sample (ºC)	pH
1.		
2.		
3.		

DATA SHEET

DETERMINATION OF pH

Date Tested :

Tested By :

Name :

Sample Number :

Sample Location

BH1 : Sample

Description :

Sample Location

BH2 : Sample

Description :

Sample Location

BH2 : Sample

Description :

TABULATION

Sample No	Temperature of Sample (ºC)	pH
1		
2		
3		

Result:-

The pH of the given sample

1 = The pH of the given

sample 2 =

The pH of the given sample 3 =

INTERPRETATION OF RESULTS

The pH of the given water sample is

INFERENCE

pH is a measure of the hydrogen ion concentration in water. Values lower than 7 indicate acidity and values higher than 7 indicate alkalinity. Drinking water with a pH between 6.5 and 8.5 is generally considered satisfactory. Acidic waters tend to be corrosive to plumbing and faucets, particularly if the pH is below 6. Alkaline waters are less corrosive. Waters with a pH above 8.5 may tend to have a bitter taste.

The pH of the water samples are well within the limit of the drinking water standards. The pH of the ground water is slightly towards the alkaline side because of some soil and rocks chemicals might have dissolved in it. In case of the pH of the fresh water, aquatic plants uses up hydrogen molecules for photosynthesis, which causes the concentration of hydrogen ions to decrease and therefore the pH is towards the alkaline side. The sea water is mostly alkaline in nature because of the presence of different type of salts.

EVALUATION

1. pH is defined as_____.
   a) Logarithm of Hydrogen ions concentration
   b) Negative logarithm of Hydrogen ions concentration
   c) Hydrogen ion concentration
   d) OH ion concentration

2. pH of neutral water is_____.
   a) less than 7
   b) more than 7
   c) 7.0
   d) 0.0

3. The acceptable value of pH of potable water is_____. a) 7.0 to 8.5
   b) 6.5 to 9.5
   c) 6 to 8.5
   d) 6.5 to 10

4. The inner solution present in the glass electrode of pH meter is_____.

    a) HCl
    b) KCl
    c) NaCl
    d) MgCL

5. The buffer solution can be stored for a minimum period at room temperature.

    a) True
    b) False

6. Possible reasons for a relatively low pH value in a river water sample is due to____.

    a) Organic material decomposition to form acidic substances
    b) Running long distances
    c) Presence of fishes
    d) Presence of aquatic plants

7. Possible reasons for a relatively high pH value in a river water sample is due to____.

    a) Running over clay
    b) Running long distances
    c) Running of fishes
    d) Presence of aquatic plants

8. A weak acid is one that ionize incompletely in aqueous solution.

    a) True
    b) False

9. A strong base is one that ionizes incompletely in aqueous solution.

    a) True
    b) False

10. The measurement of pH made by determining the e.m.f of the_____.

    a) cell constant
    b) solution
    c) electrode cell
    calomel electro

Ex. No.2 EXPERIMENT ON DETERMINATION OF TURBIDITY

PREAMBLE:

"**How to determine turbidity in Water and Wastewater**".

Test procedure is in accordance to IS: 3025 (Part 10) - Reaffirmed 2002.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

(1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 2130 B.

(2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 180.1.

AIM

To determine the turbidity of the given water sample with the stipulations as per **IS: 3025 (Part 10) - Reaffirmed 2002.**

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.

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.

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.

MATERIALS

REQUIRED

APPARATUS

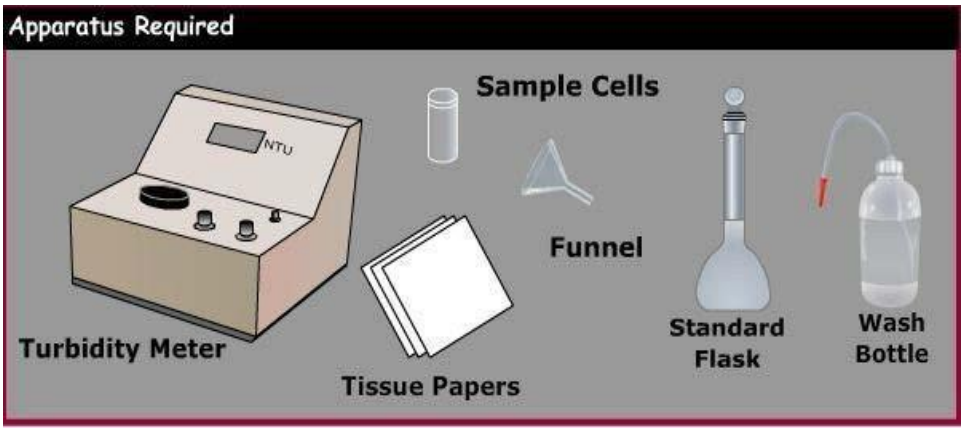
REQUIRED

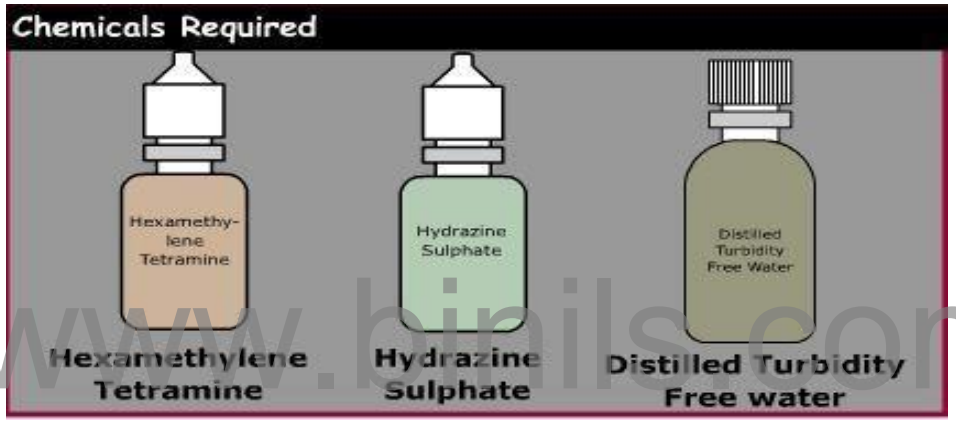
1. Turbidity Meter
2. Sample Cells
3. Standard flasks
4. Funnel
5. Wash Bottle
6. Tissue Papers

CHEMICALS REQUIRED

1. Hexamethylenetetramine
2. Hydrazine sulphate

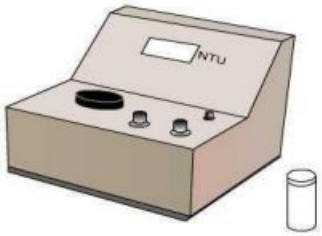
3. Distilled water





PROCEDURE CHART

Switch on the Turbidity meter
(Atleast 30 min before the test)

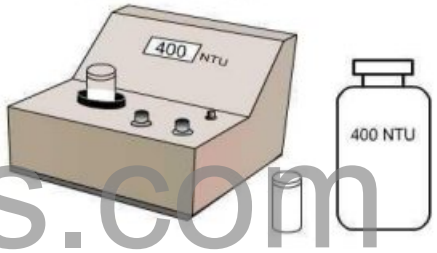


1

Prepare 400 NTU
solution

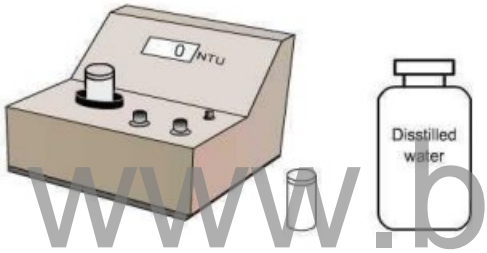
2

Calibrate the turbidity meter
to 400 NTU using
the standard solution
by adjusting the
calibration knob



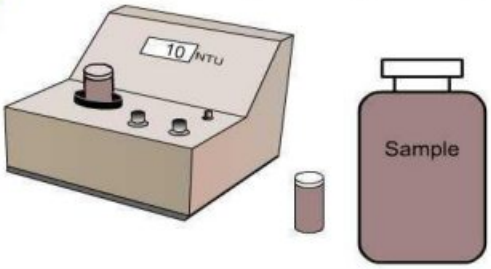
3

Calibrate the turbidity meter
to 0 NTU using disstilled water
and by adjusting the
calibration knob



4

Read the Turbidity meter
by inserting 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.

PROCEDURE

For testing the given water sample first the reagents are to be prepared. Then the turbidity meter is required to be calibrated.

PREPARATION OF REAGENTS

1. Hydrazine Sulphate

- Weigh accurately 1 g of hydrazine sulphate and dissolve it in turbidity free distilled water.
- Take 100 mL standard measuring flask and place a funnel over it.
- Transfer it to a 100 mL standard flask and make up to 100 ml using turbidity free distilled water.

2. Hexamethylene Tetramine

- Weigh accurately 10 g of Hexamethylene tetramine and dissolve it in turbidity free distilled water.

- Take 100 mL standard measuring flask and place a funnel over it.
- Transfer it to a 100 mL standard flask and make up to 100 ml using turbidity free distilled water.

3. Standard 4000 NTU Solution

- Mix 5 mL of hydrazine sulphate solution and 5 mL of Hexamethylenetetramine solution in a 100 mL standard measuring flask.
- Allow the mixture to stand for 24 hours.
- After 24 hours, make up the volume to 100 mL using turbidity free distilled water.
- The standard 4000 NTU solution is ready.

CALIBRATION OF TURBIDITY METER

Using the standard solution calibrate the instrument.

The instrument is having four knobs, out of which the two knobs in the bottom is the set zero knob, this is for setting the instrument to zero.

The one which is there in the top left hand side is the calibration knob, used for the calibration.

The other one in the top is the knob for setting the detection range. It is adjusted to 1000 NTU range.

Step 1

To the sample cells, add turbidity free distilled water up to the horizontal mark, wipe gently with soft tissue. Place it in the turbidity meter such that the vertical mark in the sample cell should coincide with the mark in the turbidity meter and cover the sample cell. Now using the set zero knob, adjust the reading to zero.

Step 2

According to our need, prepare a standard solution. In this case, a 200 NTU solution is prepared by diluting the standard 4000 NTU solution and added to the sample cells, up to the horizontal mark, wipe gently with soft tissue. Place it in the turbidity meter such that the vertical mark in the sample cell should coincide with the mark in the turbidity meter and cover the sample cell.

If the instrument is not showing 200 NTU, using the calibration knob adjust the reading to 200 NTU.

Repeat the procedure for two / three times. Now the instrument is calibrated.

TESTING OF WATER SAMPLE

- To the sample cells, add sample water up to the horizontal mark, wipe gently with soft tissue and place it in the turbidity meter such that the vertical mark in the sample cell should coincide with the mark in the turbidity meter and cover the sample cell.
- Check for the reading in the turbidity meter. Wait until you get a stable reading.
- The turbidity of the given water sample is 8.4 NTU.

CALCULATION

For determining the Turbidity of the given water sample the readings are required to be tabulated.

TABLE

Sample No.	Temperature of Sample (ºC)	Turbidity (NTU)
1.		
2.		
3.		

DATA SHEET

DETERMINATION OF TURBIDITY DATA SHEET

Date Tested :

Tested By :

Project Name :

Sample Number : **BH1, BH2, BH3**

Sample Location

BH1 : Sample

Description :

Sample Location

BH2 : Sample

Description :

Sample Location

BH2 : Sample

Description :

TABULATION

Sample No	Temperature of Sample (ºC)	Turbidity (NTU)

Result:-

The turbidity of the given sample

1 = The turbidity of the given

sample 2 =

The turbidity of the given sample 3 =

INTERPRETATION OF RESULTS

The turbidity of the given water sample is

INFERENCE

Turbidity is a measure of light transmission and indicates the presence of suspended material such as clay, silt, finely divided organic material, plankton and other inorganic material. If turbidity is high, be aware of possible bacterial contamination. Normally the ground water is clear in nature and it will satisfy the code's need. The ground water may get contaminated by intrusion of domestic or industrial wastewater causing turbidity of the sample. Turbidity in excess of 5 NTU is usually objectionable for aesthetic reasons. In case of freshwater lakes and ponds, due to contamination and algal growth the turbidity of these water increases to very high levels. The clarity of sea water is very low because of huge amount of suspended particles, thereby increasing the turbidity.

EVALUATION

1. Turbidity is caused by Clay, Silt, Organic matter and Microbes.
    a) True
    b) False

2. The turbidity is measured based on the
    a) Light absorbing properties
    b) Light Scattering properties
    c) Particle Size
    d) Particle mass

3. The colour of the water sample affects the turbidity.
    a) True
    b) False

4. In a nephelo turbidity meter the light detectors
    are at a) 180°
    b) 360°
    c) 90°
    d) 270°

5. What is the unit of turbidity.

a) TU
b) MTU
c) NTU
d) IU

6. What is the light source for the nephelo turbidity meter?

a) Tungsten filament lamp
b) Deuterium lamp
c) Hallow Cathode lamp
d) Sodium vapour lamp

7. The turbidity affects the aquatic life in the water.

a) True
b) False

8. The standard unit of turbidity is considered as that produced by

a) 2ppm of silica in distilled water
b) 1ppm of silica in distilled water
c) 4ppm of silica in distilled water
d) 9ppm of silica in distilled water

9. The material used in the standard solution for nephelometer is____.

a) Silica
b) Clay
c) Formazin
d) Barium Chloride

10. Mixture of hydrazine sulphate and hexamethylenetetramine solution is allowed to stand for____.

a) 24 hours
b) 12 hours
c) Minimum 6 hours
d) No specific time

Ex. No. 3 EXPERIMENT ON DETERMINATION OF TOTAL HARDNESS

PREAMBLE:

"**How to determine total hardness in Water and Wastewater**".

Test procedure is in accordance to IS: 3025 (Part 21) - Reaffirmed 2002.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

(1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 2340 - C.
(2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 130.2.

AIM

To determine the total hardness of given water sample with the stipulations as per **IS: 3025 (Part 21) - Reaffirmed 2002.**

INTRODUCTION

Water that has high mineral content is known as **Hard water**. Hard water contains bicarbonate, chlorides and sulphates of calcium and magnesium.

When treated hard water with soap, it gets precipitated in the form of insoluble salts of calcium and magnesium. Hardness of water is a measure of the total concentration of the calcium and magnesium ions expressed as calcium carbonate. There are two types of hardness

1. Temporary hardness
2. Permanent hardness

Temporary Hardness is due to the presence of bicarbonates of calcium and magnesium. It can be easily removed by boiling.

Permanent Hardness is due to the presence of chlorides and sulphates of calcium and magnesium. This type of hardness cannot be removed by boiling.

ENVIRONMENTAL SIGNIFICANCE

- Scales are formed as inner coating of the pipelines prevents corrosion.
- Absolutely soft waters are corrosive and dissolve the metals.
- More cases of cardio vascular diseases are reported in soft water areas.
- Hard water is useful to growth of children due to the presence of calcium.
- Hard waters cause excessive consumption of soap used for cleaning

purpose. Sodium soaps react with multivalent metallic cations to form a precipitate, thereby lose their surfactant properties. Lathering doesn't take place until all hardness ions precipitate out.

- This precipitate adheres to surfaces of tubes, sinks, dish washer and may stain clothing.
- Scales formed mainly due to carbonate hardness act as insulations and cause enormous loss of fuel in boiler.
- Scales deposited mainly due to increase in pH to 9 at which bicarbonates are converted as carbonates are formed in distribution mains reducing their carrying capacity.

PRINCIPLE

A water sample is buffered to pH 10.1 and taken in to a conical flask. If an indicator dye like EBT, when added to a solution containing Calcium and Magnesium ions, the color of the solution turns to wine red.

EDTA, the titrant, complexes with Magnesium and Calcium ions, removing them from association with the indicator.

When all the Mg and Ca are complexed with EDTA, the indicator will turn blue. This is the end point of the titration.

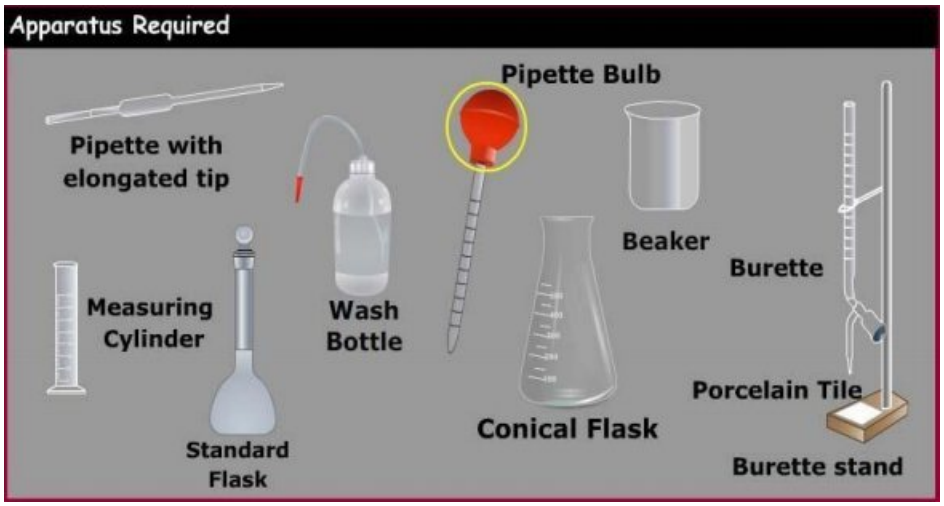
MATERIALS REQUIRED

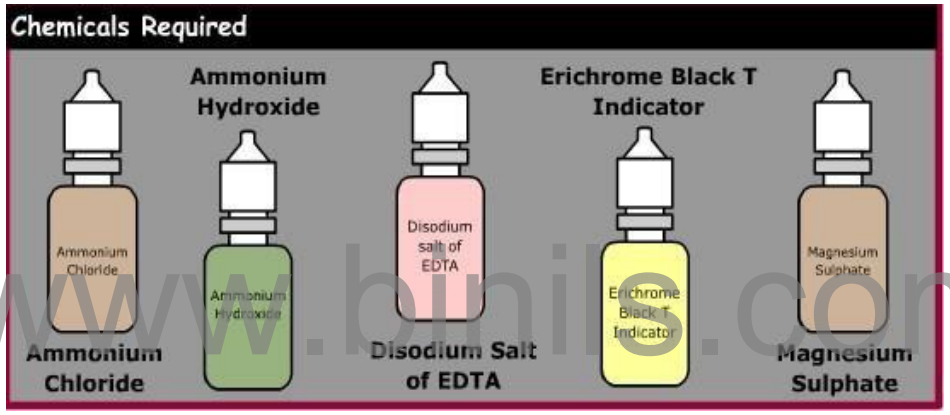
APPARATUS REQUIRED

1. Burette with Burette stand and porcelain title
2. Pipettes with elongated tips
3. Pipette bulb
4. Conical flask (Erlenmeyer Flask)
5. 250 mL graduated cylinders
6. Standard flask
7. Wash Bottle
8. Beaker

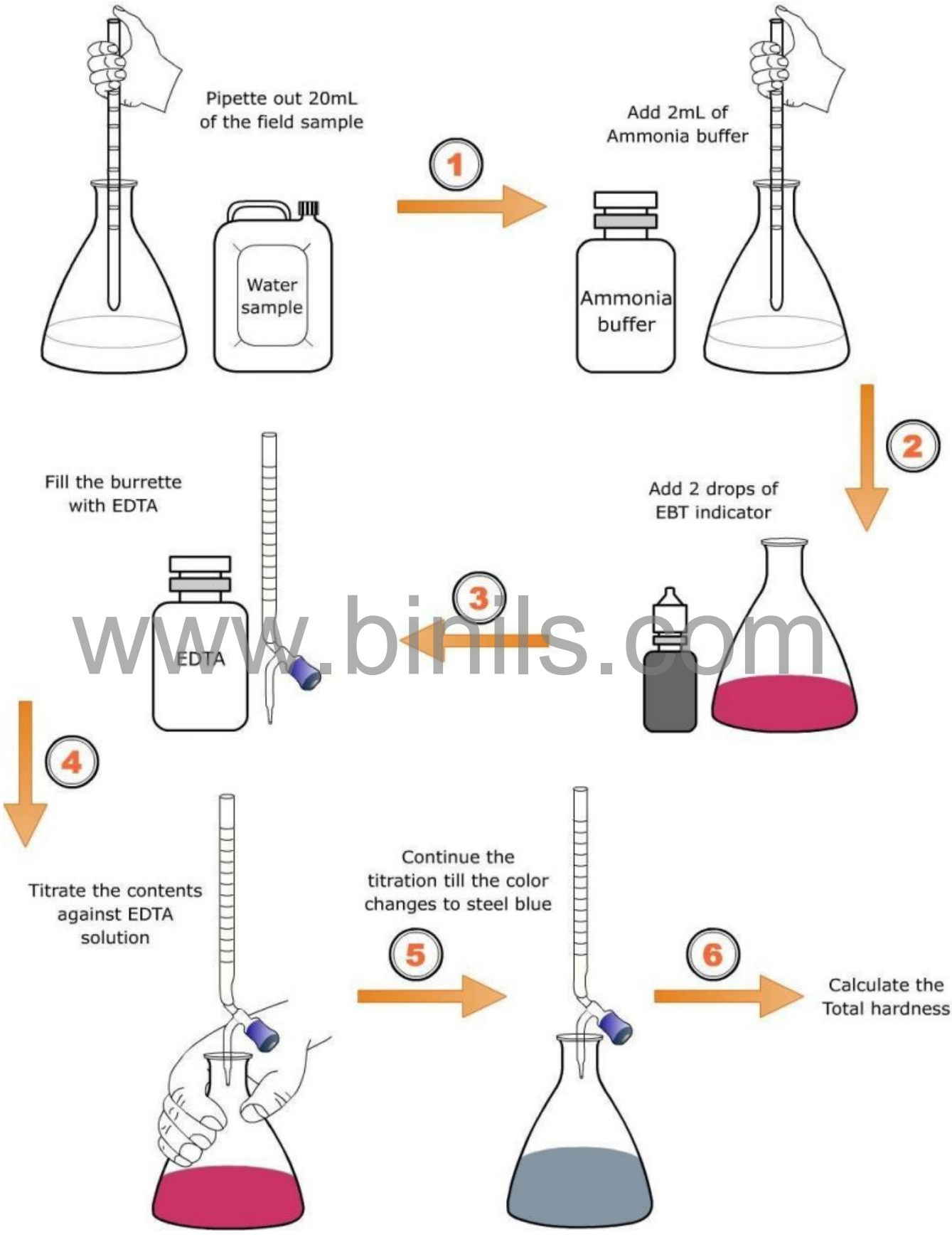
CHEMICALS REQUIRED

1. Ammonium Chloride
2. Ammonium Hydroxide
3. EDTA (Disodium Salt of EDTA)
4. Erichrome Black T
5. Magnesium sulphate





PROCEDURE CHART



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 with in two hours of collection, cool storage is not necessary. If analysis can not 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

PRECAUTIONS

- Here we are handling ammonia solution so necessary precaution should be taken for preventing the inhalation.
- It causes irritation if inhaled.
- Do not pipette out the buffer solution using either measuring cylinder, automatic pipette (or) pipette with a sucker.
- Always store EDTA solution and buffer solution in a plastic or resistant glass container.
- Discard the buffer solution if it is turbid or if it is stored for a very long period of time.

PROCEDURE

PREPARATION OF REAGENTS

Buffer Solution preparation

- Switch on the Electronic balance, keep the weighing pan, set the reading to zero.
- Measure 50 mL of distilled water and transfer it to the beaker
- Weigh 1.179g of EDTA
- Now the weight is 1.179gms
- Transfer the contents to the beaker having 50 mL of distilled water and dissolve it thoroughly.
- Weigh 16.9g of ammonium chloride.
- Add it to the contents in the beaker. And dissolve it thoroughly.
- Weigh 780mg of magnesium sulphates and transfer it to the beaker.
- Measure 143 mL of Ammonium hydroxide solution using measuring cylinder and add it to the contents in the beaker.

- Place the funnel over the 250mL standard flask and transfer the dissolved contents from beaker
- Make the volume upto 250mL mark by adding distilled water. Transfer the buffer solution to a clean reagent bottle labelled as buffer solution. This buffer solution is used to maintain the pH of water sample between 9 and 10.

Erichrome Black T

- Weigh 0.5g of Erichrome black T
- Transfer it to 100mL standard flask using funnel
- Add distilled water in the standard flask and make the volume exactly upto 100 mL mark.
- Put the lid and shake the contents well.
- Transfer the solution to a clean reagent bottle named EBT

Standard EDTA Solution (0.02 M)

- Switch on the Electronic balance, keep the weighing pan, and set the reading to zero.
- Weigh 3.723g of EDTA sodium salt
- Transfer the entire content to 1000 mL standard flask
- Fill with distilled water up to 1000 mL mark
- Put the lid and shake the contents well.
- For easy handling take the EDTA solution in a 250 mL beaker.

TESTING OF WATER SAMPLE

- Pipette 20mL of water sample and transfer it to a clean 250mL conical flask.
- Add 2mL of Ammonia buffer solution to the water sample so that the pH will be maintained between 9 and 10.
- Add few drops of EBT indicator to the conical flask and the sample turns to wine red in color.
- Before starting the titration rinse the burette with few mL of EDTA. Fill the burette with 0.02M EDTA solution and adjust to zero then fix it in burette stand.
- Titrate the sample against the EDTA solution in the burette till all calcium and magnesium ions present in the sample reacts with the EDTA. The appearance of blue colour indicates that all Ca & Mgions

are complexed with EDTA and forms a metal EDTA complex i.e., the end point of the titration.

- Note down the burette reading
- The value of titration is 29.8mL
- Repeat the titration for concordant values

CALCULATION

Burette solution: EDTA
Pipette solution: Sample
Indicator: EBT

End point: Appearance of blue color

TABLE

Sample No	Volume of Sample (mL)	Burette Reading (mL)		Volume of EDTA (mL)
		Initial	Final	
1.				
2.				
3.				

DATA SHEET

DETERMINATION OF TOTAL HARDNESS

Date Tested :

Tested By :

Project Name :

Sample Number :

Sample Location : Sample Description :

Sl.No.	**Volume of Sample (mL)**	**Burette Reading (mL)**		**Volume of EDTA (mL)**
		Initial	**Final**	
1.				
2.				
3.				

Calculation:

Volume of EDTA = mL
Normality of EDTA = N
Volume of Sample = mL
Equivalent weight of $CaCO_3$ = 50

Total Hardness = $\frac{\text{Volume of EDTA} * N * 50 * 1000}{\text{Volume of sample taken}}$

To convert the sample size from mL to L, multiply the result by 1,000

mL/L Calcium Hardness as $CaCO_3$ equivalent (mg/L) =

= **mg/L as $CaCO_3$ equivalent**

INTERPRETATION OF RESULTS

The Total Hardness of the given sample of water = **mg/L.**

INFERENCE

Hardness is the property which makes water to form an insoluble precipitate with soap and is primarily due to the presence of calcium and magnesium ions. Hard waters have no known adverse health effects and may be more palatable than soft waters. Hard water is primarily of concern because it requires more soap for effective cleaning, causes yellowing of fabrics, toughens vegetables cooked in the water and forms scales in boilers, water heaters, pipes and cooking utensils. The hardness of good quality water should not exceed 250 mg/L measured as calcium carbonate equivalents. Waters softer than 30 to 50 mg/L may be corrosive to piping depending on pH, alkalinity and dissolved oxygen.

EVALUATION

1. The cation that cannot cause hardness is_____.

    a) Ca
    b) Mg
    c) Fe
    d) H

2. Addition of complexometrically neutral Mg to buffer is to

    a) get a clear precise end point
    b) neutralize sodium present
    c) neutralize calcium present
    d) maintain pH of the buffer

3. The hard water does not lather with soap because Soap is precipitated

    a) by Ca and Mg ions
    b) only by Mg ions
    c) only by Ca ions
    d) by Na ions

4. The buffers can be stored

    a) for a month
    b) no time limit
    c) for an year
    d) as long as the solution is clear without any turbidity

5. The need for buffer in the titration is____.

    a) for maintaining volume
    b) for maintaining the pH
    c) for maintaining the temperature
    d) to nullify the error

6. The hard water consumes more soap for cleaning purposes.

    a) True
    b) False

7. E.D.T.A. means

    a) Ethylene diamine tetra acetic acid
    b) Erichrome diamine tetra acetic acid
    c) Ethylene dye toluene acid
    d) Erichrome dye toluene acid

8. The hard water

    a) is corrosive
    b) forms scales
    c) is tasteless
    d) is costly

9. Buffer solution is the solution which resists change in its

    a) pH
    b) colour
    c) turbidity
    d) conductivity

10. The Erichrome Black T is used as a catalyst.

    a) True
    b) False

Ex.No.4. EXPERIMENT ON DETERMINATION OF CALCIUM HARDNESS

PREAMBLE:

"**How to determine calcium hardness in Water and Wastewater**".

Test procedure is in accordance to IS: 3025 (Part 40) - Reaffirmed 2003.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

(1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 3500 – Ca-D.
(2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 215.2.

AIM

To determine the calcium hardness in the given water sample with the stipulations as per **IS: 3025 (Part 40) - Reaffirmed 2003**.

INTRODUCTION

Water hardness is an expression for the sum of the calcium and magnesium cat- ions concentration in a water sample.

Calcium is usually found in highest concentrations in natural water.

The presence of calcium in water results from deposits of lime stone, gypsum etc. Calcium is one of the principal cations involved in water hardness.

Calcium hardness is the estimation of hardness due to calcium in water.

These cations form insoluble salts with soap and decrease the cleaning effectiveness of soap.

They also form hard water deposits in hot water heaters.

The calcium content may range from zero to several hundred ppm.

ENVIRONMENTAL SIGNIFICANCE

The relative amounts of Calcium hardness, Carbonate and non-Carbonate hardness present in water are the factors while determining the most economical type of softening process.

Determination of hardness serves as a basis for routine control of softening processes.

Hard water typically contains high concentrations of Ca and Mg cations, which interfere with the use of the water for many applications.

These ions diminish the effectiveness of soaps and detergents for cleansing operations.

They diminish the drinking quality of water and they contribute to the accumulation of insoluble salt deposits in storage vessels or plumbing.

PRINCIPLE

The quantity of calcium in water will be determined by titrating the water sample with a standard Ethylene Diamine Tetra Acetic acid (EDTA) of known volume and concentration. Based on the stoichiometry of the reactions and the number of moles of EDTA required to reach the end point, the concentration of calcium content in water is calculated.

An indicator, ammonium purpurate which combines only with calcium is used.

The indicator imparts a pink color to the solution while there are calcium and magnesium ions that have not complexed with EDTA.

Once the endpoint has been reached and there is no more uncomplexed Ca or Mg, the solution will turn to purple color. No hint of pink color will be left.

MATERIALS REQUIRED

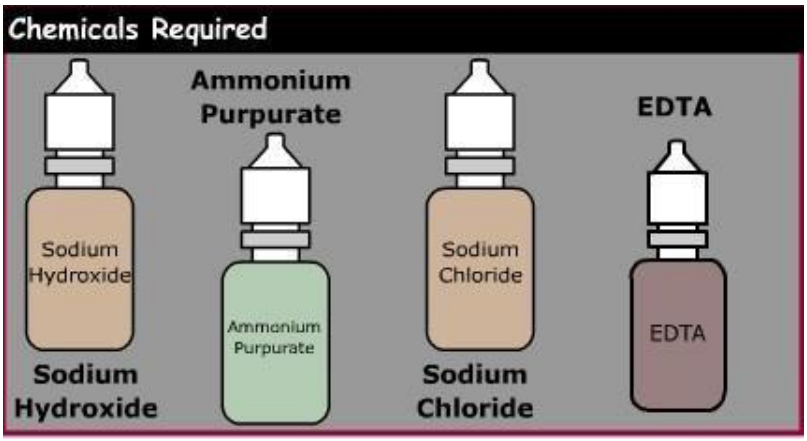
APPARATUS REQUIRED

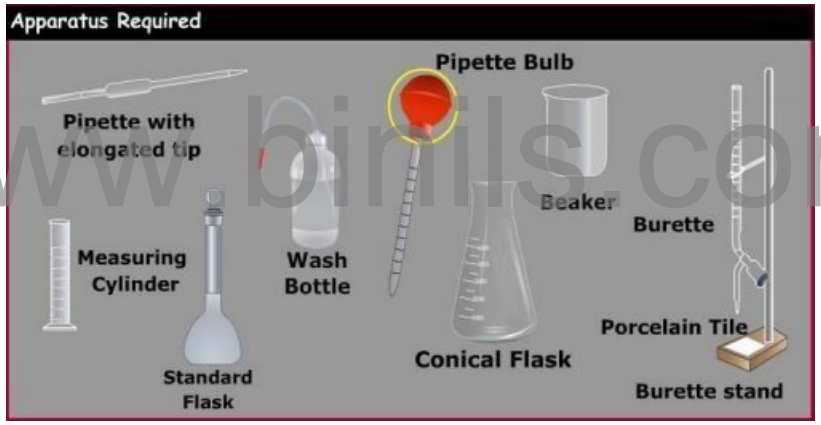
1. Burette with Stand
2. Porcelain tile
3. Pipettes with elongated tips
4. Conical flask
5. 250 mL Graduated Cylinder
6. Standard Flask
7. Beakers
8. Wash bottle

CHEMICALS REQUIRED

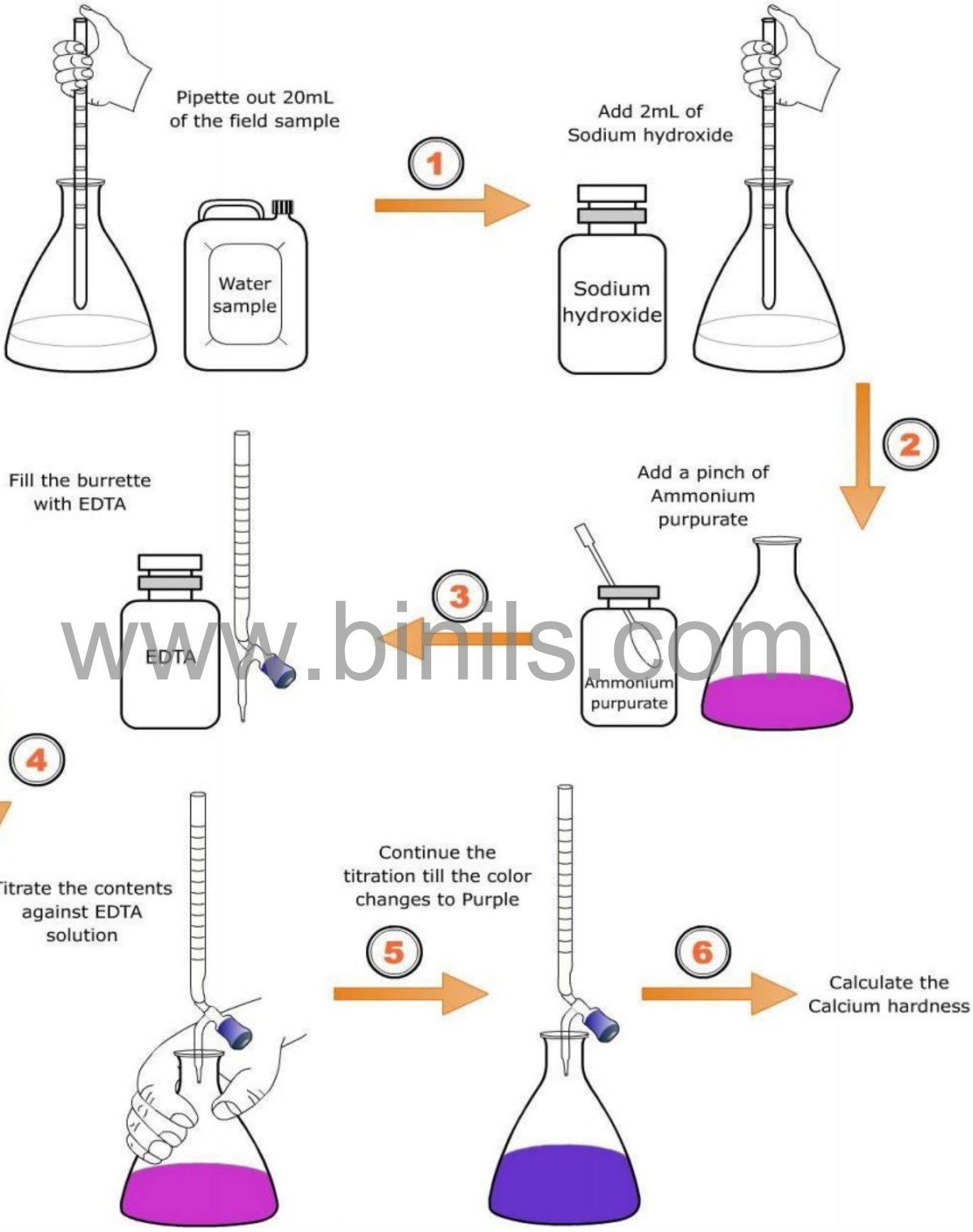
1. Ammonium Purpurate
2. Sodium Chloride

3. Sodium Hydroxide
4. EDTA





PROCEDURE CHART



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 with in two hours of collection, cool storage is not necessary. If analysis can not 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

PRECAUTIONS

The following precautions should be observed while performing the experiment:

In this experiment, we are handling the indicator in powder form, in contrast with most of all other basic experiments.

Handle the indicator powder carefully to get exact results.

Handling of the alkali, NaOH should be done with utmost care, since it causes irritation.

Since NaOH pellets are hygroscopic in nature, do not expose these pellets to air for a prolonged time period.

PROCEDURE

PREPARATION OF REAGENTS

Standard EDTA Solution (0.02 M)

1. Switch on the Electronic balance, keep the weighing pan, and set the reading to zero. Take 1000 mL of distilled water in a 1000 mL standard flask.
2. Weigh 3.723g of EDTA sodium salt.
3. Transfer the contents to the water sample. Place the lid and mix the contents thoroughly until all EDTA sodium salt dissolve in water.
4. Make the volume exactly 1000 mL by adding distilled water. Transfer the solution to a clean reagent bottle named EDTA solution.
5. This 0.02 molar EDTA solution is going to be used as a titrant in this experiment. Take this solution in a beaker for easy handling.

Ammonium purpurate

1. Weigh 0.5g of Ammonium Purpurate.
2. Transfer it to the dry beaker.
3. Weigh 100g of sodium chloride.
4. Transfer it to the beaker having ammonium purpurate mix the contents thoroughly. Use it as a dry powder.

Sodium Hydroxide (1N) solution

1. Take 100 mL of distilled water in a beaker.
2. Weigh 4 gm of Sodium hydroxide powder.
3. Transfer it to the distilled water in the beaker and mix it thoroughly.
4. Then transfer the entire content to 100 mL standard flask.
5. Rinse the glass rod and funnel with distilled water.
6. Make the volume 100 mL by adding distilled water up to the mark.
7. Take the sodium hydroxide solution in 100 mL beaker for easy handling.

TESTING OF SAMPLE

- Pipette 20 mL of water sample and transfer it to a clean 250 mL conical flask.
- Measure 2 mL of 1N sodium hydroxide solution using measuring cylinder. Add it to the water sample in conical flask so that the pH will be maintained between 12 and 13.
- Add few amount of Ammonium purpurate indicator to the water sample. Now the sample turns into pink color. This color change is due to the calcium and magnesium contents present in water.
- Before starting the titration rinse the burette with few mL of EDTA solution and discard it. Fill the burette with 0.02M EDTA solution. Adjust the reading to zero, then fix it in burette stand. Ensure that, there is no any air bubble inside the burette.
- Titrate the water sample against the EDTA solution in the burette till all calcium and magnesium ions present in the sample reacts with the EDTA to form a metal EDTA complex by changing the color of the sample to purple. i.e., the end point.

- Note down the burette reading
- The value of titration is 5.7 mL
- Repeat the titration for concordant values

CALCULATION

To determine Calcium Hardness in the given water sample, the readings are required to be tabulated.

Burette solution: EDTA

Pipette solution: Sample

Indicator: Ammonium Purpurate

End point: Appearance of Purple color.

TABLE

Sample No.	Volume of Sample (mL)	Burette Reading (mL)		Volume of EDTA (mL)
		Initial	Final	
1.				
2.				
3.				

DATA SHEET

DETERMINATION OF CALCIUM HARDNESS

Date Tested :

Tested By :

Project Name :

Sample Number :

Sample Location : Sample Description :

Sl.No.	Volume of Sample (mL)	Burette Reading (mL)		Volume of EDTA (mL)
		Initial	Final	
1.				
2.				
3.				

Calculation:

Volume of EDTA = mL
Normality of EDTA = N
Volume of Sample = mL
Equivalent weight of CaCO3 = 50

Calcium Hardness = Volume of EDTA * N * 50 * 1000 / Volume of sample taken

To convert the sample size from mL to L, multiply the result by 1,000

mL/L Calcium Hardness as $CaCO_3$ equivalent (mg/L) =

= **mg/L as $CaCO_3$ equivalent**

Calcium Present in the sample = Ca hardness in mg/L as $CaCO_3$* molecular weight of Ca

= mg/L

INTERPRETATION OF RESULTS

The Calcium Hardness of the given sample of water = **mg/L**

and Calcium ion concentration in the given sample of water = **mg/L**

INFERENCE

Calcium and magnesium ions are important contributors to water hardness. When water is heated, they break down and precipitate out of solution, forming scale. Maximum limits have been established. Magnesium concentrations more than 100 mg/L may have a laxative effect on some people.

EVALUATION

1. In the determination of hardness by titration, EDTA is used as
    a) Oxidizing Agent
    b) Chelating Agent
    c) Neutralizing Agent
    d) Binding Agent

2. As per drinking water standards in India, the limit of Mg in drinking water is
    a) 40 ppm
    b) 20 ppm
    c) 30 ppm
    d) 50 ppm

3. As per drinking water standards in India, the limit of Ca in drinking water is
    a) 75 ppm

b) 50 ppm
c) 100 ppm
d) 150 ppm

4. The other name of Ammonium Purpurate is_____.

a) Dioxide
b) Murexide
c) EBT
d) Trioxide

5. The presence of Ca, in the natural water is due to the passage through_____.

a) Lime Stone
b) Atomsphere
c) Stratosphere
d) Clouds

6. The temporary hardness of water is due to

a) Sulfate of calcium and magnesium
b) Chlorides of calcium and magnesium
c) Carbonate and bicarbonate of calcium and magnesium
d) Nitrates of calcium and magnesium

7. The permanent hardness of water is due to

a) Carbonate and bicarbonate of calcium and magnesium
b) Sulfate and bicarbonates of calcium
c) Chlorides and carbonates of magnesium
d) Sulfates, chlorides and nitrates of calcium and magnesium

8. Magnesium hardness with sulphate ions produce.

a) Cancer
b) Breathing problem
c) Laxative effect
d) Sleepiness

9. The role of NaOH in the titration is

a) NaOH reacts with Mg^{2+} ions and precipitates it out in the form of $Mg(OH)_2$
b) NaOH reacts with Ca^{2+} ions and precipitates it out in the form of

$Ca(OH)_2$

c) For pH Adjustment
d) For reducing the error

10. The calcium salts are useful for the growth of bones in human beings.

a) True
b) False

Ex.No.5 EXPERIMENT ON DETERMINATION OF RESIDUAL CHLORINE

PREAMBLE:

"**How to determine residual chlorine in Water and Wastewater**".

Test procedure is in accordance to IS: 3025 (Part 26) - Reaffirmed 2003.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

(1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 4500-Cl C.
(2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 330.2.

AIM

To determine residual chlorine in the given water sample with the stipulations as per **IS: 3025 (Part 26) - Reaffirmed 2003**.

INTRODUCTION

- Why we should measure residual chlorine?
- What do we mean by the term "Residual Chlorine"?
- What is the source or reason for presence of chlorine in water or waste water?
- Is it mandatory to measure residual chlorine in water or waste water?

Before learning what is residual chlorine and why we need to measure residual chlorine. Let us first understand the importance of adding chlorine.

Treated or filtered water is deemed to be fit for consumption only if it is devoid of disease producing microorganism.

Chlorination is primarily adopted to destroy or deactivate disease-producing microorganisms in the public water supplies and polluted rivers.

Chlorine is usually added to water in gaseous form or as sodium or calcium hypochlorite.

It has been practiced over several years.

When chlorine is added to water, some of the chlorine reacts first with organic materials and metals in the water and is not available for disinfection (this is called the chlorine demand of the water).

The remaining chlorine concentration after the chlorine demand is accounted for is called total chlorine.

Total chlorine is further divided into: 1) the amount of chlorine that has reacted with nitrates and is unavailable for disinfection which is called combined chlorine and, 2) the free chlorine, which is the chlorine available to inactivate disease- causing organisms, and thus a measure to determine the potability of water

The word "residual" means "remainder" or "that which is left", and as the name suggests the chlorine residual test is used to measure the amount of chlorine remaining in the water at the time the test is made.

The chlorine residual is usually tested in finished water which is ready to be released into the distribution system, although operators must also ensure that there is adequate residual at the extreme ends of the distribution system.

Although the pros and cons of disinfection with chlorine have been extensively debated, it remains the most widely used chemical for disinfection of water.

Excess Chlorination may produce adverse effects. Potentially carcinogenic chloroorganic compounds such as chloroform may be formed.

To fulfill the primary purpose of chlorination and to minimize any adverse effects, it is essential that proper testing procedures be used.

Several methods for measurement of total residual chlorine are available including iodometric methods, amperometric titration methods, and N,Ndiethyl-p- phenylenediamine (DPD) methods.

In this module we are going to learn Iodometric method of residual chlorine determination.

ENVIRONMENTAL SIGNIFICANCE

Chlorine residuals determination is used to control chlorination of domestic and industrial wastewaters.

Active chlorine (free and combined) should be determined at each stage in the treatment process of drinking water and in the water mains in order to guarantee bacteriologically impeccable water.

Chlorine determination is important to avoid bad odour and change in the taste of water. It is determined in the swimming pools to avoid ill effects due to excess chlorination.

Determination of chlorine residual in water distribution is useful to find the source of contamination or leakage points, so as to supply wholesome water to the consumer.

Thus, the main purpose for the chlorination of water supplies and polluted waters serves primarily to destroy or deactivate disease-producing micro-organisms.

PRINCIPLE

The starch-iodide titration method, one of the oldest methods for determining chlorine, is very non-specific for oxidants and generally is used for total chlorine testing at levels above 1 mg/L Cl_2.

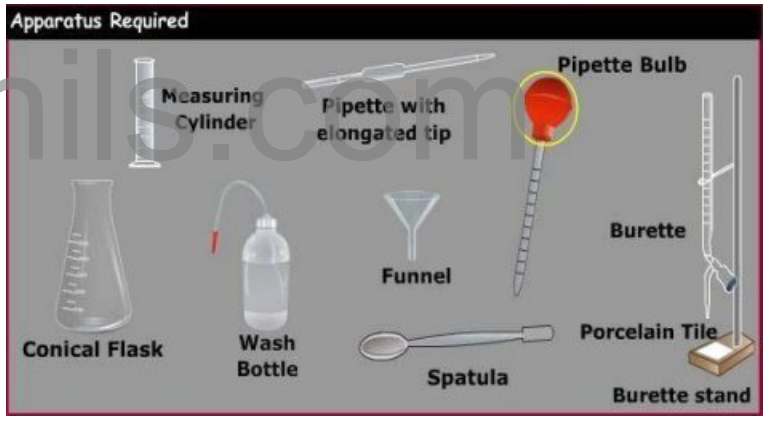
Chlorine will liberate free iodine from potassium iodide (KI) solutions at pH 8 or less. The liberated iodine is titrated with a standard solution of sodium thiosulphate ($Na_2S_2O_3$) with starch as the indicator.
This method is based on reaction with thiosulfate solution

The end point of the titration is indicated by the disappearance of the blue-colored, starch-iodide complex.

MATERIALS REQUIRED

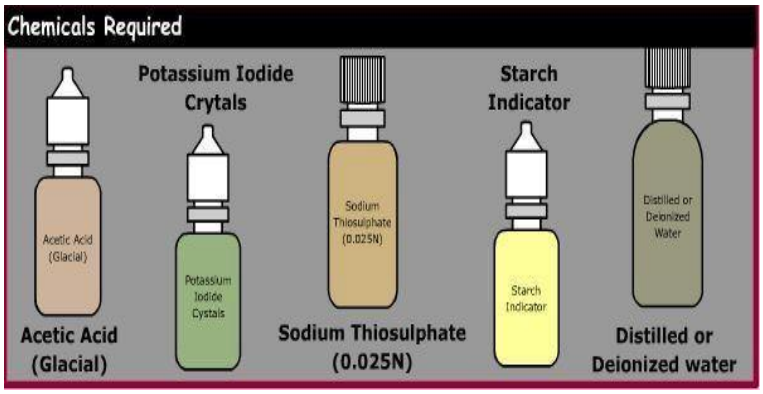
APPARATUS REQUIRED

1. Burette & Burette Stand
2. Porcelain Tile
3. Pipettes with elongated tips
4. Pipette Bulb
5. Wash Bottle
6. 250 mL Graduated Cylinder
7. 500 mL Conical Flask (Erlenmeyer flask)

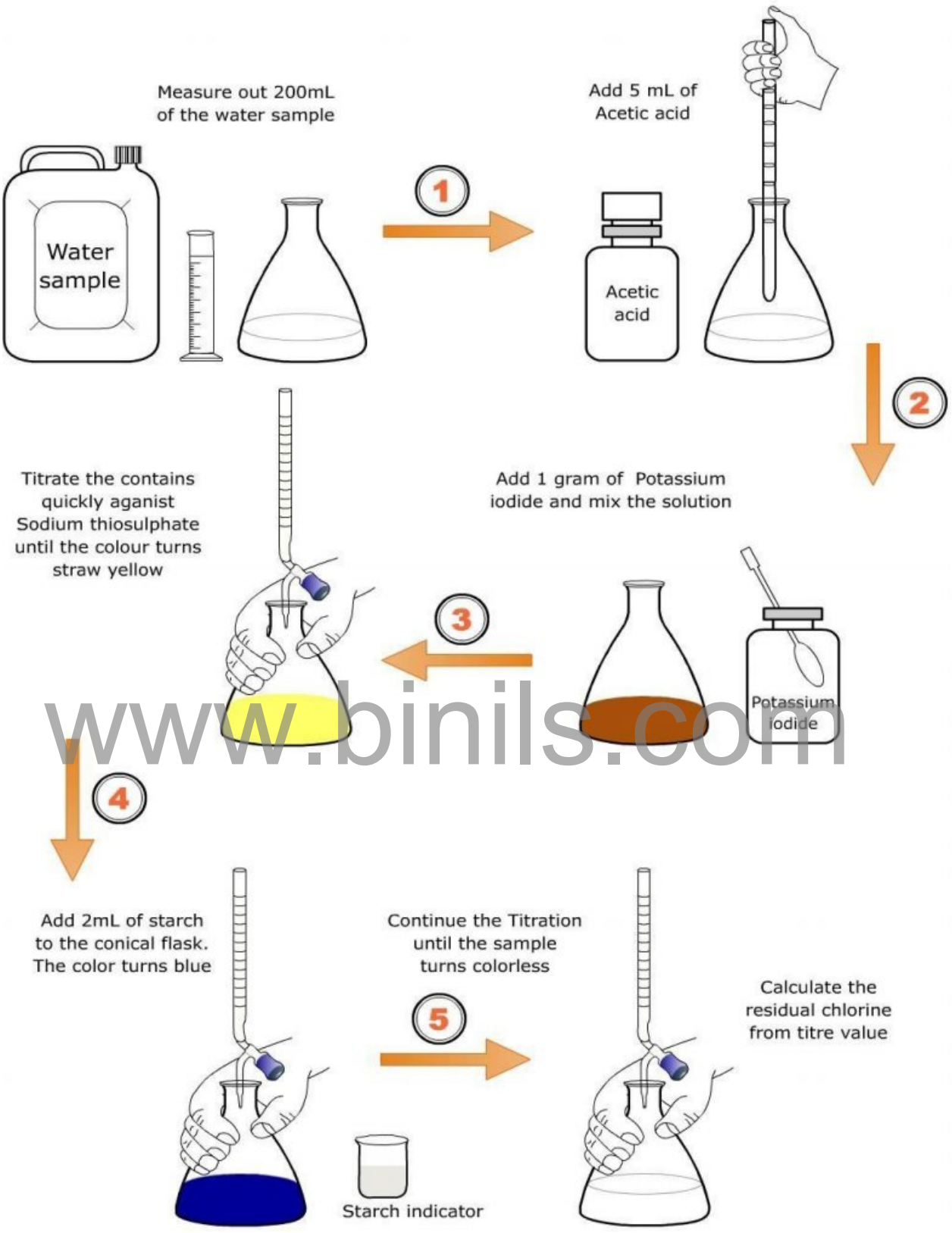


CHEMICALS REQUIRED

1. Acetic Acid, Conc. (glacial)
2. Potassium Iodide, KI, crystals
3. Sodium thiosulphate
4. Starch indicator
5. Distilled or Deionized Water



PROCEDURE CHART



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 with in two hours of collection, cool storage is not necessary. If analysis can not be started with in the two hours of sample collection to reduce the change in sample, keep all samples at 4^0C.

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

PRECAUTIONS

The following precautions should be observed while performing the experiment:

- This experiment is a basic experiment and hence there will not be any major difficulties in performing the experiment. The entire procedure should be done in quick time without exposing the solutions to the ambient air.
- Do not expose the potassium iodide crystals in the air. If possible do the experiment in iodine flask instead of conical flask.
- Chlorine in water solutions is not stable. As a result, its concentration in samples decreases rapidly.
- Samples to be analyzed for chlorine cannot be stored or preserved. Tests must be started immediately after sampling. Therefore, samples taken for the chlorine residual test must be grab samples only and excessive agitation must be avoided.
- Exposure to sunlight or other strong light, air, or agitation will further reduce the quantity of chlorine present in solutions.

PROCEDURE

They are two different methods are available to estimate the residual chlorine as per our IS Code.

1) **Iodometric Method:** This method is more precise than colorimetric method where residual concentration exceeds 1mg/L, but for lower concentration it is not so accurate.
2) **Stabilized Neutral Ortho-Toluidine method:** This method is useful to determine free available chlorine and combine chlorine. This method is sensitive to low residual chlorine concentrations.

PREPARATION OF REAGENTS

Sodium Thiosulphate solution (0.01N)

Weigh approximately 2.482 g of sodium thiosulphate $(Na_2S_2O_35H_2O)$.
Transfer to the beaker and dissolve it in boiled distilled water.
Transfer it to the standard flask and make it up to 1000 mL.

TESTING OF SAMPLE

- Rinse the burette with sodium thiosulphate and then fill the burette with sodium thiosulphate.
- Fix the burette to the stand.
- Take 200 mL of a given sample in a conical flask.
- Add 5 mL Acetic acid. To acidify the sample. It is used to reduce the pH between 3 and 4 in the conical flask.
- Add about 1 g Potassium Iodide (KI) measured using the spatula and dissolve it by thoroughly mixing it with stirring rod.
- Perform the titration quickly, since iodine liberate faster.
- Titrate the solution with standard Na2S2O3 solution until the yellow color of liberated Iodine is almost faded out. (Pale yellow color)
- Add 1 mL of starch solution and continue the titration until the blue color disappears.
- In many cases residual chlorine is very low and starch needs to be added before starting up the titration.
- Note down the burette reading (to know the volume of sodium thiosulphate added).
- The reading is 10.6 mL

CALCULATION

To determine Residual Chlorine in the given water sample, the readings are required to be tabulated.

TABLE

Sample No.	Temperature of Sample (ºC)	Volume of Sample (mL)	Burette Reading (mL)		Volume of Titrant (mL) ($Na_2S_2O_3$ solution used)	Residual Chlorine (mg/L)
			Initial	Final		

Burette Solution: Sodium

Thiosulphate Pipette Solution:

Sample

Indicator: Starch

End point : Disappearance of blue color

DATA SHEET

DETERMINATION OF RESIDUAL CHLORINE

Date Tested :

Tested By :

Project Name :

Sample Number :

Sample Location :

Sample Description :

TABULATION

Sample No	**Volume of Sample (mL)**	**Burette Reading (mL)**		**Volume of Titrant (mL)** ($Na_2S_2O_3$ solution used)
		Initial	**Final**	
1.				
2.				
3.				

Calculation:

Volume of Sodium thiosulphate V_1 = mL

Normality of Sodium thiosulphate N_1 = N

Volume of Sample V_2 = mL

Equivalent weight of Chlorine = 35.45

$$\text{Residual Chlorine} = \frac{\text{Volume of Sodium thiosulphate} * N * 35.45 * 1000}{\text{Volume of sample taken}}$$

To convert the sample size from mL to L, multiply the result by 1,000 mL/L

Residual Chlorine (mg/L) =

= **mg/L**

INTERPRETATION OF RESULTS

The amount of residual chlorine in the given sample of water = **2.3 mg/L**

INFERENCE

Sample provided for the experiment is obtained from over head tank supplied by the government agency. Residual chlorine is the water measured to be 2.3 mg/L. It is more than permissible amount. Active chlorine should be present at each stage of water treatment and distribution. The residual chlorine at the consumers end should be 0.2 mg/l. Presence of excessive chlorine gives bad odour and taste and is harmful also. It may lead to cancer, skin and eye irritation. To avoid the excess chlorination, water sample need to boiled before the domestic use.

EVALUATION

1. Disinfection means
    a) Killing of disease producing bacteria and other microorganisms
    b) Killing of all bacteria and other microorganisms
    c) Removing infection from water
    d) Removing sewage from water

2. Bleaching powder is mixed in water for
    a) Making it clean
    b) Disinfection of water
    c) Adjusting its pH
    d) Making it soft water

3. Residual chlorine means the Chlorine
    a) required for the disinfection of water normally
    b) required for the disinfection of water in the rainy season
    c) available after completion of the disinfection
    d) required as the superchlorination

4. Potable water is
    a) tasty water
    b) wholesome water
    c) Mineral water
    d) water free from disease producing elements and bacteria.

5. The amount of residual chlorine in water should be

a) 0.2 mg/litre
b) 2.0 mg/litre
c) 2.5 mg/litre
d) 4.0 mg/litre

6. Residual chlorine is detected in water by_____.

a) Erichrome black T
b) Bleaching powder
c) Methyl orange
d) Orthotolidine

7. Chlorine is often added to wastewater for disinfection before effluent discharge A potential problem with this procedure is

a) Toxic chlorinated hydrocarbons may be formed
b) Chlorine contributes to depletion of the ozone layer
c) Chlorine gas is poisonous and may threaten nearby homes
d) chlorine is a nonrenewable resource and may soon be depleted

8. Water requires_____mg/L of chlorine to destroy the microorganisms present.

a) 2
b) 3
c) 4
d) 1

9. Chlorination is effective if the pH is_____. a)

7.2 to 8.5
b) 6.8 to 7.2
c) 6 to 7
d) Neutral

10. Titration should be carried out with

a) Neutral pH
b) pH 10 - 11
c) pH 3 - 4

Ex. No. 6 EXPERIMENT ON DETERMINATION OF ALKALINITY OF WATER

PREAMBLE:

"**How to determine alkalinity in Water and Wastewater**".

Test procedure is in accordance to IS: 3025 (Part 23) - Reaffirmed 2003.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

(1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 2320.
(2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 310.1.

AIM

To determine the alkalinity of given water sample with the stipulations as per **IS: 3025 (Part 23) - Reaffirmed 2003.**

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 cations 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 mixed indicator, the presence of CO_3^{2-} and HCO_3^- ions in the solution changes the colour to blue. While adding sulphuric acid, the color changes to red, this color change indicates that all the CO_3^{2-} and HCO_3^- ions has been neutralized. This is the end point.

MATERIALS

REQUIRED

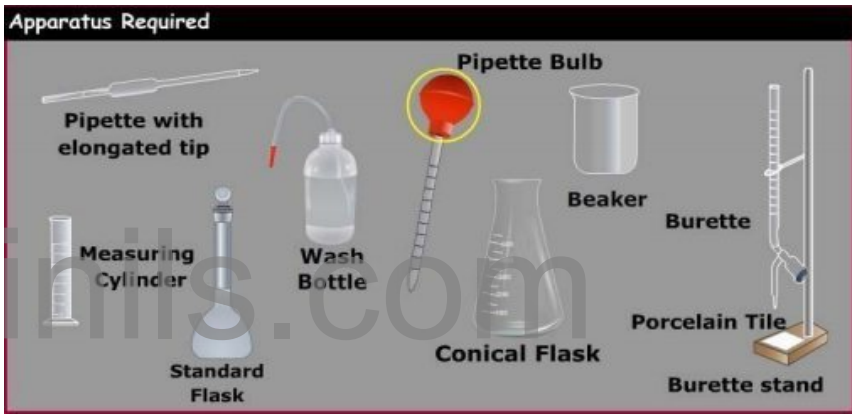
APPARATUS

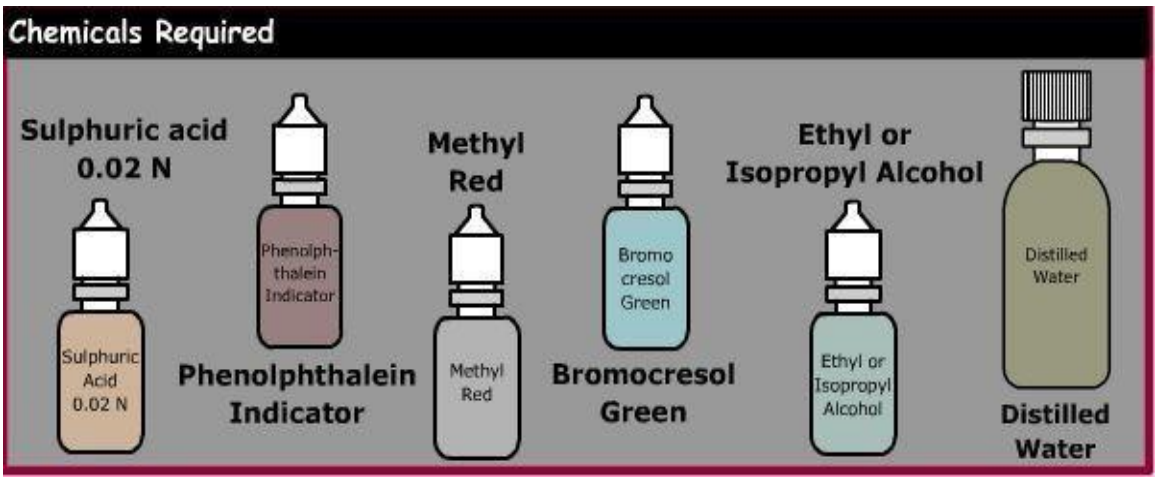
REQUIRED

1. Burette with Burette stand and porcelain title
2. Pipettes with elongated tips
3. Pipette bulb
4. Conical flask (Erlenmeyer Flask)
5. 250 mL Measuring cylinders
6. Standard flask
7. Wash Bottle
8. Beakers

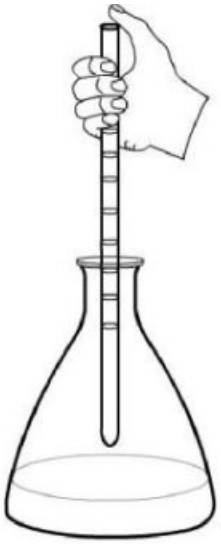
CHEMICALS REQUIRED

1. Standard sulphuric acid
2. Phenolphthalein
3. Mixed Indicator
4. Bromocresol Green
5. Methyl Red
6. Ethyl alcohol
7. Distilled Water

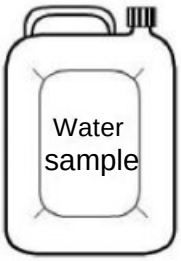




PROCEDURE CHART

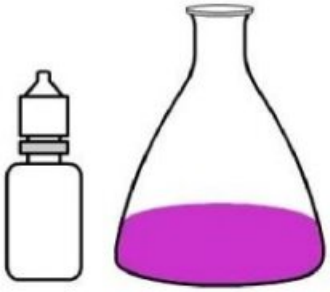


Pipette out 100mL of the field sample

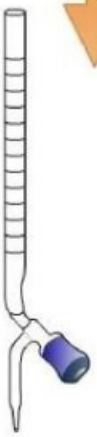


1

Add 2 drops of Phenolphthalein indicator



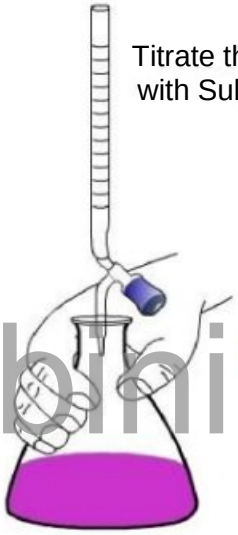
2



Fill the burrette with Sulphuric Acid

3

Titrate the contents with Sulphuric Acid

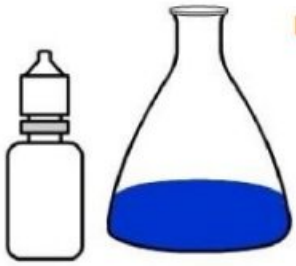


4

Continue the titration till the color changes to colourless

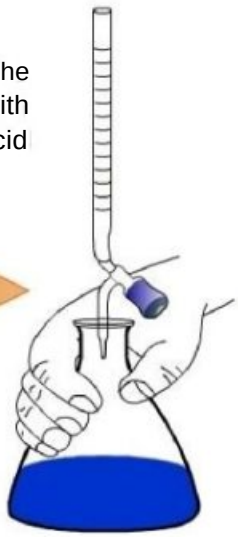
5

Add 2 drops of mixed indicator



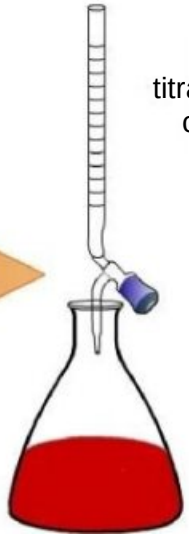
6

Continue the titration with sulphuric acid



7

Continue the titration till the color changes to red



Calculate the Alkalinity of the sample

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.

PRECAUTIONS

The following precautions should be observed while performing the experiment:

1. Do not keep the indicator solution open since it contains the alcohol which tends to evaporate.
2. The mixed indicator solution is containing dye in it; care should be taken so that it is not spilled to your skin.
3. If it spills on your skin, the scar will remain at least for two to three days.

PROCEDURE

PREPARATION OF REAGENTS

For testing the given sample, first the reagents are required to be prepared.

Sulphuric Acid Solution (0.02N):

- Take approximately 500 mL of distilled water in a 1000 mL standard flask.
- Pipette 20 mL of concentrated 0.1 Normality Sulphuric acid and add slowly along the sides of the standard flask.
- Then make up the volume up to 1000 mL mark. Now the strength of this solution is 0.02 N.

Phenolphthalein Indicator Preparation:

- Weigh 1g of phenolphthalein and add to 100 mL of 95% ethyl alcohol or to 100 mL of distilled water. Use the readymade Phenolphthalein indicator available in the market.

Mixed Indicator Preparation:

- Dissolve 100 mg Bromocresol green and 20 mg of methyl red in 100 mL of 95% ethyl alcohol or use 100 mL of distilled water. Mixed indicator also readily available in the market. So it can be used as indicator in this experiment.

TESTING OF WATER SAMPLE

- Rinse the burette with 0.02N Sulphuric acid and discard the solution.
- Fill the burette with 0.02N sulphuric acid and adjust it to zero.
- Fix the burette in the stand.
- Using a measuring cylinder exactly measure 100 mL of sample and pour it into a 250 mL of conical flask.
- Add few drops of phenolphthalein indicator to the contents of conical flask. The colour of the solution will turn to pink. This colour change is due to alkalinity of hydroxyl ions in the water sample.
- Titrate it against 0.02N sulphuric acid till the pink color disappears. This indicates that all the hydroxyl ions are removed from the water sample. Note down the titter value (V1). The value of titration is 0.5mL .This value is used in calculating the phenolphthalein alkalinity.
- To the same solution in the conical flask add few drops of mixed indicator. The colour of the solution turns to blue. This colour change is due to CO_3^{2-} & HCO^- ions in watersample.
- Continue the titration from the point where stopped for the phenolphthalein alkalinity. Titrate till the solution becomes red. The entire volume (V2) of sulphuric acid is noted down and it is accountable in calculating the total alkalinity.

- The value of titration is 8.3mL.
- Repeat the titration for concordant values.

CALCULATI

ON TABLE

Table -1 Phenolphthalein Alkalinity:

Sl.No.	Volume of Sample (mL)	Burette Reading (mL)		Volume of Sulphuric acid (mL)
		Initial	Final	
1.				
2.				
3.				

Burette solution: Sulphuric Acid Solution

Pipette solution: Sample.

Indicator: Phenolphthalein Indicator.

End point: Disappearance of pink color.

Table - 2 Total Alkalinity:

Sl.No.	Volume of Sample (mL)	Burette Reading (mL)		Volume of Sulphuric acid (mL)
		Initial	Final	
1.				
2.				
3.				

Burette solution: Sulphuric Acid Solution

Pipette solution: Sample.

Indicator: Mixed Indicator.

End point: Appearance of Red color.

DETERMINATION OF ALKALINITY

DATA SHEET

Date Tested :

Tested By :

Project Name : Sample Number :

Sample Location : Sample Description :

Sl.No.	Volume of Sample (mL)	Burette Reading (mL)		Volume of Sulphuric acid (mL)
		Initial	Final	

Calculation:

Volume of Sulphuric Acid = mL
Normality of Sulphuric Acid = N
Volume of Sample = mL
Equivalent weight of $CaCO_3$ = 1000

$$\text{Phenolphthalein Alkalinity} = \frac{(\text{volume of } H_2SO_4(v1) * \text{Normality} * 50 * 1000)}{\text{Volume of sample taken}}$$

To convert the sample size from mL to L, multiply the result by 1,000 mL/L to convert the sample size from mL to L

Alkalinity as $CaCO_3$ equivalent (mg/L) **=** mg/L as $CaCO_3$ equivalent

Total Alkalinity:

Sl.No.	Volume of Sample (mL)	Burette Reading (mL)		Volume of Sulphuric acid (mL)
		Initial	Final	
1				
2				
3				

Calculation:

Volume of Sulphuric Acid = 8.3 mL
Normality of Sulphuric Acid = 0.02 N
Volume of Sample = 50 mL
Equivalent weight of $CaCO_3$ = 1000

$$\text{Total Alkalinity} = \frac{(\text{volume of } H_2SO_4(v1) * \text{Normality} * 50 * 1000)}{\text{Volume of sample taken}}$$

To convert the sample size from mL to L,

multiply the result by 1,000 mL/L to convert the sample size from mL to L

Alkalinity as $CaCO_3$ equivalent (mg/L) =

= **mg/L as $CaCO_3$ equivalent**

INTERPRETATION OF RESULTS

Value of P and T	Alkalinity due to		
	OH	CO_3^{2-}	HCO_3^-
P=0	0	0	T
P< ½ T	0	2P	T-2P
P= ½ T	0	2P	0
P> ½ T	2P-T	2P-T	0
P=T	T	0	0

- To find the different values of Alkalinity due to Hydroxyl, Carbonate and Bicarbonate ions take P as Phenolphthalein Alkalinity and T as Total Alkalinity.

- If P=0, The Alkalinity due to Hydroxyl and carbonate ions is 0. Alkalinity due to Bicarbonate ion is equal to the Total Alkalinity

- If P < ½ ,T then the Alkalinity due to Hydroxyl ion is 0. The Alkalinity due to carbonate ion is 2P. i.e. 2P = 10 mg/L. Alkalinity due to Bicarbonate ion is equal to the Total Alkalinity minus 2 times Phenolphthalein Alkalinity

- If P = ½ ,T then the Alkalinity due to Hydroxyl ion is 0. The Alkalinity due to carbonate ion is 2P. Alkalinity due to Bicarbonate ion is equal to 0

- If P > ½ ,T then the Alkalinity due to Hydroxyl and carbonate ions is 2P- T. Alkalinity due to Bicarbonate ion is 0

- If P=T, The Alkalinity due to Hydroxyl is equal to the Total Alkalinity. Alkalinity due to carbonate and Bicarbonate ions is 0.

- If P > ½, T then the Alkalinity due to Hydroxyl and carbonate ions is 2P–T. Alkalinity due to Bicarbonate ion is 0. If P = T, The Alkalinity due to Hydroxyl is equal to the Total Alkalinity. Alkalinity due to carbonate and Bicarbonate ions is 0.

INFERENCE

Alkalinity is a measure of the capacity of water to neutralize acids. The predominant chemical system present in natural waters is one where carbonates, bicarbonates and hydroxides are present. The bicarbonate ion is usually prevalent. However, the ratio of these ions is a function of pH, mineral composition, temperature and ionic strength. Water may have a low alkalinity rating but a relatively high pH or vice versa, so alkalinity alone is not of major importance as a measure of water quality. Alkalinity is not considered detrimental to humans but is generally associated with high pH values, hardness and excess dissolved solids. High alkalinity waters may also have a distinctly flat, unpleasant taste. Based on the testing, it is found that the alkalinity of the sample is 83 mg/L. As per the provisional code, alkalinity should not exceed 200 mg/L for potable water. For the fresh water alkalinity ranges between 20 – 100 mg/L. Alkalinity of tested sample is within the limits specified in the standards. Hence the water sample is fit for drinking.

EVALUATION

1. Alkalinity of water is an indication of
    a) Base neutralizing capacity
    b) Acid neutralizing capacity
    c) Quantity of base present
    d) Quality of base present

2. Mixed indicator is a combination of
    a) Bromcresol Blue and Methyl Orange
    b) Bromcresol Green and Methyl Red
    c) Bromcresol Blue and Methyl Red
    d) Bromcresol Green and Methyl Orange

3. Alkalinity is present due to all except .
    a) Bromates
    b) Phosphates
    c) Silicates
    d) Chlorides
4. Alkalinity is not caused by
    a) Carbonates ions
    b) Bicarbonates ions
    c) Hydroxyl ions
    d) Chloride ions

5. The phenolphthalein alkalinity is present then the pH of that water will be More than

a) 8.3
b) 9.3
c) 7.3
d) 6.3

6. Alkalinity of natural water is mainly due to the presence of_____.

a) Bicarbonates
b) Bromates
c) Phosphates
d) Silicates

7. The bicarbonate equivalence point normally occur at

pH a) 2.5
b) 3.5
c) 4.5
d) 5.5

8. What is ppm?

a) Parts per meter square
b) Parts per meter
c) Parts per million
d) Parts per millimeter

9. The normality of the acid used in the titration is____.

a) 0.2 N
b) 0.02 N
c) 0.002 N
d) 2.0 N

10. A standard solution is a

a) Solution of accurately known strength
b) Solution of accurately known pH
c) Coloured solution
d) Colourless solution

Ex. No. 7 EXPERIMENT ON DETERMINATION OF CHLORIDES

PREAMBLE:

"**How to determine chlorides in Water and Wastewater**".

Test procedure is in accordance to IS: 3025 (Part 32) - Reaffirmed 2003.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

(1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 4500 - Cl^- - B.
(2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 9253.

AIM

To determine the chlorides of given water sample with the stipulations as per **IS: 3025 (Part 32) - Reaffirmed 2003.**

INTRODUCTION

Chlorides are widely distributed as salts of calcium, sodium and potassium in water and wastewater. In potable water, the salty taste produced by chloride concentrations is variable and dependent on the chemical composition of water. The major taste producing salts in water are sodium chloride and calcium chloride. The salty taste is due to chloride anions and associated cations in water. In some water which is having only 250 mg /L of chloride may have a detectable salty taste if the cat-ion present in the water is sodium. On the other hand, a typical salty taste may be absent even if the water is having very high chloride concentration for example 1000 mg /L.

This is because the predominant cation present in the water is not sodium but either calcium or magnesium may be present.

ENVIRONMENTAL SIGNIFICANCE

- Chlorides associated with sodium (Sodium Chloride) exert salty taste when its concentration is more than 250 mg/L. These impact a salty taste to water. Chlorides are generally limited to 250 mg/L in water supplies intended for public water supply. In many areas of the world where water supplies are scarce, sources containing as much as 2000 mg/L are used for domestic purposes without the development of adverse effect, once the human system becomes adapted to the water.

- It can also corrode concrete. Magnesium chloride in water generates hydrochloric acid after heating which is also highly corrosive and creates problem in boilers.
- Chloride determinations in natural waters are useful in the selection of water supplies for human use.
- Chloride determination is used to determine the type of desalting apparatus to be used.
- Chloride determination is used to control pumping of ground water from locations where intrusion of seawater is a problem.
- Chlorides interfere in the determination of chemical oxygen demand (COD).

PRINCIPLE

The amount of chloride present in water can be easily determined by titrating the given water sample with silver nitrate solution.

The silver nitrate reacts with chloride ion according to1 mole of $AgNO_3$ reacts with 1 mole of chloride. The titrant concentration is generally 0.02 M.

Silver chloride is precipitated quantitatively, before red silver chromate is formed. The end of titration is indicated by formation of red silver chromate from excess silver nitrate.

The results are expressed in mg/L of chloride (Cl^- with a molecular weight of 35.453 g/mol).

MATERIALS

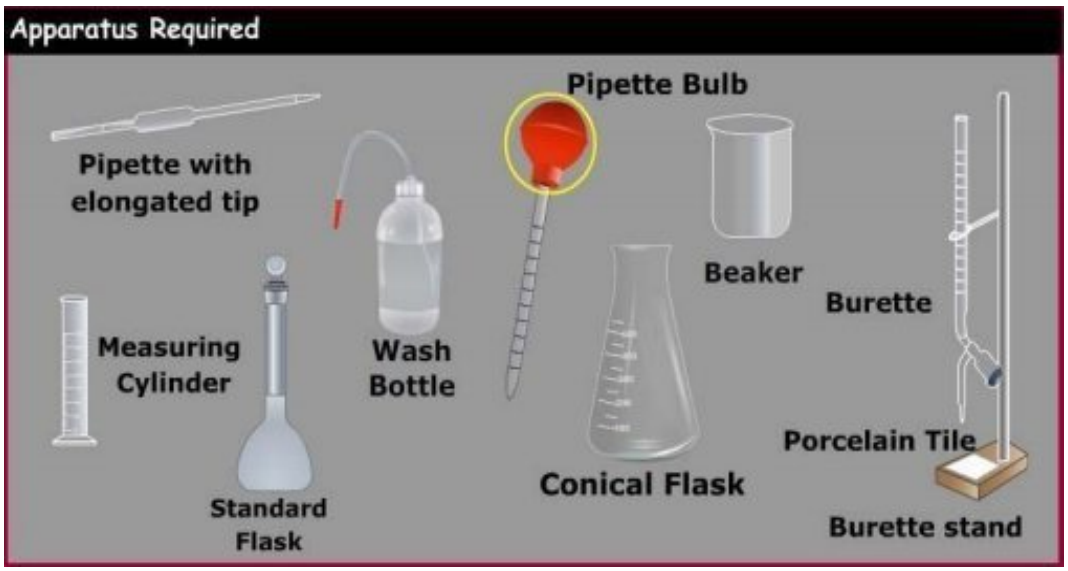
REQUIRED APPARATUS

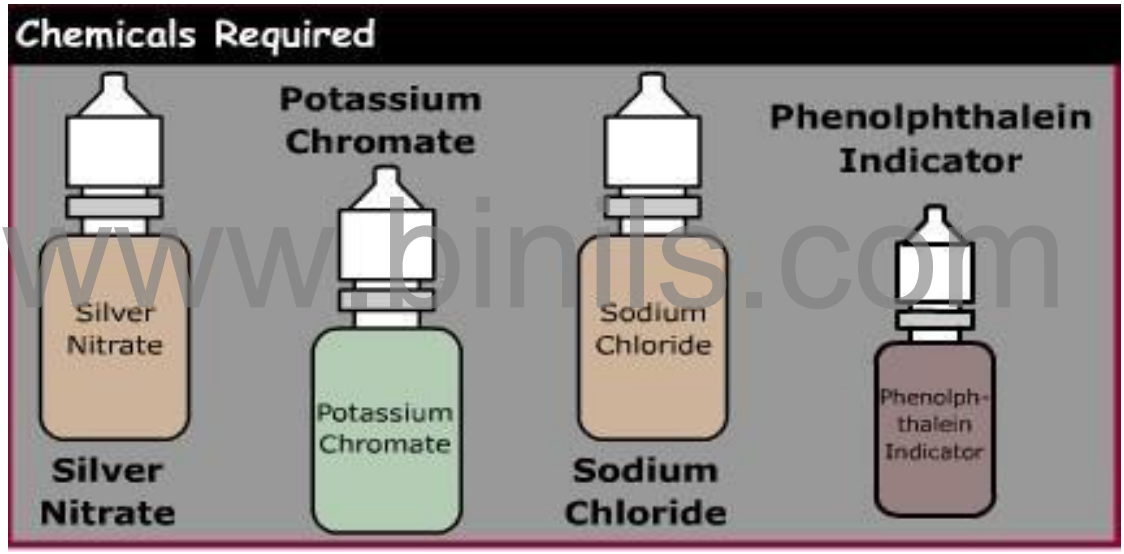
REQUIRED

1. Burette with Burette stand and porcelain tile
2. Pipettes with elongated tips
3. Conical flask (Erlenmeyer Flask)
4. Standard flask
5. Beaker
6. Wash bottle

CHEMICALS REQUIRED

1. Silver nitrate
2. Phenolphthalein Indicator
3. Sodium chloride
4. Potassium chromate





PROCEDURE CHART

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 with in two hours of collection, cool storage is not necessary. If analysis can not be started with in the two hours of sample collection to reduce the change in sample, keep all samples at 4^0C.

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

PRECAUTIONS

- $AgNO_3$ should be stored in a brown amber bottle and should not be exposed to sunlight.
- While handling $AgNO_3$, care should be taken so that it is not spilled on your skin.
- If it spills on your skin, the scar will remain at least for ten to fifteen days.

PROCEDURE

PREPARATION OF REAGENTS

Standard Sodium Chloride Solution

- Switch on the Electronic balance, keep the weighing pan, and set the reading to zero.
- Weigh 1.648g of Sodium chloride
- Transfer the contents to the beaker containing distilled water. Using glass rod, dissolve the contents thoroughly.
- Transfer the contents in the beaker to a 100 mL standard flask; fill distilled water up to 100 mL mark.
- Transfer it to 100mL standard flask using funnel

Standard Silver Nitrate (0.0282 N)

- Weigh 4.791g of Silver nitrate and transfer it to the beaker with distilled water.
- Transfer the contents in the beaker to a 100 mL standard flask, fill distilled water up to 100 mL mark.
- Standardize it against 0.0282 N NaCl solution. Store it in an amber bottle.

Potassium Chromate Indicator

- Weigh 25 g of Potassium Chromate. Transfer it to the beaker contains distilled water. Add few drops of Silver Nitrate solution until slight red precipitate is formed.
- Allow it to stand for 12 hours. After 12 hours filter the solution using filter paper and dilute the filtrate to 1000 mL using distilled water.

TESTING OF WATER SAMPLE

- Before starting the titration rinse the burette with silver nitrate solution. Fill the burette with silver nitrate solution of 0.0282 N. Adjust to zero and fix the burette in stand.
- Take 20 mL of the sample in a clean 250mL conical flask
- Add 1 mL of Potassium Chromate indicator to get light yellow color
- Titrate the sample against silver nitrate solution until the color changes from yellow to brick red. i.e., the end point.
- Note the volume of Silver nitrate added **(A).**
- The value of titration is 3.3 mL.
- Repeat the procedure for concordant values.

Blank Titration

- Take 20 mL of the distilled water in a clean 250mL conical flask
- Add 1 mL of Potassium Chromate indicator to get light yellow color
- Titrate the sample against silver nitrate solution until the color changes from yellow to brick red. i.e., the end point.
- Note the volume of silver nitrate added for distilled water (B).
- The value of titration is 0.2 mL

CALCULATION

TABLE

Sample No	Volume of Sample (mL)	Burette Reading (mL)		Volume of $AgNO_3$ (mL)
		Initial	Final	
1.				
2.				
Blank (B)				

Burette solution: Silver Nitrate **Pipette solution**: Sample **Indicator**: Potassium chromate

End point: Appearance of Brick red color.

DATA SHEET

DETERMINATION OF CHLORIDES

Date Tested :

Tested By :

Project Name :

Sample Number :

Sample Location : Sample Description :

Sl.No.	Volume of Sample (mL)	Burette Reading (mL)		Volume of EDTA (mL)
		Initial	Final	
1.				
2.				
Blank (B)				

Specimen Calculation:

Volume of Silver Nitrate for sample (V_S) = mL

Volume of Silver Nitrate for Blank (V_B) = mL

Normality of EDTA = N

Volume of Sample = mL

Equivalent weight of Chlorine = 35.45

Chlorides mg/ L = $\frac{(V_S - V_B) * \text{Normality} * 35.45 * 1000}{\text{Volume of sample taken}}$

To convert the sample size from mL to L, multiply the result by 1,000 mL/L

Chlorides mg/ L =

INTERPRETATION OF RESULTS

The amount of chloride present in the given water sample is = **155 mg/L.**

INFERENCE

The high concentrations of chloride ions mostly results in an unpleasant salty taste of water and it also aides the corrosion of plumbing system. Very high chloride content of water may also produce laxative effect. An upper limit of 250 mg/L has been set for the chloride ions. An increase in the normal chloride content of your water may indicate possible pollution from human sewage, animal manure or industrial wastes. As all aware the sea water is full of sodium chloride, the chloride levels will be much higher compared to the fresh water sources.

EVALUATION

1. The limit of chlorides in drinking water as per IS code is

    a) 200 ppm
    b) 225 ppm
    c) 250 ppm
    d) 500 ppm

2. Silver nitrate is stored in a brown bottle

    a) to avoid decomposition by sun light
    b) because it is dark in colour
    c) because the solution is colourless
    d) to avoid heat

3. The colour of Silver Chromate is

    a) Milky White
    b) pale Yellow
    c) Colourless
    d) Brick Red

4. When both hardness and chloride content are very high above 500 mg/L, then the water will be

    a) Non salty in nature
    b) Fit for drinking
    c) Salty in nature
    d) Soft water

5. Presence of chloride can corrode_____.

    a) GI pipes
    b) Rubber tubes
    c) PVC pipes
    d) Glass pipes

6. The chloride concentration in sewage is

    a) More concentrated than the municipal water supplied
    b) Equal concentration to the municipal water supplied
    c) Less concentrated than the municipal water supplied
    d) Only in trace

7. Chloride consumed by human beings

    a) Pass through the fecal matter as it is
    b) Gets changed into other forms
    c) Gets disappeared in the body
    d) Stored in bones

8. Chloride gives salty taste to water particularly when present as____.

    a) Sodium chloride
    b) Magnesium chloride
    c) Potassium chloride
    d) Zinc chloride

9. The point at which a clear visual change is observed after the reaction between titrant and titrates is called

    a) End point
    b) Equivalence point
    c) Equal point
    d) Double equivalence point

10. Most common ion in the water is

    a) Fluoride
    b) Nitrate
    c) Chloride
    d) Sulphate

Ex.No.8 EXPERIMENT ON DETERMINATION OF SULPHATES

PREAMBLE:

"**How to determine Sulphates in Water and Wastewater**".

Test procedure is in accordance to IS: 3025 (Part 24) - Reaffirmed 2003.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

(1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 4500 E.
(2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 375.4.

AIM

To determine the sulphates in the given water sample with the stipulations as per **IS: 3025 (Part 24) - Reaffirmed 2003.**

INTRODUCTION

Sulphates is widely distributed in nature and may be present in natural waters in concentration ranging from few hundred to several thousand mg/L. Sulphates occur naturally in numerous minerals, including barite, epsomite and gypsum. These dissolved minerals contribute to the mineral content of drinking-waters.

Acid Mine Drainage (AMD) may contribute large amounts of sulphates through pyrite oxidation. Sulfate is the second most abundant anion in seawater. Its high concentration owes to the high to moderate solubility of the salts that it forms with the major cations in seawater, namely, Na, Mg^{2+}, and Ca^{2+}.

ENVIRONMENTAL SIGNIFICANCE

- Sulphates are of considerable concern because they are indirectly responsible for two serious problems often associated with the handling and treatment of wastewater. They are odour and sewer corrosion problem result from the reduction of sulphates to hydrogen sulphide under anaerobic conditions.
- The amount of sulphates in wastewater is a factor of concern in determining the magnitude of problems that can arise from reduction of sulphates to hydrogen sulphide. For example knowledge of the sulphates content of the sludge or waste fed to digestion units provides a means of estimating the hydrogen sulphide content of the gas produced. From this

information, the design engineer can determine whether scrubbing facilities will be needed to remove hydrogen sulphide and size of the units required.

PRINCIPLE

The turbidimetric method of measuring sulphates is based upon the fact that barium sulphates tends to precipitate in a colloidal form of uniform size and that this tendency is enhanced in presence of a sodium chloride, hydrochloric acid and glycerol.

$$SO_4^{2-} + BaCl_2 \longrightarrow BaSO_4$$

The absorbance of the barium sulphates formed is measured by a spectrophotometer at 420 nm and the sulphates ion concentration is determined by comparison of the reading with a standard curve.

MATERIALS

REQUIRED

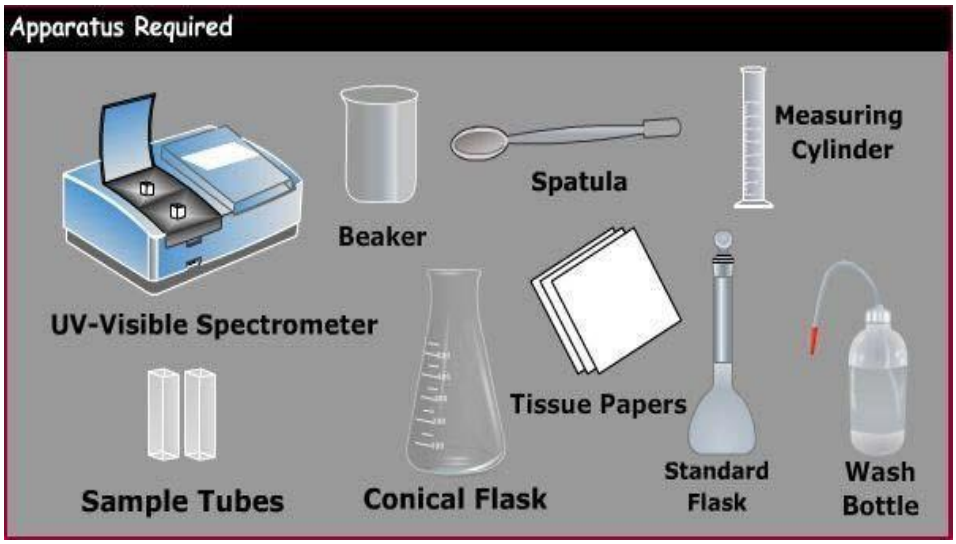
APPARATUS

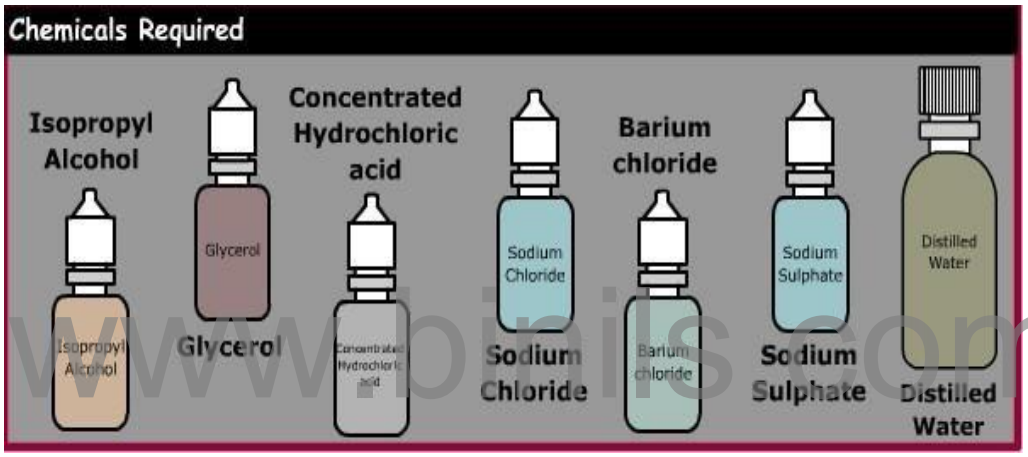
REQUIRED

1. UV-Visible Spectrometer
2. Sample tubes
3. Standard flask
4. Beaker
5. Spatula
6. Measuring Cylinder
7. Wash Bottle
8. Tissue Paper

CHEMICALS REQUIRED

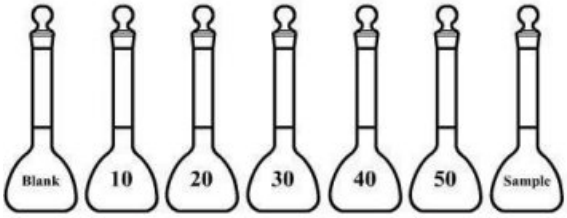
1. Isopropyl Alcohol
2. Glycerol
3. Concentrated Hydrochloric acid
4. Sodium Chloride
5. Barium chloride
6. Sodium sulphate
7. Distilled water





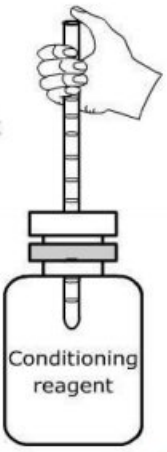
PROCEDURE CHART

Prepare a series of standards
10, 20, 30, 40, 50, a blank
(distilled water)
and known volume of sample



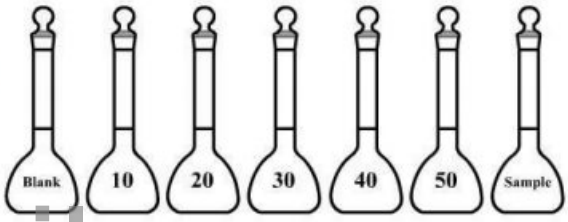
1

Add 5mL of
Conditioning
reagent to all the flask



2

Make of the volume to 100mL
using distilled water

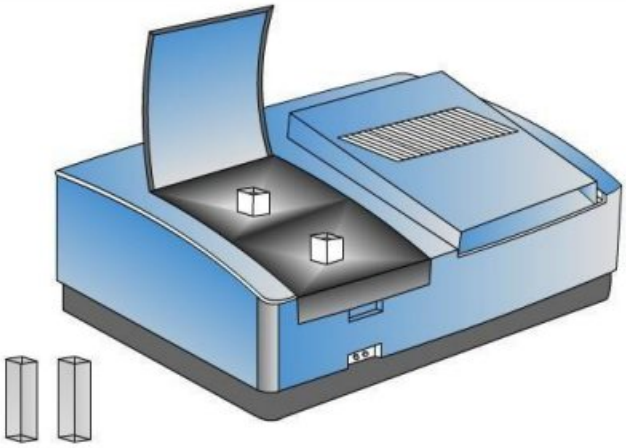


3

Add a pinch of
Barium chloride
to all the flask

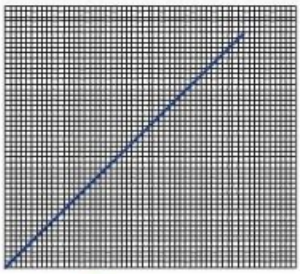
4

Immediately measure
the turbidity
using UV-Visible
spectrometer



Using the values
draw standard graph

5



6

Using the equation from the
graph calculate the
concentration of sulphate

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.
- During storage of samples, some organic matter present in it may reduce sulphate SO_4^{2-}, to sulphide SO_3^{2-} , to avoid this, store sample at temperature of about 4°C. Do not allow the samples to freeze.
- Analysis should begin as soon as possible.

PRECAUTIONS

i. If the total cation concentration is more than 250 mg/L or if the heavy metal ion concentration is more than 10 mg/L, the sample is passed through an ion exchange column, to remove these ions.
ii. Colour and turbidity due to the sample matrix can cause positive interferences which must be accounted. The sample used for the analysis should be free of turbidity. If the sample is
iii. turbid filtered it through 0.45µm filter paper.
iv. Silica in concentrations over 25 mg/L will also interfere and should be treated properly.
v. If the sample containing large quantities of organic matter, then the sample cannot precipitate barium sulphate satisfactorily.

PROCEDURE

PREPARATION OF

REAGENTS Conditioning

reagent

- Measure exactly 25 ml glycerol and pour it to a dry clean beaker.
- Then, measure 15 mL of concentrated hydrochloric acid and add it to the same beaker.
- To the same beaker, add exactly 50 mL of 95 % isopropyl alcohol and mix well.
- Accurate weigh 37.5 g sodium chloride and dissolve it in distilled water.
- Then mix all the contents and make up the final volume to 250 mL using distilled water.

Standard sulphate solution

- Weigh accurately 1.479 g anhydrous sodium sulphate and dissolve it in distilled water.
- Take 1000 mL standard measuring flask and place a funnel over it.

- Transfer it to the 1000 mL standard flask and make up to 1000 mL using distilled water.
- (1 mL = 1.0 mg SO_4^{2-})

Preparation of Blank, Standards and sample for Testing

- Take six 50 mL glass stoppered standard flask (four for standards, one for the sample and one for the blank).
- Add 10 mL of the standard sulphate solution to the first standard flask, 20 mL to the second, 30 mL to the third and 40 mL to the fourth.
- To the fifth standard flask add 20 mL of the sample water.
- The sixth standard flask is for the blank, to this standard flask add distilled water alone.
- Add 5 mL of conditioning reagent to all the standard flasks.
- Then make up the volume to the 100 mL mark using distilled water.

Introduction to UV - spectrometer and spectra manager - software

The UV visible spectrometer is used to measure the Sulphate content of the given sample. This spectrometer is connected to the computer system, loaded with the software spectra manager. Spectra manager is the software which receives input from UV - visible spectrometer; it manipulates the data and displays the absorbance reading of the solution placed in the chambers of the spectrometer. To open the software double click the icon spectra manager. Different choices of measurement will appear namely:

1. Quantitative analysis
2. Spectrum analysis
3. Time course measurement
4. Fixed wavelength measurement
5. Absorbance transmittance meter
6. Environment

To measure the sulphate content select the fixed wavelength measurement method. Double click fixed wavelength measurements, the system information are transferred and then the following window will appear. Click parameter option and select absorbance, fast response and enter starting value of the wave length in nanometers. For this experiment load 420 nm as the starting value. Click ‘add’ and enter the sample number, here it is 100.Then load the number of cycles the

analysis has to be made. Enter 1 for this case. Click ok. The details of the entries will be displayed as shown. Note that the absorbance is not zero so to reset the reading to zero, take distilled water in the two sample tubes and place them in

the chambers of the spectrometer. Now click the button ‘auto zero’ the instrument resets and shows 0.0000 as reading.

TESTING OF SAMPLE

Transfer blank to the sample tubes and place it in the chamber. Now click ‘Blank’, the value of absorbance for blank is displayed as 0.0185. Then take standard 1 in the sample tube, place it in the chamber. Click ‘start’ and observe the reading is 0.0902. Similarly for standard 2 the absorbance is 0.2377 for standard 3 the reading is 0.4604, for standard 4 the reading is 0.6177, finally for standard 5 the value of the reading is 0.8024. Now transfer the given sample from standard flask to sample tube and place it in the chamber. Click ‘start’ the absorbance reading obtained is 0.7824.

CALCULATI

ON TABLE

Sample No.	Volume of Sample/ Standard (ml)	Absorbance

DETERMINATION OF SULPHATES DATA SHEET

Date Tested :

Tested By :

Project Name : Sample Number :

Sample Location : Sample Description :

Sample No.	Volume of Sample/ Standard (ml)	Absorbance
Blank	0	
Std 1		
Std 2		
Std 3		
Std 4		
Std 5		
1		
2		
3		

Calculation:

From the calibration graph, **Y = mX + C**

Whereas,

Y = Absorbance of the sample m= Slope of the Straight line X = Concentration of sulphate in mg

Concentration of Sulphate in mg/ L = $\frac{X * 1000}{\text{mL of sample taken}}$

To convert the sample size from mL to L, multiply the result by 1,000 mL/L

Concentration of Sulphate in mg/L = mg/L

INTERPRETATION OF RESULTS

The sulphates concentration in the given sample of water = **mg/L.**

INFERENCE

Water containing high levels of sulphates, particularly magnesium sulphate and sodium sulphate may have a laxative effect on persons using the water for the first time. These effects vary with the persons and appear to last only until the person becomes accustomed to using the water. High sulphates content also affects the taste of water and will form a hard scale in boilers and heat exchangers. For these reasons the upper recommended limit for sulphates in water is 250 mg/L.

EVALUATION

1. In this method, the sulphates present are estimated in the form of
    a) Barium Sulphate
    b) Calcium Sulphate
    c) Sodium Sulphate
    d) Magnesium Sulphate

2. The turbidity is measured using UV-visible spectrometer at
    a) 410 nm
    b) 420 nm
    c) 430 nm
    d) 440 nm

3. For plotting a standard graph, the minimum requirement is
    a) 3 standards
    b) 2 standards
    c) 1 standards
    d) 4 standards

4. The conditioning reagent, contains _____
    a) Glycerol
    b) Phenol

c) Sodium sulphate
d) Sodium Hydroxide

5. The minimum detectable concentration by turbidimetric method is

   a) 10 ppm
   b) 5 ppm
   c) 1 ppm
   d) No limitations

6. A calibration curve is the plot of

   a) absorbance against concentration of solutions
   b) absorbance of solutions against time
   c) time against concentration of solutions
   d) concentration of solutions against time

7. As per Beer - Lambert's Law the amount of light absorbed is_____the concentration of the

   a) inversely proportional
   b) directly proportional
   c) greater than
   d) smaller than

8. Transmittance is the ratio of the intensity of transmitted light to that of incident light.

   a) True
   b) False

9. The optical density is_____to concentration of the substance.

10. Blank solution is used in colorimetric estimation to

    a) nullify the absorbance caused due to the colouring impurities present in the reagents
    b) check the instrument
    c) nullify the error caused by the instrument
    d) nullify the error caused by colour

Ex.No.9 EXPERIMENT ON DETERMINATION OF OPTIMUM COAGULANT DOSAGE

PREAMBLE:

"How to determine optimum coagulant dosage for Water and Wastewater".

Test procedure is in accordance to IS: 3025 (Part 50) - Reaffirmed 2002.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

(1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 4500-H^+ B.

(2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79- 020, USEPA, Method 150.1.

AIM

To determine the PH of the given water sample with the stipulations as per **IS: 3025 (Part 50) - Reaffirmed 2002**

INTRODUCTION

In plain sedimentation, very fine suspended particles of size 0.006mm to 0.002mm are not removed, since they required a detention period of 10 hours to 4 days which is impracticable. In addition to this fine suspended particle, water also contains electrically charged colloidal particles which are continuously in motion and never settle down due to gravity. It has been found that, the above mentioned impurities can be removed by sedimentation with coagulation.

It has been found that when certain chemicals (i.e. coagulants) are added to water an insoluble, gelatinous precipitate is formed. This precipitate during its formation and descent through the water absorbs very fine suspended and colloidal impurities there by reducing the turbidity of the water

ENVIRONMENTAL SIGNIFICANCE

Coagulation of raw water using the optimum coagulant dose removes colloidal impurities from the water. These colloidal impurities are normally associated with organic matter containing pathogenic bacteria which are responsible for water borne diseases. The chemical coagulation also makes the process of disinfection more effective. Coagulation also removes objectionable colour, taste and odour's from

water. Usually the dose of Alum varies between 5mg/lit for relatively clear water to about 85 mg/lit for very turbid waters. The average dose is about 20mg/lit.also reduce the germicidal potential of chlorine. High pH induces the formation of trihalomethanes, which are causing cancer in human beings.

PRINCIPLE

The amount of coagulant required for coagulation depends on the turbidity of the waste water. The use of optimum amount of coagulant is indicated by the formation of the large feathery flakes. This can be approximately determined in the laboratory by Jar test. The test involves rapid mixing to disperse the chemicals (coagulants) in the sample and slow mixing for the floc formation.

MATERIALS REQUIRED

APPARATUS REQUIRED

1. Jar test apparatus
2. Nephelo turbidity meter

CHEMICALS REQUIRED

1. ALUM Solution

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 optimum dosage, keep samples at 4^0 C. Do not allow the samples to freeze.

Analysis should begin as soon as possible.

PROCEDURE

Three major steps are involved in the experiment. They are

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

PREPARATION OF REAGENTS ALUM SOLUTIONS

- Dissolve 1.0 gram of Alum in 1 lit of distilled water so that each ml. of Alum solution contains one milligram of Alum.

TESTING OF SAMPLE

- Using 1000 ml measuring cylinder, measure 800 ml sample into 1000 ml tall form beakers and place them in position in multiple stirrer, taking care to keep a minimum of 5 mm gap between the stirrer blade and the inner surface of the beaker.
- Take a further 250 ml of sample and determine its pH, turbidity and color.
- Transfer the required volumes of coagulant into the coagulant vessels using a graduated pipette of 10 ml.
- Switch the stirrer on to fast, measure the temperature in one of the beaker and add coagulant after stirring vigorously for at least one minute. Start the stop watch on adding the coagulant. Quickly rinise the coagulant vessels with distilled water and add the rinsing to the beakers.
- One minute after adding the coagulant switch the stirrer to slow and observe beakers carefully, noting the time taken for pin-point floc to appear in each beaker.

- After 15 minutes of slow stirring switch off the stirrer and carefully remove the beakers from the stirring apparatus.
- Allow them to stand for 15 minutes then carefully decant 150 to 200 ml of supernatant from each beaker in clean beaker or flask. A decanted sample is nearly always satisfactory but occasionally it may be found to have small amount of floc that tends to float. In such situation collect supernatant sample at a depth of 30-40 mm using glass siphon.
- Repeat these steps with different concentration of chemicals, different flash mix speed and different setting time to arrive at the optimum conditions. Optional pH, if not already known, for the coagulants of interest, may be determined by conducting test at various pH.

CALCULATION

1. The dose of coagulant versus floc formation is plotted as graph.
2. The dose of coagulant which gives the best floc is the optimum dose of coagulants.

DETERMINATION OF OPTIMUM DOSAGE OF COAGULANT DATA SHEET

Date Tested :

Tested By :

Project Name :

Sample Number :

Sample Location

BH1 : Sample

Description :

Sample Location

BH2 : Sample

Description :

Sample Location

BH2 : Sample

Description :

TABULATION

Jar No	Amount of coagulant added (g)	Floc formation (ml)

Result:-

INTERPRETATION OF RESULTS

The optimum dosage for the given sample is

EVALUATION

1) What is the best coagulant for drinking water?
2) What is the best coagulant for sewage?
3) Why did the pH change after adding coagulant?

Ex.No. 10 EXPERIMENT ON DETERMINATION OF DISSOLVED OXYGEN

PREAMBLE:

"**How to determine dissolved oxygen in Water and Wastewater**".

Test procedure is in accordance to IS: 3025 (Part 38) - Reaffirmed 2003.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

(1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 4500-O G.
(2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 360.1.

AIM

To determine dissolved oxygen (DO) in the given water sample with the stipulations as per **IS: 3025 (Part 38) - Reaffirmed 2003.**

INTRODUCTION

Before performing this experiment, few questions may arise to the learners:

1. What is meant by Dissolved Oxygen (DO)? Is it oxygen in dissolved form?
2. Why we need to determine DO?
3. What are the methods available to determine DO?
4. Is it measured in natural water or wastewater?
5. Whether is it mandatory as per our codal provision to determine DO?

The term Dissolved Oxygen is used to describe the amount of oxygen dissolved in a unit volume of water. Dissolved oxygen (DO) is essential for the maintenance of healthy lakes and rivers. It is a measure of the ability of water to sustain aquatic life.
The dissolved oxygen content of water is influenced by the source, raw water temperature, treatment and chemical or biological processes taking place in the distribution system.

The presence of oxygen in water is a good sign. Depletion of dissolved oxygen in water supplies can encourage the microbial reduction of nitrate to nitrite and sulfate to sulfide. It can also cause an increase in the concentration of ferrous iron in solution, with subsequent discoloration at the tap when the water is aerated.

Hence, analysis of dissolved oxygen is an important step in water pollution control and wastewater treatment process control. There are various methods available to measure Dissolved Oxygen, which we will discuss in detail.

In a healthy body of water such as a lake, river, or stream, the dissolved oxygen is about 8 parts per million. The minimum DO level of 4 to 5 mg/L or ppm is desirable for survival of aquatic life.

Now imagine that a source of oxygen demanding wastes, such as feed lot, a paper mill or a food processing plant, is built besides the river. The facility begins operating and discharging wastes into the river.

This increases the BOD and affects the concentration of DO in the waters downstream.The wastes serve as the food for certain aerobic bacteria. as it moves downstream, the conc. of bacteria increases. Because these bacteria remove oxygen from water, their population increase causes a decline in the amount of DO.

Beyond certain point, most of the wastes break down. The conc. of DO rises as the river recovers oxygen from the atmosphere and aquatic plants.
Thus DO test is the basis for BOD test which is an important parameter to evaluate organic pollution potential of a waste.

It is necessary for all aerobic biological wastewater treatment processes to control the rate of aeration.

ENVIRONMENTAL SIGNIFICANCE

Drinking water should be rich in dissolved oxygen for good taste.

DO test is used to evaluate the pollution strength of domestic and industrial waste. Higher values of DO may cause corrosion of Iron and Steel.

Algae growth in water may release oxygen during its photosynthesis and DO may even shoot upto 30 mg/L.

Oxygen is poorly soluble in water. Its solubility is about 14.6 for pure water at 0°C under normal atmospheric pressure and it drops to 7 mg/l at 35°C.

Higher temperature, biological impurities, Ammonia, Nitrates, ferrous iron, chemicals such as hydrogen sulphide and organic matter reduce DO values.

Aerobic bacteria thrive when oxygen is available in plenty. Aerobic conditions do prevail when sufficient DO is available within water. End products of aerobiosis are stable and are not foul smelling.

It is necessary to know DO levels to assess quality of raw water and to keep a check on stream pollution.

DO test is the basis for BOD test which is an important parameter to evaluate organic pollution potential of a waste.

DO test is necessary for all aerobic biological wastewater treatment processes to control the rate of aeration.

PRINCIPLE

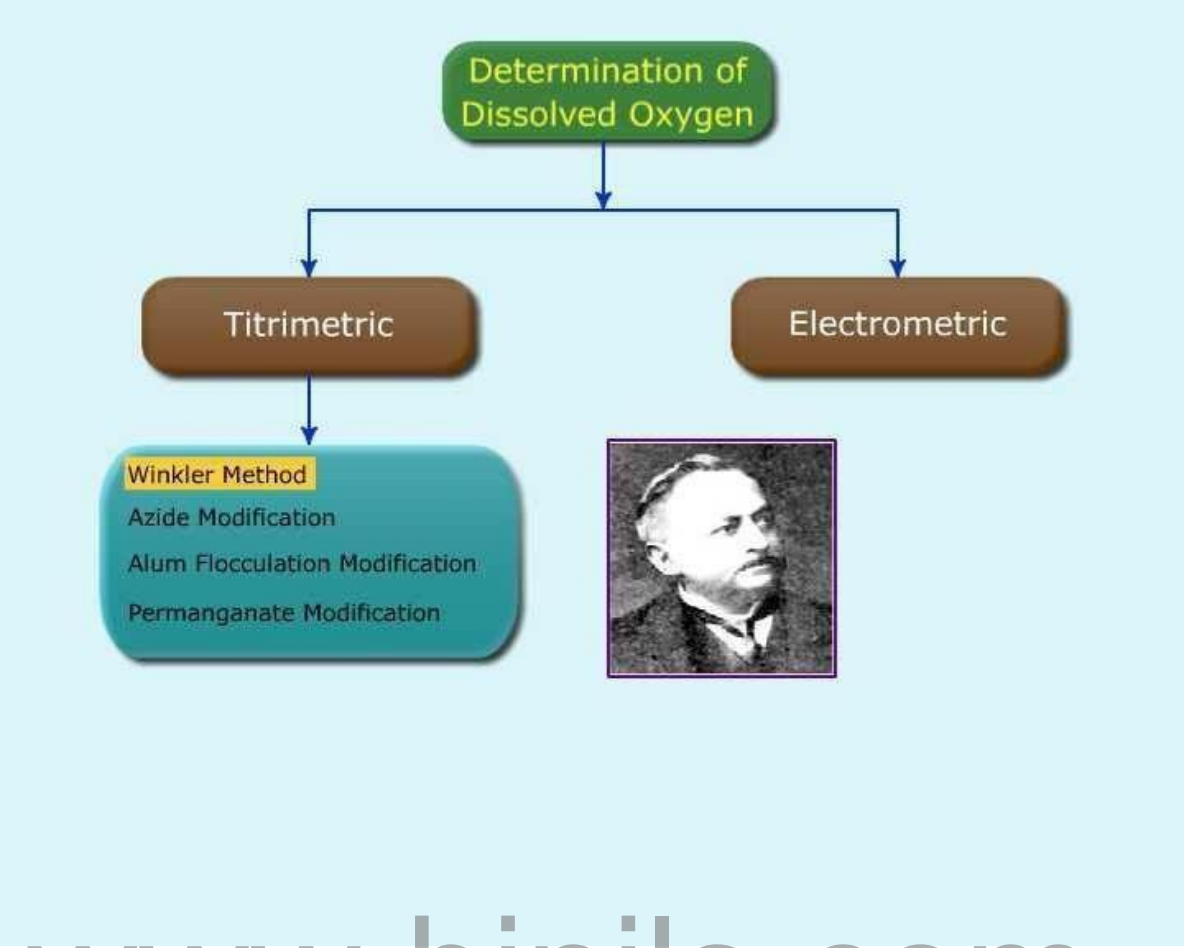
Dissolved Oxygen can be measured either by titrimetric or electrometric method.

(1) Titrimetric Method

Titrimetric method is based on the oxidizing property of DO while the electrometric method (using membrane electrodes) is based on the rate of diffusion of molecular oxygen across a membrane. It is most accurate method to determine DO.

There are different titrimetric methods based on the nature of sample to be tested.

(a) Winkler Method
(b) Azide Modification
(c) Alum Flocculation Modification
(d) Permanganate Modification



However, in all the above the basic principle remains same.

Choice of the method depends upon the type of sample to be tested
Azide Modification:

In this method, interference caused by nitrate is removed effectively. Presence of nitrate is most interference in biologically treated effluent and incubated BOD samples.

Alum Flocculation Modification:

If the sample contains suspended solids (especially effluent samples), then this method will be suitable.

Permanganate Modification:

If the sample contains iron (Fe^{2+}) ions. Addition of 1mL of potassium fluoride and azide solution can be adopted to suppress the interference due to (Fe^{3+}).

This method is not useful when the sample contains sulphites, thiosulphates and high BOD.

The Titrimetric principle:

Divalent Manganese salt in solution is precipitated by strong alkali to divalent manganese hydroxide.

$$MnSO_4 + 2\,KOH \rightarrow Mn(OH)_2 + K_2SO_4$$

Addition of Potassium iodide or Potassium hydroxide is added to create a pinkish brown precipitate.

In the alkaline solution, dissolved oxygen present in the sample rapidly oxidized to form trivalent or higher valency hydroxide.

$$2Mn(OH)_2 + O_2 \rightarrow 2MnO(OH)_2$$

$MnO(OH)_2$ appears as a brown precipitate. There is some confusion about whether the oxidised manganese is tetravalent or trivalent. Some sources claim that $Mn(OH)_3$ is the brown precipitate, but hydrated MnO_2 may also give the brown colour.

Iodide ions are added and acidified (acid facilitates the conversion by the brown), which reduces tetravalent hydroxides back to their stable divalent state thereby liberating equivalent amount of iodine.

$$Mn(OH)_2 + 2\,KI + H_2O \rightarrow Mn(OH)_2 + I_2 + 2KOH$$

Thiosulphate solution is used, with a starch indicator, to titrate the iodine.

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

This iodine is equivalent to dissolved oxygen present in the sample.

(2) Electrometric Method

The electrode method offers several advantages over the titrimetric method including speed, elimination or minimization of interferences, field compatibility, continuous monitoring and insitu measurement.

Dissolved oxygen can be measured by a special sensor kept in an electrochemical cell by the amperometric method.

The cell comprises a sensing electrode, a reference electrode and a supporting electrolyte, a semi-permeable membrane, which served dual function.

It separates the water sample from the electrolyte, and at the same time, permits only the dissolved oxygen to diffuse from the water sample through the membrane into the supporting electrolyte.

The diffusion current created by migration of oxygen through a permeable membrane is linearly proportional to the concentration of molecular oxygen in the sample.

The diffusion current created by migration of oxygen through a permeable membrane is linearly proportional to the concentration of molecular oxygen in the sample.

The sample is treated with manganous sulphate, alkaline-iodide-azide reagent and finally sulfuric acid. The first two chemicals combine with dissolved oxygen to form a compound which, when acid is added, releases free iodine (from the potassium iodide).

MATERIALS REQUIRED

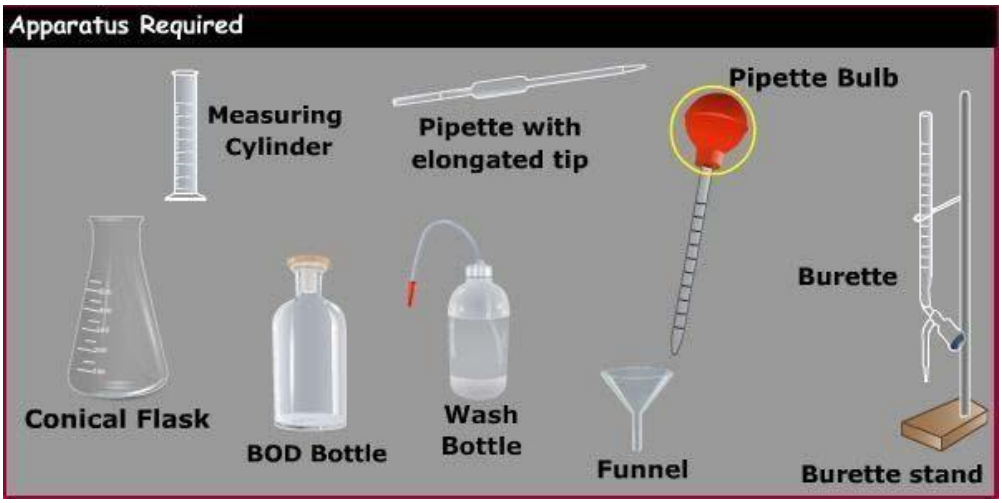
APPARATUS REQUIRED

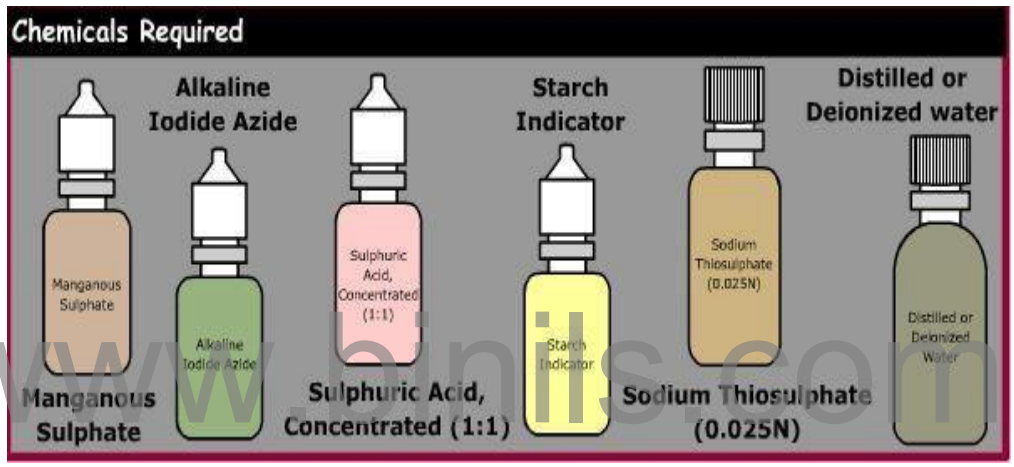
1. Burette
2. Burette stand
3. 300 mL glass stoppered BOD bottles
4. 500 mL conical flask
5. Pipettes with elongated tips
6. Pipette bulb
7. 250 mL graduated cylinders
8. Wash bottle

CHEMICALS REQUIRED

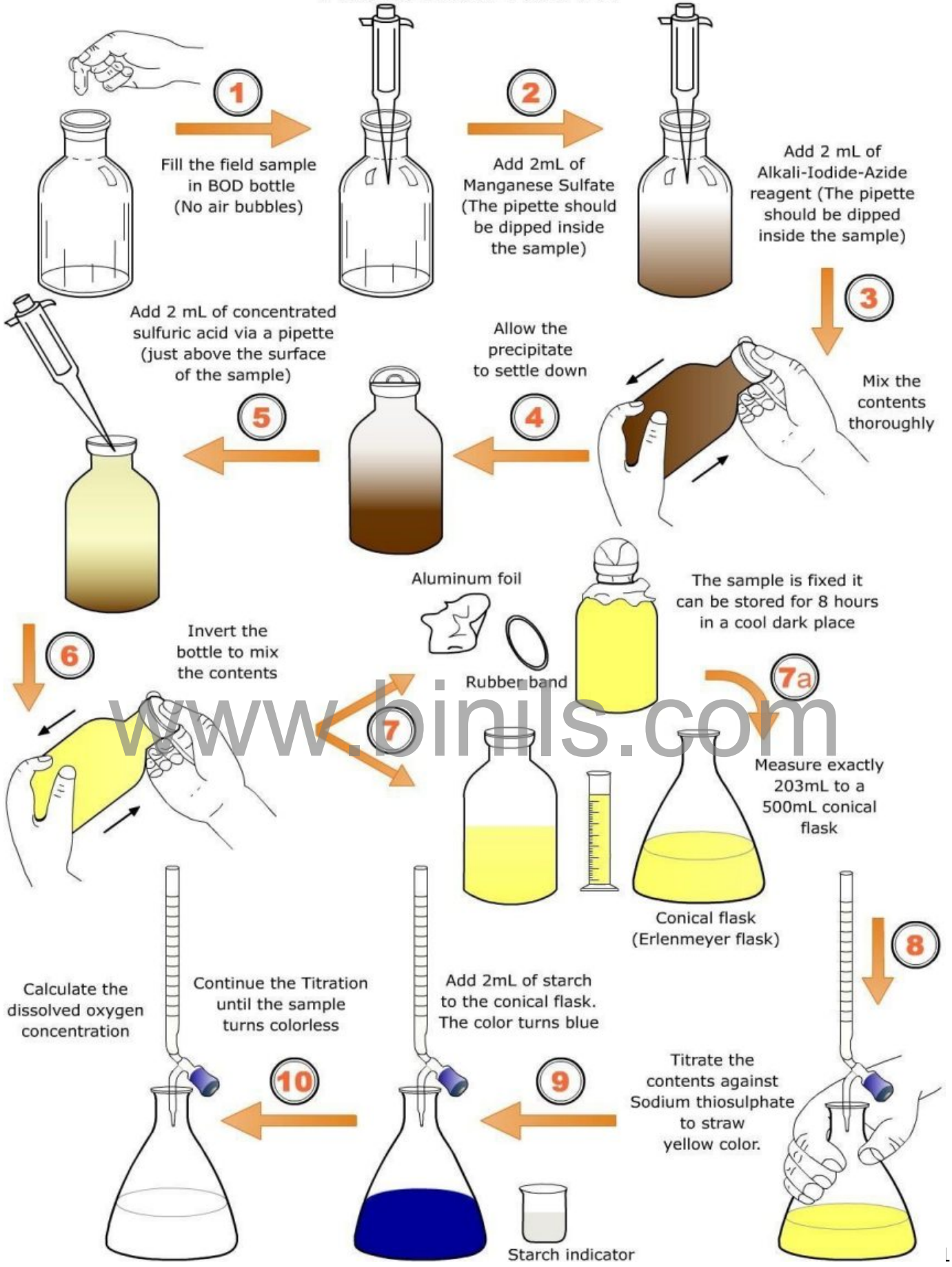
1. Manganous sulphate solution
2. Alkaline iodide-azide solution
3. Sulfuric acid, Concentrated
4. Starch indicator solution
5. Sodium thiosulphate
6. Distilled or deionized water
7. Potassium Hydroxide

8. Potassium Iodide
9. Sodium Azide





PROCEDURE CHART



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

Do not allow samples to freeze. Analysis should begin as shown as possible. Do not open sample bottle before analysis.

Begin analysis within six hours of sample collection.

PRECAUTIONS

The experiment involves lot of solutions and additions of strong acid and alkali and hence care should be taken.

- Dissolved oxygen concentrations may change drastically depending upon depth, distance, temperature and period of sampling.
- If the sample was obtained by a sampling device of some kind, the water cannot be simply poured into a BOD bottle, since this would cause aeration of the sample. Instead, the sample must be drawn off from a tube located near the bottom of the sampling device. Place the rubber tube into the bottom of the BOD bottle and fill the bottle, again allowing the bottle to overflow.
- For shallow depth use normal water samplers. However for depth greater than 150 cm (5 ft), use Kemmerer Sample Bottles.

In the case of electrode method:

- Membrane-covered electrode systems minimize the interferences often encountered with dropping mercury or rotating platinum electrodes.
- The sensing element is protected by an oxygen permeable membrane, which serves as a diffusion barrier against matrix interference problems.

PROCEDURE:

For testing the given sample, first the reagents are required to be prepared.

PREPARATION OF REAGENTS

a) Manganous Sulphate Solution

Dissolve Manganese Sulphate

- 480g of $MnSO_4.4H_2O$ (or)
- 400g of $MnSO_4.2H_2O$ (or)
- 364 g of $MnSO_4.H_2O$

in freshly boiled and cooled distilled water, filter the solution and make up to 1000 mL (One litre). In this experiment, we are using Manganese sulphate Mono hydrate,

Take 364 g Manganese sulphate Mono hydrate ($MnSO_4.H_2O$) and transfer it to the beaker. To dissolve the content, place it in the magnetic stirrer.

The solution should not give blue color by addition of acidified potassium iodide solution and starch.

b) Alkaline Iodide Sodium Azide Solution

To prepare this reagent we are going to mix three different chemicals

Dissolve either

- 500 g of Sodium Hydroxide (or)
- 700 g of Potassium Hydroxide
- and 135 g of Sodium Iodide
- (or)
  150 g of Potassium Iodide

To prepare this reagent, take 700 g of Potassium hydroxide and add 150 g of potassium iodide and dissolve it in freshly boiled and cooled water, and make up to 1000 mL (One litre).

Dissolve 10 g of Sodium Azide (NaN_3) in 40 mL of distilled water and add this with constant stirring to the cool alkaline iodide solution prepared.

c) Sodium Thiosulphate Stock Solution

Weigh approximately 25 g of sodium thiosulphate $(Na_2S_2O_35H_2O)$ and dissolve it in boiled distilled water and make up to 1000 mL. Add 1 g of Sodium Hydroxide to preserve it.

d) Starch Indicator

Weigh 2 g of starch and dissolve in 100 mL of hot distilled water. In case if you are going to preserve the starch indicator add 0.2 g of salicylic acid as preservative.

e) Sulphuric Acid

TESTING OF SAMPLE

- Take two 300-mL glass stoppered BOD bottle and fill it with sample to be tested. Avoid any kind of bubbling and trapping of air bubbles. Remember –

no

bubbles!

(Or)

- Take the sample collected from the field. It should be collected in BOD bottle filled upto the rim.
- Add 2mL of manganese sulfate to the BOD bottle by inserting the calibrated pipette just below the surface of the liquid.
- Add 2 mL of alkali-iodide-azide reagent in the same manner.
- Squeeze the pipette slowly so no bubbles are introduced via the pipette (The pipette should be dipped inside the sample while adding the above two reagents. If the reagent is added above the sample surface, you will introduce oxygen into the sample).
- If oxygen is present, a brownish-orange cloud of precipitate or floc will appear.
- Allow it to settle for sufficient time in order to react completely with oxygen.
- Add 2 mL of concentrated sulfuric acid via a pipette held just above the surface of the sample.
- Carefully stopper and invert several times to dissolve the floc.
- At this point, the sample is "fixed" and can be stored for up to 8 hours if kept in a cool, dark place.

- Rinse the burette with sodium thiosulphate and then fill it with sodium thiosulphate. Fix the burette to the stand.
- Measure out 203 mL of the solution from the bottle and transfer to an conical flask.
- Titration needs to be started immediately after the transfer of the contents to conical flask.
- Titrate it against sodium thiosulphate using starch as indicator. (Add 3 - 4 drops of starch indicator solution)
- End point of the titration is first disappearance of the blue color to colorless.
- Note down the volume of sodium thiosulphate solution added which gives the dissolved oxygen in 7.9 mL
- Repeat the titration for concordant values.

CALCULATION

For determining the Dissolved Oxygen (DO) in the given water sample, the readings are required to be tabulated.

TABLE

Trial No.	Temperature (ºC)	Volume of Sample (mL)	Burette Reading (mL)		Volume of Titrant (mL)	Dissolved Oxygen (mg/L)
			Initial	Final		
1.						
2.						
3.						

Burette Solution: Sodium Thiosulphate

Pipette Solution: Sample

Indicator: Starch

End point : Disappearance of blue color

- For the calculation of DO the temperature at the time of measurement is 20º C and the volume of sample taken is 200 mL.
- sodium thiosulphate is taken in the burette
- For the first titration the Initial reading is 0 mL and the final reading is 7.8. The volume of sodium thiosulphate consumed to get the end point is 7.8 mL.
- For the second titration the Initial reading is 0 mL and the final reading is 7.9. The volume of sodium thiosulphate consumed to get the end point is 7.9 mL.
- For the third titration the Initial reading is 0 mL and the final reading is 7.9. The volume of sodium thiosulphate consumed to get the end point is 7.9 mL.
- For the second and third titration, we have achieved concordant value. So we can go for the calculations.

DETERMINATION OF DISSOLVED OXYGEN DATA SHEET

Date Tested :

Tested By :

Project Name : Sample Number :

Sample Location : Sample Description :

Trial No.	Temp (°C)	Volume of Sample (mL)	Burette Reading (mL)		Volume of Titrant (mL)	Dissolved Oxygen (mg/L)
			Initial	Final		
1.						
2.						
3.						

Model Calculation:

Volume of Sodium thiosulphate V_1 = mL
Normality of Sodium thiosulphate N_1 = N
Volume of Sample V_2 = mL

$$Dissolved\ Oxygen = \frac{Volume\ of\ Sodium\ Thiosulphate * 0.2 * 1000}{Volume\ of\ Sample\ taken}$$

= mg/L

INTERPRETATION OF RESULTS

The Dissolved Oxygen in the given sample of water at 27ºC = **mg/L.**

INFERENCE

Dissolved oxygen of the tested sample is 7.9 mg/L. Test results shows the water is in healthy condition and fit for aquatic life. IS code does not mentioned minimum standards for DO. However, for healthy water body, the dissolved oxygen is about 8 parts per million.

EVALUATION

1. Winkler titration method is based on_____property of Dissolved Oxygen.

    a) Reduction
    b) Oxidation
    c) Redox
    d) Decomposition

2. Dissolved oxygen in the water mainly depends upon Organic content of the water.

    a) True
    b) False

3. The ingredients of Alkali are NaOH, NaI

    a) NaN_4
    b) NaN_3
    c) NaN_2
    d) NaN

4. The precipitate formed after the addition of MnSO4 and Alkali azide is_____.

    a) Manganese Hydroxide
    b) Sodium sulphate
    c) Potassium sulphate
    d) Manganese oxide

5. Dissolved Oxygen depends only on Physical Properties of the water.

    a) True
    b) False

6. Along the stream the increase in dissolved oxygen in water will be at the

a) riffles
b) warm pool
c) bank erosion
d) top

7. The dissolved Oxygen in potable water_____.

a) imparts freshness
b) improves taste
c) improves smell
d) imparts colour

8. Sulphide and Sulphur dioxide interfere in the determination of dissolved oxygen.

a) True
b) False

9. The sample obtained for testing Dissolved Oxygen can be preserved by

a) adding the reagents and stored at 10 to 20 for up to 8 hours
b) storing at room temperature for up to 24 hours
c) storing at 0 for up to 24 hours
d) adding the reagents and stored at room temperature for up to 24 hours

10. Minimum DO in the fresh water for the survival of aquatic life is

a) 0 mg/l
b) 2 mg/l
c) 8 mg/l
d) 4 mg/l

Ex.NO 11 EXPERIMENT ON DETERMINATION OF TOTAL ORGANIC AND INORGANIC SOLIDS IN WATER

PREAMBLE:

"**How to determine total organic and inorganic solids in Water and Wastewater**". Test procedure is in accordance to IS: 3025 (Part 18) - Reaffirmed 2002.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

(1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 2540 E.

(2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 160.4.

AIM

To determine total organic and inorganic solids in the given water sample with the stipulations as per **IS: 3025 (Part 18) - Reaffirmed 2002.**

INTRODUCTON

The term total volatile solids refer to materials that are completely volatilised from water at higher temperature (550ºC). These solids are often referred to the organic content of the water. The term total fixed solids can be referred to materials which are not volatilised from water at higher temperature (550ºC). These solids are often referred to the inorganic content of the water.

ENVIRONMENTAL SIGNIFICANCE

- The water which consists of high volatile solids is not suitable for drinking purpose and indicates that the water may have been polluted by domestic wastes or other organic wastes.
- Volatile solids test is normally applied to sludges. It is indispensable in the design and operation of sludge digest, vacuum filter and incineration plants.
- Before the development of the COD test, it is used to find out the strength of industrial and domestic wastewater. It is helpful in assessing the amount biologically inert organic matter, such as lignin in case of wood pulping waste liquours.

- The determination of volatile and fixed components in the residue is useful in the control of waste water plant operation because it offers an approximate amount of organic matter present in the solid fraction of wastewater.

PRINCIPLE

The sample is evaporated in a weighed dish on a steam bath and is dried to a constant mass in an oven at 103-105 C. The residue obtained is ignited to constant weight at 550(C. The remaining solids represent the total fixed solids and the weight lost during the ignition represents the total volatile solids.

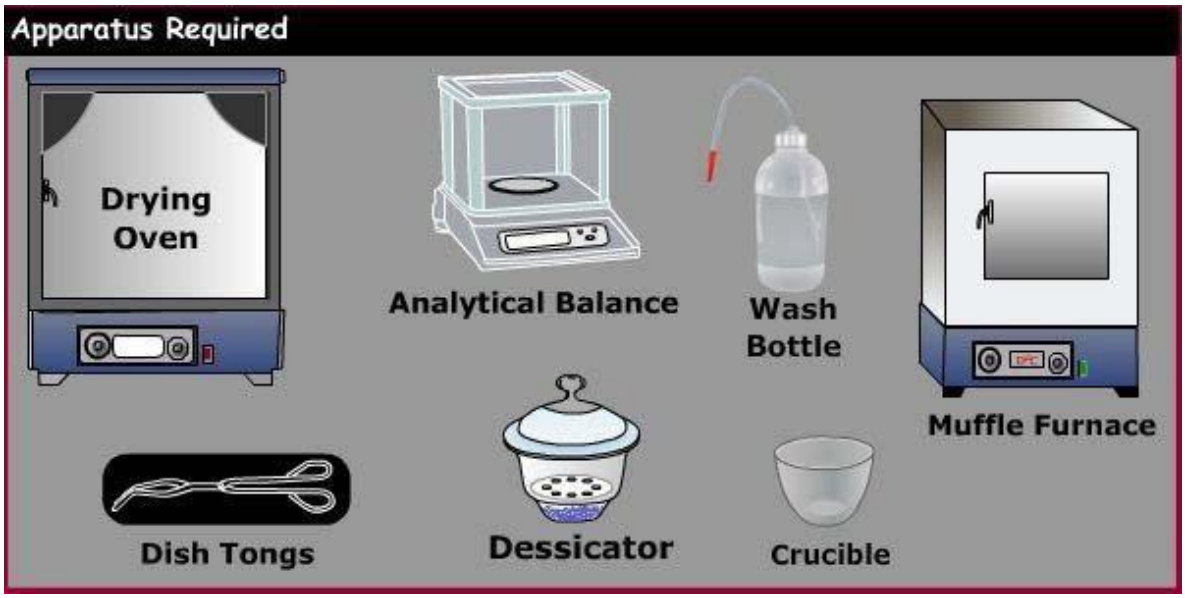
MATERIALS

REQUIRED

APPARATUS

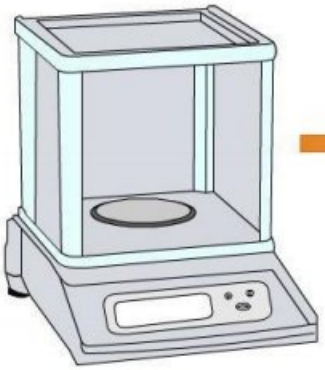
REQUIRED

1. Evaporating Dish
2. Water Bath (Steam Bath)
3. Oven
4. Desiccators
5. Weighing balance
6. Dish Tongs
7. Magnetic Stirrer
8. Wash Bottle



PROCEDURE CHART

Switch on the balance (Atleast 30 min before the test)

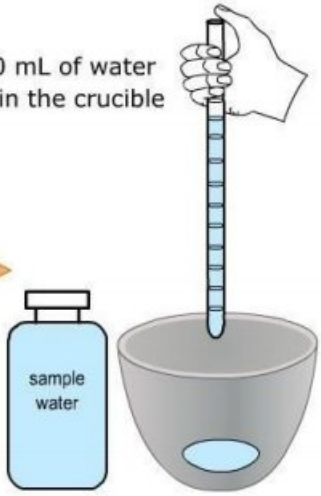


1

Notedown the intial dry weight of the crucible

2

Take 20 mL of water sample in the crucible



3

Place the crucible inside the oven at 103^{0}C

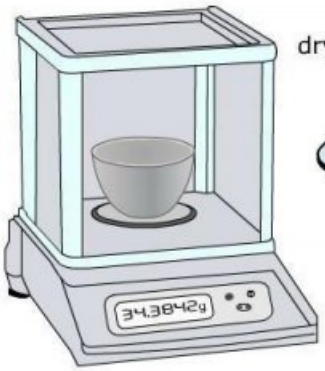
4

After drying in the oven cool to room temperature in dessicator, Note down the dry weight of the crucible

5

Place the crucible inside the muffle furnace at 550^{0}C

6



After drying in the oven cool to room temperature in dessicator, Note down the final dry weight of the crucible

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:

- Negative errors in volatile solids may be produced by loss of volatile matter during drying in the oven.
- In the presence of high concentration fixed solids, the determination of low concentration of volatile solids may be subject to considerable error. In those cases, the measure of volatile components by some other method like total organic carbon is advisable.
- 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.

PROCEDURE

TESTING OF SAMPLE

- To measure total volatile solids and fixed solids, take a clean silica crucible which has been washed and dried in a hot air oven at 105ºC for one hour and ignited at 550ºC to remove all organic materials present in it.
- Now weigh the empty silica crucible in analytical balance. Let's denote the weight measured as W1 = g
- Using pipette transfer 75mL of unfiltered sample in the porcelain dish.
- Switch on the oven and allowed to reach 105°C. Check and regulate oven and furnace temperatures frequently to maintain the desired temperature range.
- Place the silica crucible in the hot air oven and care should be taken to prevent splattering of sample during evaporation or boiling.
- Dry the sample to get constant mass. Drying for long duration is done to eliminate

necessity of checking for constant mass.

- Cool the container in a desiccator. Desiccators are designed to provide an environment of standard dryness. This is maintained by the desiccant found inside. Don't leave the lid off for prolonged periods or the desiccant will soon be exhausted.
- We should weigh the dish as soon as it has cooled to avoid absorption of moisture due to its hygroscopic nature.
- Samples need to be measured accurately, weighed carefully, and dried and cooled completely.
- Note the weight with residue as **W_2 = g**
- Switch on the furnace and allow it to reach 550°C. Check and regulate the furnace temperatures frequently to maintain the desired temperature range.
- Place the silica crucible in the furnace and care should be taken while keep the crucible inside the furnace since it will be too hot.
- Allow it to ignite for 20 minutes to get constant mass.
- As above, cool the silica crucible in a desiccator to room temperature.
- Weigh the dish as soon as it has cooled to avoid absorption of moisture due to its hygroscopic nature.
- Note the weight with residue as **W_3 = g**

CALCULATI

ON Total Volatile

Solids

Initial weight of the evaporating dish + sample **(W_1)** = ...g
Final weight of the evaporating dish + sample after drying at 105ºC (**W_2**) = ...g
Final weight of the evaporating dish + sample after drying at 550ºC (**W_3**) = ...g

Weight of volatile substance **(W)** = $W_2 - W_3$ g

Amount of total solids present in the sample =

$\frac{1000 \times 1000\ W}{V}$ W = weight of total residue in (mg). (Therefore multiply W with 1000)

V = Volume of the sample (mL) (To convert mL to L)= mg/L

Total Fixed Solids

Initial weight of the evaporating dish **(W_1)** = ... g
Final weight of the evaporating dish + sample after drying at 105ºC (**W_2**) = ... g
Final weight of the evaporating dish + sample after drying at 550ºC (**W_3**) = ... g

Weight of non volatile substance **(W)** = $W_3 - W_1$ g

Amount of total fixed solids present in the sample = $\frac{1000*1000\,W}{V}$

W = weight of total residue in (mg). (Therefore multiply W with 1000)

V = Volume of the sample (mL) (To convert mL to L) = mg/L

TABLE

Total Volatile Solids

Description		**Weight (g)**
Weight of the clean silica crucible (g)	W_1	
Weight of the silica crucible and the residue (g)	W_2	
Weight of residue (g)	W	
Weight of the silica crucible and the ash (g)	W_3	
Weight of ash (g)	W	
Volume of the Sample (mL)	V	
Total Volatile Solids (mg/L)	**TVS**	

The Weight of the clean silica crucible (g) W_1 = g

The Weight of the clean silica crucible and the residue (g) W_2 = g The Weight of the re

The Weight of the silica crucible and the ash (g) W_3 = g Weight of the ash (g) W =

The volume of the sample (mL) V = mL

Total Fixed Solids

Description		Weight (g)
Weight of the clean silica crucible (g)	W_1	
Weight of the silica crucible and the residue (g)	W_2	
Weight of residue (g)	W	
Weight of the silica crucible and the ash (g)	W_3	
Weight of ash (g)	W	
Volume of the Sample (mL)	V	
Total Fixed Solids (mg/L)	**TFS**	

The Weight of the clean silica crucible (g) W_1 = g

The Weight of the silica crucible and the residue (g) W_2 = g Weight of the residu

Weight of the silica crucible and the ash (g) W_3 = g Weight of the ash (g) W_a=

Volume of the sample (mL) V = mL

DETERMINATION OF TOTAL VOLATILE SOLIDS
DATA SHEET

Date Tested :

Tested By :

Project Name :

Sample Number :

Sample Location :

Sample Description :

Description		**Weight (g)**
Weight of the clean silica crucible (g)	W_1	
Weight of the silica crucible and the residue (g)	W_2	
Weight of residue (g)	W	
Weight of the silica crucible and the ash (g)	W_3	
Weight of ash (g)	W	
Volume of the Sample (mL)	V	
Total Volatile Solids (mg/L)	**TVS**	

Calculation:

W_2 = g

W_3 = g V = mL

Weight of residue (g) W = $W_2 - W_3$

=

Weight of residue in mg (To convert W (g) to W (mg), multiply W (g) with 1000) W (mg) =

Multiply the weight of the dry solids (in mg) by 1,000 mL/L to convert the sample size from mL to L.

Total Volatile Solids (mg/L)

V = Volume of the sample (mL) (To convert mL to L, multiply by 1000)

DETERMINATION OF TOTAL FIXED SOLIDS DATA SHEET

Date Tested :

Tested By :

Project Name :

Sample Number :

Sample Location : Sample Description :

Description		**Weight (g)**
Weight of the clean silica crucible (g)	W_1	
Weight of the silica crucible and the residue (g)	W_2	
Weight of residue (g)	W	
Weight of the silica crucible and the ash (g)	W_3	
Weight of ash (g)	W	
Volume of the Sample (mL)	V	
Total Fixed Solids (mg/L)	**TFS**	

Calculation:

W_1 = g

W_3 = g

V = mL

Weight of residue (g) W = $W_3 - W_1$

=

Weight of residue in mg (To convert W (g) to W (mg), multiply W (g) with 1000) W (mg) =

=

Multiply the weight of the dry solids (in mg) by 1,000 mL/L to convert the sample size from mL to L.

Total Fixed Solids (mg/L)

V = Volume of the sample (mL) (To convert mL to L, multiply by 1000)

= m**g/L**

INTERPRETATION OF RESULTS

In the given sample, total volatile solids is equivalent to **mg/L** and total fixed solids is **mg/L.**

INFERENCE

In domestic wastewater, solids are about 50 percent organic, which in turn contaminates the ground and fresh water. These solids are generally from vegetable, dead animal matter, and also include synthetic organic compounds. They can be ignited or burned. Since the organic fraction can be driven off at high temperatures, they are called volatile solids. Inorganic solids are frequently called mineral substances and include sand, gravel and silt as well as the mineral salts in the water supply which produce the hardness and mineral content of the water. Mostly, they are non-combustible. They are called non volatile solids.

EVALUATION

1. The Total Volatile Solids determination is very important in the control of

   a) Water treatment plant
   b) Sewage treatment plant
   c) Desalination plant
   d) Effluent treatment plant

2. The crucible with sample, should be placed in the muffle furnace for atleast_____.

   a) one hour
   b) two hours
   c) 20 minutes
   d) 10 minutes

3. The Total Fixed Solids is the measure of

   a) all the solids present
   b) inorganic solids present
   c) the salt content
   d) organic solids

4. The method used for the determination of solids is_____.

    a) volumetric method
    b) gravimetric method
    c) instrumentation method
    d) visual method

5. The crucible after ignition should be cooled in a desiccator

    a) because it is hot
    b) to avoid moisture absorption
    c) to cool
    d) to incubate

6. Putrescible solid means

    a) pure solids
    b) dissolved solids
    c) solids with high BOD
    d) suspended solids

7. The solid organic matter (sludge) digested by Aerobic treatment.

    a) True
    b) False

8. The determination of total volatile solids is interfered by

    a) Loss of volatile solids during the drying process
    b) Large volatile solids water sample
    c) Dissolved salts
    d) Suspended salts

9. While placing the crucible in muffle furnace it is advisable to wear gloves made of

    a) Leather
    b) Rubber
    c) Resin
    d) Polythene

10. The Total Volatile Solids is the measure of

    a) all the solids present
    b) organic solids present
    c) the salt content
    d) inorganic salts present

Ex. No. 12 EXPERIMENT ON DETERMINATION OF TOTAL SOLIDS IN WATER

PREAMBLE:

"**How to determine total solids in Water and Wastewater**".

Test procedure is in accordance to IS: 3025 (Part 15) - Reaffirmed 2003.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

(1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 2540 B.

(2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 160.3.

AIM

To determine the total solids in the given water sample. Test procedure is in accordance to **IS: 3025 (Part 15) - Reaffirmed 2003.**

INTRODUCTION

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

Solids can be classified as either filterable or non filterable. Filterable solids may either be settleable or non settleable. Solids can also be classified as organic or inorganic.

Total Solids is the term applied to the material residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature.

Measurement of Solids can be made in different water samples (industrial, domestic and drinking water) and it is defined as residue upon evaporation of free water.

Thus, **Total solids are nothing but summation of total dissolved solids and total suspended 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 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.

In the case of water:

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.

In the case of waste water:

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.

PRINCIPLE

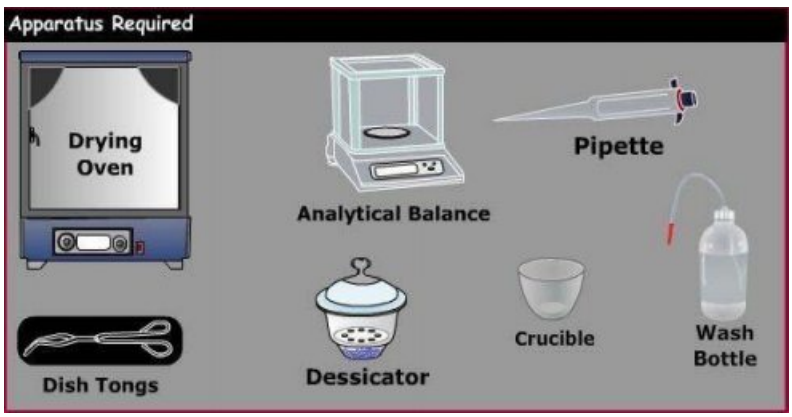
The sample is evaporated in a weighed dish on a steam bath and is dried to a constant mass in an oven either at 103-105□C or 179-181□C.

Total solids/residue is calculated from increase in mass.

MATERIALS REQUIRED

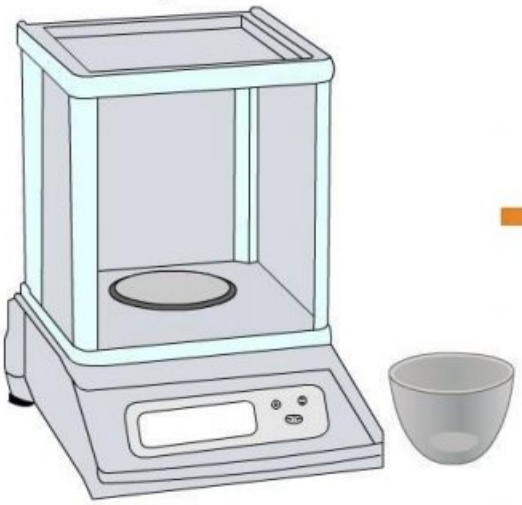
APPARATUS REQUIRED

1. Crucible
2. Oven
3. Desiccators
4. Analytical Balance
5. Dish Tongs
6. Magnetic Stirrer
7. Wash Bottle



PROCEDURE CHART

Switch on the balance (Atleast 30 min before the test)



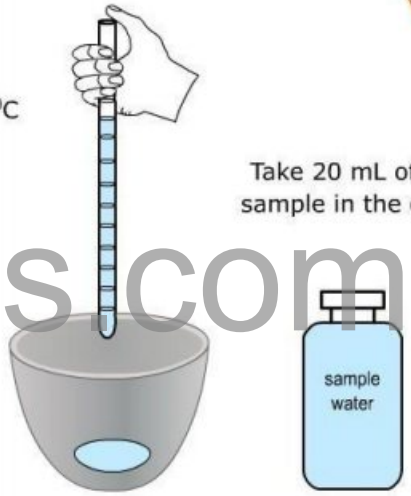
1

Notedown the initial dry weight of the crucible

2

Place the crucible inside the oven at 103^{0}C

3



Take 20 mL of water sample in the crucible

4

After drying in the oven cool to room temperature in dessicator

5

Note down the final dry weight of the crucible

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 4^0 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.

- 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.

PROCEDURE

- To measure total solids, take a clean porcelain dish which has been washed and dried in a hot air oven at 105□C for one hour.

Now weigh the empty evaporating dish in analytical balance. Let's denote the weight measured as (W1).

- Now we should have to decide what should be the volume of sample to be taken for analysis.
- Volume may be estimated either from values of specific conductance or general thumb rule.
- In general, select a sample volume that will yield residue between 2.5 and 200 mg after drying.
- Using pipette transfer 75mL of unfiltered sample in the porcelain dish.
- Switch on the oven and allowed to reach 105°C. Check and regulate oven and furnace temperatures frequently to maintain the desired temperature range.

- Place it in the hot air oven and care should be taken to prevent splattering of sample during evaporation or boiling.
- Dry the sample to get constant mass. Drying for long duration usually 1 to 2 hours is done to eliminate necessity of checking for constant mass.
- Cool the container in a desiccator. Desiccators are designed to provide an environment of standard dryness. This is maintained by the desiccant found inside. Don't leave the lid off for prolonged periods or the desiccant will soon be exhausted.
- Keep desiccator cover greased with the appropriate type of lubricant in order to seal the desiccator and prevent moisture from entering the desiccator as the test glassware cools.
- We should weigh the dish as soon as it has cooled to avoid absorption of moisture due to its hygroscopic nature.

- Samples need to be measured accurately, weighed carefully, and dried and cooled completely.
- Note the weight with residue as **(W_2).**

CALCULATION

Initial weight of the Crucible **(W_1)** = ... g
Final weight of the Crucible + sample (**W_2**) = ... g
Weight of residue **(W)** = W_2 - W_1 g

Amount of total solids present in the sample = $\frac{1000 * 1000w}{v}$

W = weight of total residue in (mg). (Therefore multiply W with 1000) V = Volume of the sample (mL)(To convert mL to L)

= ..... mg/L

The readings are required to be tabulated.

TABLE

Description		Weight (g)

DATA SHEET

Date Tested :

Tested By :

Project Name :

Sample Number :

Sample Location : Sample Description :

Description		**Weight (g)**
Initial Weight of the Crucible (g)	W_1	
Final Weight of the Crucible + sample (g)	W_2	
Weight of residue(g)	W	
Volume of the Sample (mL)	V	
Total Solids (mg/L)	**TS**	

Specimen Calculation:

W_1 = g
W_2 = g
V = mL
Weight of residue (g) W = $W_2 - W_1$
=
= g

Weight of residue in mg (To convert W (g) to W (mg), multiply W (g) with 1000) W (mg) =
= mg

Multiply the weight of the dry solids (in mg) by 1,000 mL/L to convert the sample size from mL to L.

Total Solids (mg/L)

V = Volume of the sample (mL) (To convert mL to L, multiply by 1000)

= mg/L

INTERPRETATION OF RESULTS

In the given sample, a total solid is equivalent to **mg/L.**

INFERENCE

Total solids are nothing but summation of Total Dissolved Solids and Total Suspended Solids. Regular monitoring of total solids can help detect trends that might indicate increasing erosion in developing watersheds. Total solids also affect water clarity. Total Solids may indicate the presence of agricultural activities, dredging, or mining upstream from your sample site.

EVALUATION

1. After Evaporation, the evaporating dishes needs to be
   a) weighed immediately
   b) kept in air for cooling to room temperature
   c) cooled to room temperature in a dessicator
   d) cooled to a temperature less that 25ºC

2. Total Solids are referred to materials left after evaporation.
   a) True
   b) False

3. A sample was stored @ 4 ºC for 4 days. During the analysis, the temperature of the sample should be
   a) maintained at 4 ºC
   b) brought to room temperature
   c) below room temperature
   d) brought above room temperature by adding boiled distilled water

4. The determination of total solids in wastewater gives an idea about
   a) the foulness of the sewage
   b) pH of the sewage
   c) temperature of the sewage
   d) colour of the sewage

5. The evaporating dishes needs to be cleaned and dried at _____ to remove the existing organic content.

a) 100° C
b) 250° C
c) 450° C
d) 550° C

6. Sewage contains about 99% of _____.

a) water
b) solids
c) clay
d) microbes

7. Interference in the determination of total solids is due to _____.

a) Oil and Greese
b) Large water sample
c) Dissolved salts
d) Suspended salts

8. For analysis of total solids the sample used should be

a) homogenous sample
b) supernatant of the sample
c) settled sample
d) clear sample

9. The sewage contain

a) suspended and dissolved solids.
b) no solids
c) only dissolved solids
d) only suspended solids

10. The major dissolved substances in natural water are comprised of

a) iron, manganese, silica and nitrate
b) calcium, magnesium, sodium, bicarbonate, sulfate and chloride
c) all anions
d) all cations

Ex.No. 13 **EXPERIMENT ON DETERMINATION OF TOTAL DISSOLVED AND SUSPENDED SOLIDS IN WATER**

PREAMBLE:

"**How to determine total dissolved and suspended solids in Water and Wastewater**".

Test procedure is in accordance to IS: 3025 (Part 16 & Part 17).

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

(1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 2540 C and 2540 D.

(2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 160.1.

AIM

To determine total dissolved and suspended solids in the given water sample with the stipulations as per **IS: 3025 (Part 16 & Part 17).**

INTRODUCTION

The term total dissolved solids refer to materials that are completely dissolved in water. These solids are filterable in nature. It is defined as residue upon evaporation of filterable sample. The term total suspended solids can be referred to materials which are not dissolved in water and are non filterable in nature. It is defined as residue upon evaporation of non filterable sample on a filter paper.

ENVIRONMENTAL SIGNIFICANCE

- Dissolved minerals, gases and organic constituents may produce aesthetically displeasing colour, taste and odour.
- 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.

- 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.

- 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 odours.
- 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.

PRINCIPLE

A well mixed sample is filtered through a standard glass fiber filter, and the filtrate is evaporated to dryness in a weighed dish and dried to constant weight at 179-
181 C. The increase in dish weight represents the total dissolved solids.

A well mixed sample is filtered through a weighed standard glass fiber filter and the residue retained on the filter is dried to a constant weight at 103-105 C. The increase n weight of the filter represents the total suspended solids. If the suspended material clogs the filter and prolongs filtration, the difference between the total solids and total dissolved solids may provide an estimate of the total suspended solids.

MATERIALS

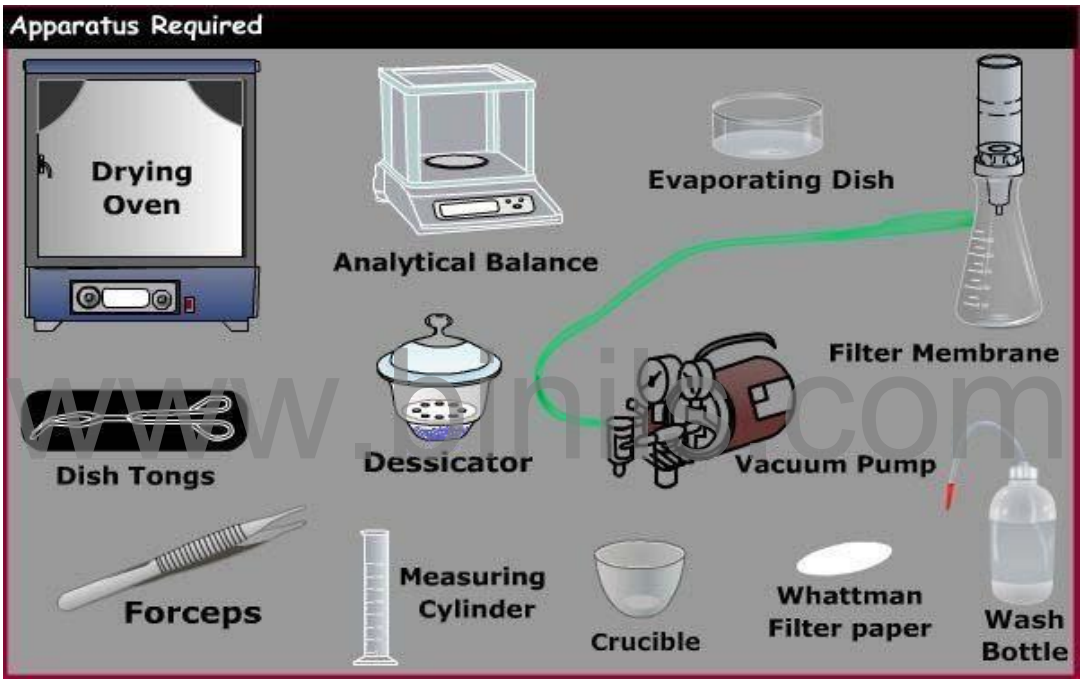
REQUIRED

APPARATUS

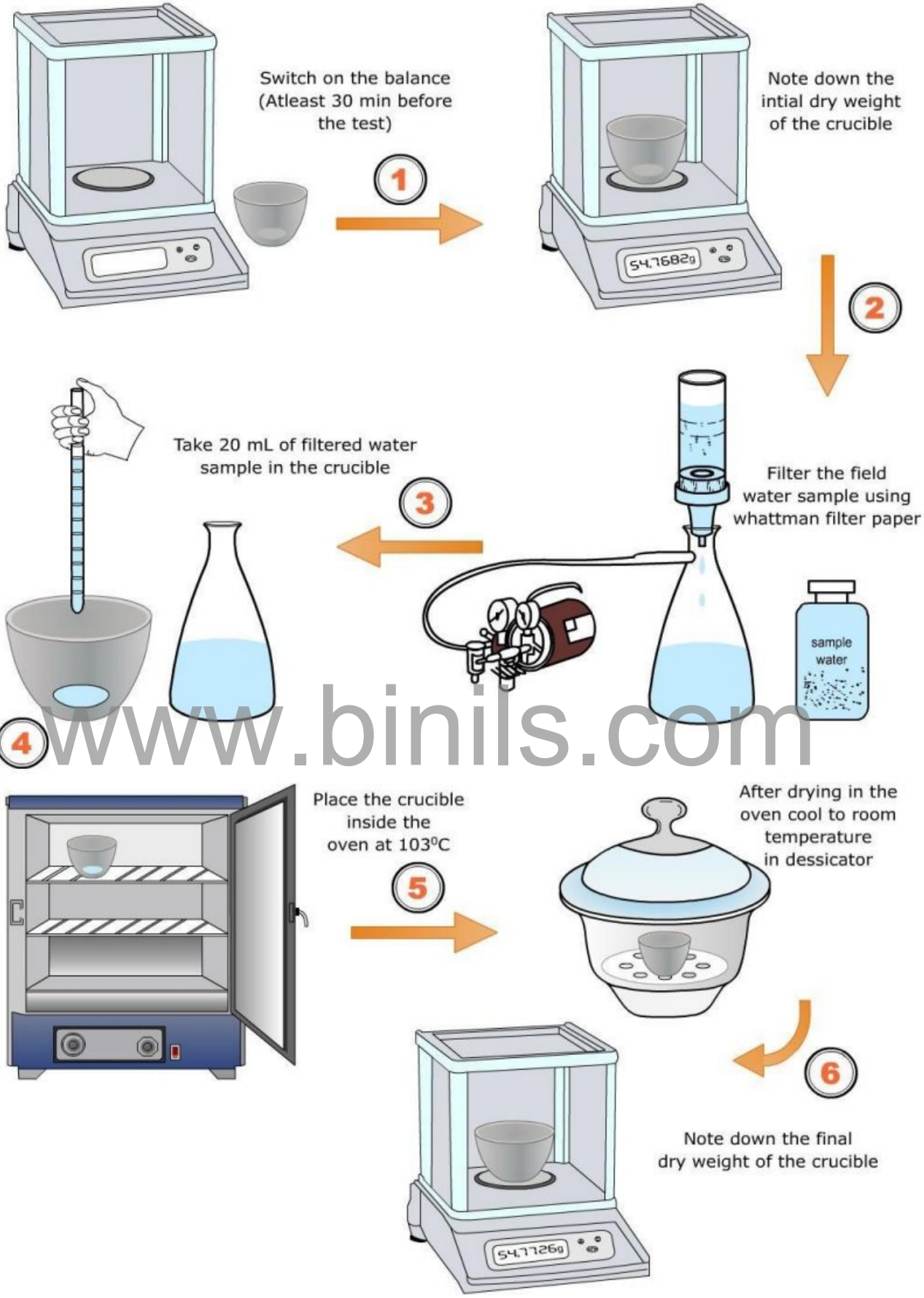
REQUIRED

1. Evaporating Dish
2. Water Bath

3. Oven
4. Desiccators
5. Analytical Balance
6. Graduated Cylinders
7. Dish Tongs
8. Gooch Crucibles
9. Filter
10. Vacuum Pumps
11. Crucible tongs
12. Forceps, Smooth -tipped



PROCEDURE CHART



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 4^0 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 sulfate 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.
- 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.
- Samples with high concentrations or bicarbonate require additional drying at 180ºC to ensure that all of the bicarbonate is converted to carbonate.

PROCEDURE

TESTING OF SAMPLE FOR TOTAL DISSOLVED SOLIDS

To measure total dissolved solids, take a clean porcelain dish which has been washed and dried in a hot air oven at 180(C for one hour.

- Now weigh the empty evaporating dish in analytical balance. Let's denote the weight measured as W1 = 35.4329 g.
- Mix sample well and pour into a funnel with filter paper. Filter approximately 80 -100 mL of sample.

- Using pipette transfer 75mL of unfiltered sample in the porcelain dish.
- Switch on the oven and allowed to reach 105°C. Check and regulate oven and furnace temperatures frequently to maintain the desired temperature range.
- Place it in the hot air oven and care should be taken to prevent splattering of sample during evaporation or boiling.
- Dry the sample to get constant mass. Drying for long duration usually 1 to 2 hours is done to eliminate necessity of checking for constant mass.
- Cool the container in a desiccator. Desiccators are designed to provide an environment of standard dryness. This is maintained by the desiccant found inside. Don't leave the lid off for prolonged periods or the desiccant will soon be exhausted. Keep desiccator cover greased with the appropriate type of lubricant in order to seal the desiccator and prevent moisture from entering the desiccator as the test glassware cools.
- We should weigh the dish as soon as it has cooled to avoid absorption of moisture due to its hygroscopic nature. Samples need to be measured accurately, weighed carefully, and dried and cooled completely.
- Note the weight with residue as W_2 = 35.4498 g.

TESTING OF SAMPLE FOR TOTAL SUSPENDED SOLIDS

- Place filtration apparatus with weighed filter in filter flask.
- Mix sample well and pour into a graduated cylinder to the selected volume.
- Apply suction to filter flask and seat filter with a small amount of distilled water.
- Pour selected volume into filtration apparatus.
- Draw sample through filter into filter flask.
- Rinse graduated cylinder into filtration apparatus with three successive 10 mL portions of distilled water, allowing complete drainage between each rinsing.
- Continue suction for three minutes after filtration of final rinse is completed.

- Dry filter in an oven at 103-105°C for at least 1 hour.
- Cool filter in desiccator to room temperature.
- When cool, weigh the filter and support.

CALCULATI

ON TABLE

Total Dissolved Solids

Description		**Weight (g)**
Weight of the clean porcelain evaporating dish (g)	W1	
Weight of the dish and the residue (g)	W2	
Weight of residue(g)	W	
Volume of the Sample (mL)	V	
Total Dissolved Solids (mg/L)	**TDS**	

Tabulation for Total Dissolved Solids (TDS):

Weight of the clean porcelain evaporating dish (g) W_1

= Weight of the dish and the residue (g) W_2 =

Weight of residue (g) W =

The volume of the sample (mL) V =

Total Suspended Solids

Description		Weight (g)
Weight of the clean filter paper (g)	W_1	
Weight of the filter paper and the residue (g)	W_2	
Weight of residue(g)	W	
Volume of the Sample (mL)	V	
Total Suspended Solids (mg/L)	**TSS**	

Tabulation for Total Suspended Solids (TSS)

Weight of the clean filter paper (g) W_1 =

Weight of the clean filter paper and the residue (g) W_2

= Weight of residue (g) W =

Volume of the sample (mL) V =

DETERMINATION OF TOTAL DISSOLVED SOLIDS DATA SHEET

Date Tested :

Tested By :

Project Name :

Sample Number :

Sample Location : Sample Description :

Description		**Weight (g)**
Weight of the clean porcelain evaporating dish (g)	W_1	
Weight of the dish and the residue (g)	W_2	
Weight of residue(g)	W	
Volume of the Sample (mL)	V	
Total Dissolved Solids (mg/L)	**TDS**	

Model Calculation:

W_1 = g
W_2 = g
V = mL

Weight of residue (g) W = $W_2 - W_1$

Weight of residue in mg (To convert W (g) to W (mg), multiply W (g) with 1000) W (mg) =

Multiply the weight of the dry solids (in mg) by 1,000 mL/L to convert the sample size from mL to L.

Total Dissolved Solids (mg/L)

V = Volume of the sample (mL) (To convert mL to L, multiply by 1000)

=

= **mg/L**

DETERMINATION OF TOTAL SUSPENDED SOLIDS DATA SHEET

Date Tested :

Tested By :

Project Name :

Sample Number :

Sample Location : Sample Description :

Description		**Weight (g)**
Weight of the clean filter paper (g)	W_1	
Weight of the filter paper and the residue (g)	W_2	
Weight of residue(g)	W	
Volume of the Sample (mL)	V	
Total Suspended Solids TDS (mg/L)		

Model Calculation:

W_1 = g
W_2 = g
V = mL

Weight of residue (g) W = W_2 - W_1
=
= g

Weight of residue in mg (To convert W (g) to W (mg), multiply W (g) with 1000) W (mg) =
= mg

Multiply the weight of the dry solids (in mg) by 1,000 mL/L to convert the sample size from mL to L.

Total Suspended Solids (mg/L)

V = Volume of the sample (mL) (To convert mL to L, multiply by 1000)

=

= **mg/L**

INTERPRETATION OF RESULTS

In the given sample, total dissolved solid is to **mg/L** and total suspended solid is to **mg/L.**

INFERENCE

Water can be classified by the amount of TDS per litre:

- fresh water < 1500 mg/L TDS
- brackish water 1500 to 5000 mg/L TDS
- saline water > 5000 mg/L TDS

The following charts give some common ranges for TSS results and possible removal efficiencies for various types of treatment.

Sample	**Common Ranges, mg/L**	
Influent	Weak < 150	400+ Strong
Primary Effluent	Weak <60	150+ Strong
Secondary Effluent	Good 10 -	60+ Bad
Tertiary Effluent	Less than 3	
Activated Sludge		
Mixed Liquor (MLSS)	1,000 - 5,000	
Return or waste sludge	2,000 - 12,000	
Digester Supernatent	3,000 - 10,000	
Sludge	20,000 - 60,000	

EVALUATION

1. The pore size of the filter paper used for filtration is

   a) 2.0µm or smaller

b) 2.0µm or bigger
c) 2.0µm
d) 20.0µm

2. The type of crucible used for the experiment is made of_____.

a) Porcelain
b) Clay
c) Silver
d) Iron

3. Total Suspended Solids are mostly responsible for

a) Turbidity.
b) colour
c) Odour
d) Taste

4. The chemical substance used in the desiccators is_____.

a) Calcium Chloride
b) Calcium Carbonate
c) Sodium Chloride
d) Sodium Hydroxide

5. Always the Total Suspended Solids value will be

a) Less than Total Dissolved Solids
b) Greater than Total Dissolved Solids
c) Less than Total Solids
d) Greater than Total Solids

6. High total dissolved solids indicates lower level of hardness.

a) True
b) False

7. The concentration of dissolved solids in water can be determined by specific conductance.

a) True
b) False

8. The settleable suspended solids with diameter 0.15 to 0.2mm are generally

a) inorganic
b) Organic
c) algae
d) fungi

9. The dissolved solids that impose BOD are_____.

a) volatile solids
b) non volatile solids
c) inorganic solids
d) total solids

10. As per IS Code the acceptable TDS value is

a) 250 ppm
b) 500 ppm
c) 750 ppm
d) 900 ppm

Ex.No.14 EXPERIMENT ON DETERMINATION OF CHEMICAL OXYGEN DEMAND

PREAMBLE:

"**How to determine chemical oxygen demand in Water and Wastewater**". Test procedure is in accordance to IS: 3025 (Part 58) - Reaffirmed 2006.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

(1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 5220 C.
(2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 410.1.

AIM

To determine chemical oxygen demand in the given water sample with the stipulations as per **IS: 3025 (Part 58) - Reaffirmed 2006**.

INTRODUCTION

Before performing this experiment, few questions may arise to the learners:

- ✓ What is meant by chemical oxygen demand?
- ✓ Why do we need to determine COD?
- ✓ What are the methods available to measure COD?
- ✓ Is it measured in water or wastewater?
- ✓ Whether is it mandatory to determine COD as per our codal provision?

The chemical oxygen demand (COD) test is commonly used to indirectly measure the amount of organic compounds in water. Most applications of COD determine the amount of organic pollutants found in surface water (e.g. lakes and rivers), making COD a useful measure of water quality. It is expressed in milligrams per liter (mg/L), which indicates the mass of oxygen consumed per liter of solution.

COD is the measurement of the amount of oxygen in water consumed for chemical oxidation of pollutants.

COD determines the quantity of oxygen required to oxidize the organic matter in water or waste water sample, under specific conditions of oxidizing agent, temperature, and time.

This method covers the determination of COD in ground and surface waters, domestic and industrial wastewaters. The applicable range is 3-900 mg/L.

ENVIRONMENTAL SIGNIFICANCE

COD values are particularly important in the surveys designed to determine and control the losses to sewer systems.

The ratio of BOD to COD is useful to assess the amenability of waste for biological treatment. Ratio of BOD to COD greater than or equal to 0.8 indicates that wastewater highly polluted and amenable to the biological treatment.

It is useful to assess strength of wastes, which contain toxins and biologically resistant organic substances.

COD can be related to TOC, however, does not account for oxidation state of the organic matter.

BOD value is always lower than COD value. For domestic and some industrial wastewater, COD value is about 2.5 times BOD value.

PRINCIPLE

The organic matter present in sample gets oxidized completely by potassium dichromate ($K_2Cr_2O_7$) in the presence of sulphuric acid (H_2SO_4), silver sulphate ($AgSO_4$) and mercury sulphate ($HgSO_4$) to produce CO_2 and H_2O. The sample is refluxed with a known amount of potassium dichromate ($K_2Cr_2O_7$) in the sulphuric acid medium and the excess potassium dichromate ($K_2Cr_2O_7$) is determined by titration against ferrous ammonium sulphate, using ferroin as an indicator. The dichromate consumed by the sample is equivalent to the amount of O_2 required to oxidize the organic matter.

MATERIALS REQUIRED

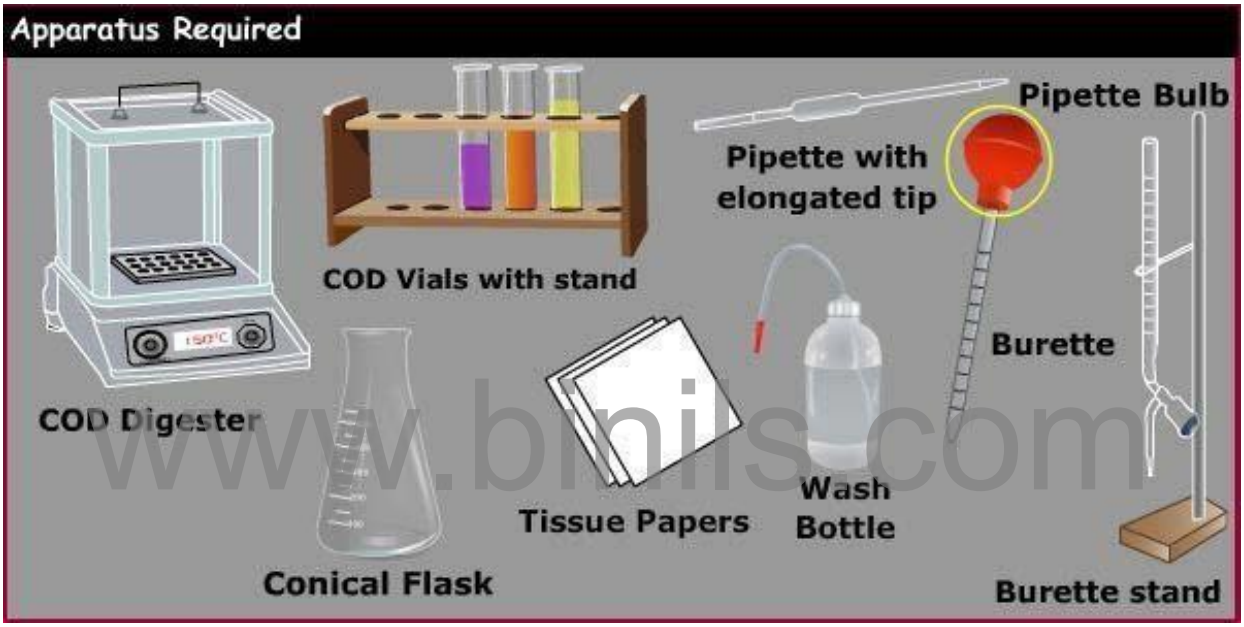
APPARATUS

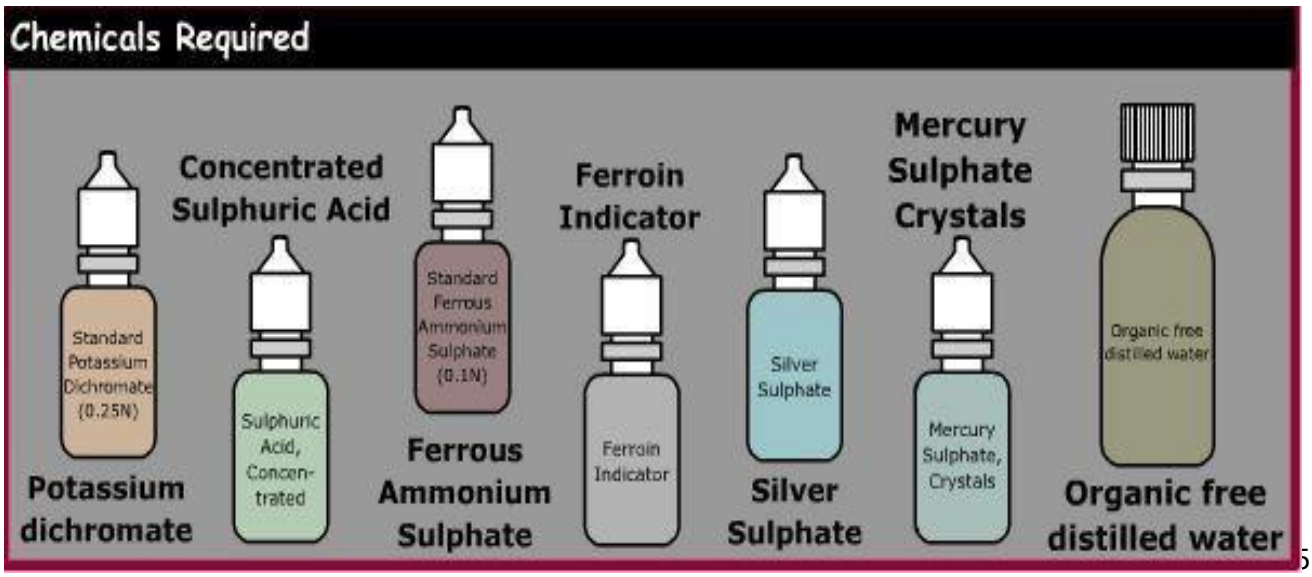
REQUIRED

1. COD Digester
2. Burette & Burette stand
3. COD Vials with stand
4. 250 mL conical flask (Erlenmeyer Flask)
5. Pipettes
6. Pipette bulb
7. Tissue papers
8. Wash Bottle

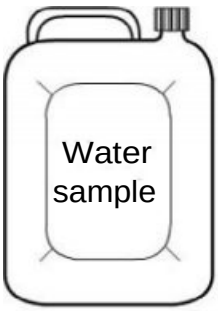
CHEMICALS REQUIRED

1. Potassium dichromate
2. Sulfuric acid
3. Ferrous ammonium sulphate
4. Silver sulphate
5. Mercury sulphate
6. Ferroin indicator
7. Organic free distilled water

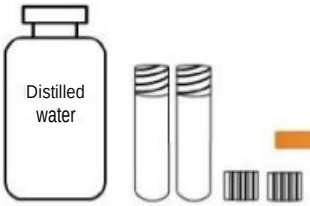




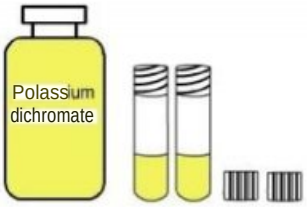
PROCEDURE CHART



Take 2.5mL water sa mple in tube and 2.5m L of distilled water another tube



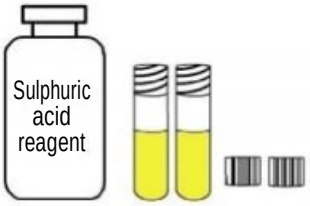
1



Add 1.5mL of Potassium dichromate to both the tubes

2

Carefully add 3.5m L of sulphuric acid reagent to both tubes

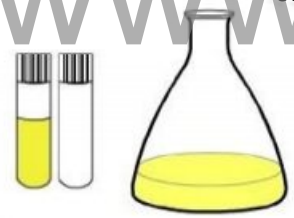


3

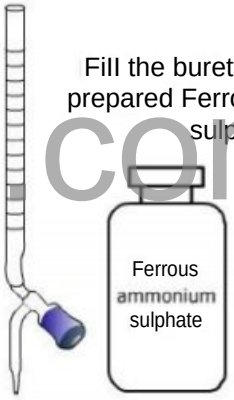
Tightly close the tubes kept in COD digestor at 150°C for 2 hours

4

After cooling to room temperature transfer the content to the conical flask



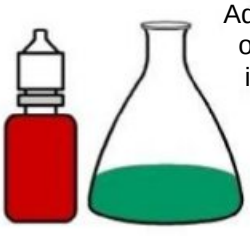
5



Fill the burette with freshly prepared Ferrous ammonium sulphate

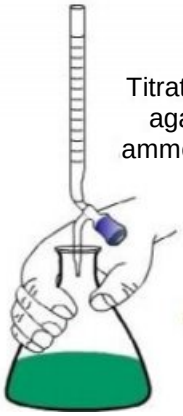
6

Add 2 drops of Ferroin indicator



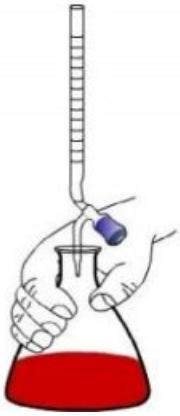
7

Titrate the contents against Ferrous ammonium sulphate



8

Continue the titration till the color changes to reddish brown



9

Calculate the COD concentration

SAMPLE HANDLING AND PRESERVATION

Samples are collected in glass bottles. Use of plastic containers is permitted if it is known that there is no organic contaminants present in it.

Biologically active samples should be tested as soon as possible. Samples containing settleable material should be well mixed, preferably homogenized, to permit removal of representative aliquots.

Samples should be preserved with sulphuric acid to a pH < 2 and maintained at 4^0 C until analysis.

Do not allow the samples to freeze.

PRECAUTIONS

The following precautions should be observed while performing the experiment:

- Chlorides are quantitatively oxidized by dichromate and represent a positive interference. Mercuric sulfate is added to the digestion tubes to complex the chlorides so that it does not interfere in the determination.
- Nitrites also interfere in the determination of COD and hence during the determination of samples with high concentration of nitrites, 120mg of sulphuric acid is added to the potassium dichromate solution.
- Traces of organic material either from the glassware or atmosphere may cause a positive error. Extreme care should be exercised to avoid inclusion of organic materials in the distilled water used for reagent preparation or sample dilution.

PROCEDURE

For testing the given sample, first the reagents are required to be prepared.

PREPARATION OF REAGENTS

a) Standard Potassium Dichromate Reagent - Digestion Solution

Weigh accurately 4.913 g of potassium dichromate, previously dried at 103ºC for 2 - 4 hours and transfer it to a beaker.

Weigh exactly 33.3g of mercuric sulphate and add to the same beaker. Measure accurately 167 mL of concentrated sulphuric acid using clean dry measuring cylinder and transfer it to the beaker. Dissolve the contents and cool to room temperature. (If not dissolved keep it over night).

Take 1000 mL standard measuring flask and place a funnel over it.

Carefully transfer the contents to the 1000 mL standard flask and make up to 1000 mL using distilled water.

This is the standard potassium dichromate solution to be used for digestion.

b) Sulphuric Acid Reagent - Catalyst Solution

Weigh accurately 5.5 g silver sulphate crystals to a dry clean 1000 mL beaker. To this carefully add about 500 mL of concentrated sulphuric acid and allow to stand for 24 hours (so that the silver sulphate crystals dissolve completely).

c) Standard Ferrous Ammonium Sulphate solution

Weigh accurately 39.2g of ferrous ammonium sulphate crystals and dissolve it in distilled water.

Take 1000 mL standard measuring flask and place a funnel over it.

Carefully transfer the contents to the 1000 mL standard flask and make up to 1000 mL mark using distilled water.

TESTING OF SAMPLE

- Take three COD vials with stopper (two for the sample and one for the blank).
- Add 2.5 mL of the sample to each of the two COD vials and the remaining COD vial is for blank; to this COD vial add distilled water.

- Add 1.5 mL of potassium dichromate reagent - digestion solution to each of the three COD vials.
- Add 3.5 mL of sulphuric acid reagent - catalyst solution in the same manner.
- **CAUTION:** COD vials are hot now.
- Cap tubes tightly. Switch on the COD Digester and fix the temperature at 150º C and set the time at 2 hours.
- Place the COD vials into a block digester at 150°C and heat for two hours.
- The digester automatically switches off. Then remove the vials and allow it to cool to the room temperature.
- Meanwhile, get ready with the burette for the titration.
- Fill the burette with the ferrous ammonium sulphate solution, adjust to zero and fix the burette to the stand.

- Transfer the contents of the blank vial to conical flask.
- Add few drops of ferroin indicator. The solution becomes bluish green in colour.
- Titrate it with the ferrous ammonium sulphate taken in the burette.
- End point of the titration is the appearance of the reddish brown colour.
- Note down the volume of ferrous ammonium sulphate solution added for the blank (A) is **mL.**
- Transfer the contents of the sample vial to conical flask.
- Add few drops of ferroin indicator. The solution becomes green in colour.
- Titrate it with the ferrous ammonium sulphate taken in the burette.
- End point of the titration is the appearance of the reddish brown colour.
- Note down the volume of ferrous ammonium sulphate solution added for the sample (B) is **mL.**

CALCULATION

For determining the Chemical Oxygen Demand in the given water sample, the readings should be tabulated.

TABLE

Sl No.	Sample	Volume of Sample (mL)	Burette Reading (mL)		Volume of 0.1 N FAS (mL)
			Initial	Final	
1.					
2.					
3.					

Burette Solution: Ferrous Ammonium

Sulphate Pipette Solution: Sample

Indicator: Ferroin Indicator

End point: Appearance of reddish brown color

DETERMINATION OF CHEMICAL OXYGEN DEMAND DATA SHEET

Date Tested :

Tested By :

Project Name :

Sample Number :

Sample Location : Sample Description :

Sl No.	Sample	Volume of Sample (mL)	Burette Reading (mL)		Volume of 0.1 N FAS (mL)
			Initial	Final	
4.					
5.					
6.					

Specimen Calculation:

Volume of Ferrous Ammonium sulphate for blank (A) = mL
Volume of Ferrous Ammonium sulphate for Sample (B) = mL
Normality of Ferrous Ammonium sulphate N = N Volume of Sample

Chemical Oxygen Demand = (A - B * N * 8 * 1000)

Volume of sample taken

To convert the sample size from mL to L, multiply the result by 1,000 mL/L to convert the sample size from mL to L.

Residual Chlorine (mg/L) =

= **mg/L**

INTERPRETATION OF RESULTS

The COD of the given sample of water = **mg/L**.

INFERENCE

Chemical oxygen demand does not differentiate between biologically available and inert organic matter, and it is a measure of the total quantity of oxygen required to oxidize all organic material into carbon dioxide and water. COD values are always greater than BOD values. For domestic and some industrial wastewater COD is about 2.5 times BOD.

EVALUATION

1. Potassium dichromate is considered as the best
    a) Oxidizing agent
    b) Reducing agent
    c) Redox agent
    d) Chemical agent

2. Mercury Sulphate is added to reduce the interference of
    a) Chlorides.
    b) Sulphates
    c) Organic pollutants
    d) Hardness

3. Silver Sulphate is added as
    a) Oxidizing agent
    b) Reducing agent
    c) Redox agent
    d) Catalyst

4. Ferroin indicator is
    a) Phenanthroline mono hydrate
    b) Ferric sulphate
    c) Phenanthroline mono hydrate and Ferric Sulphate
    d) Ferrous Sulphate

5. After refluxing,_____solution is titrated against FAS.

a) excess potassium dichromate
b) consumed potassium dichromate
c) initially added potassium dichromate
d) potassium dichromate and silver sulphate

6. H_2SO_4 is added to FAS solution

a) as it is a component of the reagent
b) to prevent hydrolysis of ferrous sulphate into ferrous hydroxide
c) to provide acidic medium
d) to neutralise the medium

7. The products formed after COD analysis are_____.

a) Carbon di oxide and water
b) Water alone
c) Carbon di oxide alone
d) Carbon monoxide and water

8. In industrial waste water, COD value is about_____BOD value.

a) 2.5 times
b) 3.5 times
c) 4.5 times
d) 5.5 times

9. Sulphuric acid is added

a) as it assists in oxidizing the nitrogen compounds
b) to provide acidic medium
c) to neutralise the medium
d) as catalyst

10. A blank solution is

a) identical in all respects to the test solution except for the absence of test solute
b) identical in all respects to the test solution
c) a solution without any reagents
d) a solution without distilled water

Ex.No.15 EXPERIMENT ON DETERMINATION OF POTASSIUM AND SODIUM IN WATER USING FLAME PHOTOMETER

PREAMBLE:

"**How to determine potassium and sodium in Water using Flame Photometer**".

Test procedure is in accordance to IS: 9497 - 1980

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

(1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 2130 B.

(2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 180.1.

AIM

To determine potassium of the given water sample using flame photometer with the stipulations as per **IS: 9497- 1980.**

INTRODUCTION

For a given element of light of wavelength other than that characteristic of the element to be determined must be eliminated before accurate intensity measurements can be made. This is accomplished by selective filtration of emitted light, by use of interference of filters, or by a suitable silt arrangement in light dispersing devices such as prism or grating monochromators.

ENVIRONMENTAL SIGNIFICANCE

Sodium is the sixth most abundant element in The Earth's crust, which contains 2,83% of sodium in all its forms. Sodium is, after chloride, the second most abundant element dissolved in seawater. The most important sodium salts found in nature are sodium chloride (halite or rock salt), sodium carbonate (trona or soda), sodium borate (borax), sodium nitrate and sodium sulfate. Sodium salts are found in seawater (1.05%), salty lakes, alkaline lakes and mineral spring water. The production of salt is around 200 million tonnes per year; this huge amount is mainly extracted from salt deposits by pumping water down bore holes to dissolve it and pumping up brine. The sun and many other stars shine with visible light in which the yellow component dominates and this is given out by sodium atoms in a high-energy state.

Potassium is an essential element and is present in all animal and plant tissues. The primary source of potassium for the general population is the diet, as potassium is found in all foods, particularly vegetables and fruits. Some food additives are also potassium salts (e.g. potassium iodide). Some individuals require potassium supplements, which are given under medical supervision; others take potassium supplements without supervision, although this is not recommended. Potassium permanganate may be used in the drinking-water treatment process. Resulting levels of potassium in drinking-water are relatively low compared with levels resulting from the use of water softeners using potassium chloride. Where potassium permanganate is used in water treatment, concentrations of added potassium can be up to a maximum of 10 mg/l, but normally concentrations would be less than this. Although concentrations of potassium normally found in drinking-water are generally low and do not pose health concerns, the high solubility of potassium chloride and its use in treatment devices such as water softeners can lead to significantly increased exposure.

PRINCIPLE

When a solution containing salts of sodium or potassium is atomized into a gas flame, light characteristic of these elements is emitted, the intensity being a function of concentration. Salts of many other elements cause similar emission. The flame photometer consists of apparatus for giving a reproducible amount of emitted light for a given concentration of element in the test solution, and for determining the intensity of such emission as a function of concentration of the element without excessive interference from other emitted light.

MATERIALS REQUIRED

APPARATUS REQUIRED

1. Flame Photometer

CHEMICALS REQUIRED

1. Con. Hydrochloric Acid
2. Con. Nitric Acid
3. Hydrofluoric Acid
4. Potassium Chloride
5. Sodium Chloride

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.

PROCEDURE

For testing the given water sample first the reagents are to be prepared. Then the Flame Photometer is required to be calibrated.

PREPARATION OF REAGENTS

1. Standard Sodium Solution

- Weigh accurately 2.542 g of sodium chloride previously dried at 120^{o} C to constant mass and cooed to room temperature in a desicator over silica gel.
- Transfer the weighed salt with water to a 250 ml beaker, dissolve in about 100 ml of water.
- Add 10 ml of hydrochloric acid.
- Transfer the solution quantitatively to one litre one mark volumetric flask and dilute to the mark.
- Mix well and store in a polyethylene bottle.
- This solution contains 1 mg/ml of sodium

2. Standard Sodium Solution

- Weigh accurately 1.9068 g of potassium chloride previously dried at 400^{o} C to constant mass and cooed to room temperature in a desiccators over silica gel.
- Transfer the weighed salt with water to a 250 ml beaker; dissolve in about 100 ml of water.
- Add 10 ml of hydrochloric acid.
- Transfer the solution quantitatively to one litre one mark volumetric flask and

dilute to the mark.

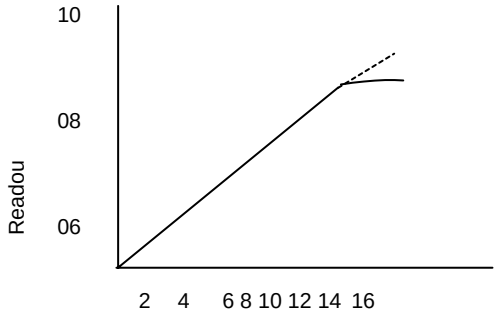
- Mix well and store in a polyethylene bottle.
- This solution contains 1 mg/ml of sodium

1. Standard Matching solution

- Into a 100 ml volumetric flask take 10 ml of the standard solution, add 2 ml concentrated hydrochloric acid and make up the volume.
- This solution contains 100 µg/ml of (sodium/potassium).
- Dilute 1,2,3,4 and 5 ml of this solution to 100 ml in 100 ml volumetric flask. The strength of the solution in these flasks as 1,2,3,4 and 5 µg/ml of sodium/potassium respectively

CALIBRATION OF TURBIDITY METER

It is important to understand that the principles of flame photometry are such that, overCertain concentration ranges, light emitted from the flame is directly proportional to the concentration of the species being aspirated. The graph below shows that the direct relationship between the flame emission and concentration is only true at relatively low concentrations. Above these low levels the flame begins to saturate and the flame emission ceases to increase in a linear relationship to concentration.



If the samples being analysed lie on the linear part of the curve then the user can take direct concentration readings from the digital display. If, however, the concentration of samples are above the levels shown on the graph then the user has the choice of either:

a. diluting the samples so that they lie on the linear part of the curve, or

b. constructing a calibration curve and relating the digital display reading to the concentration by cross-reference to the curve.

A calibration curve is prepared using standard solutions containing known concentrations of the elements to be determined and if necessary, other materials to ensure that the standard and sample backgrounds match. The concentration range covered by the calibration curve will depend upon the expected concentration of the samples so that the sample readings fall somewhere in the middle of the calibration curve. Once the calibration curve has been plotted, the readings for the sample solutions are compared with the curve to allow the sample concentrations to be established. It is important to realise that each element has its own characteristic curve and separate calibration curves must be constructed. If the same estimation is performed on a routine basis, the calibration curve need only be prepared once and checked periodically. Instrument re-calibration is easily achieved by setting the blank solution to read zero and the top standard to read the same value as it did when the calibration curve was initially prepared.

T**ESTING OF WATER SAMPLE**

- To the sample cells, add sample water up to the horizontal mark, wipe gently with soft tissue and place it in the flame photometer such that the vertical mark in the sample cell should coincide with the mark in the flame photometer and cover the sample cell.

- Check for the reading in the meter. Wait until you get a stable reading.
- The concentration of alkali metal in the given water sample is .

CALCULATION

For determining the Na and K of the given water sample the readings are required to be tabulated.

TABLE

Sample No.	Temperature of Sample (ºC)	Na Concentration (ppm)	K Concentration (ppm)
1.			
2.			
3.			

DETERMINATION OF Na & K

DATA SHEET

Date Tested :

Tested By :

Project Name :

Sample Number :

Sample Location

BH1 : Sample

Description :

Sample Location

BH2 : Sample

Description :

Sample Location

BH2 : Sample

Description :

TABULATION

Sample No.	Temperature of Sample (ºC)	Na Concentration (ppm)	K Concentration (ppm)
1.			
2.			
3.			

Result:-

Ex.No.16 EXPERIMENT ON DETERMINATION OF FLUORIDE USING ION SELECTIVE ELECTRODE

PREAMBLE:

"**How to determine fluoride in Water and Wastewater**".

Test procedure is in accordance to IS: 3025 (Part 60) - Reaffirmed 2008.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

(1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 4500-H^+ B.

(2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 150.1.

AIM

To determine the fluoride of the given water sample with the stipulations as per **IS: 3025 (Part 60) - Reaffirmed 2008**

INTRODUCTION

The electro chemical technique method is directly suitable for measuring fluoride concentration from 0.2 mg/l to 2 .0 mg/l. After the addition of a known amount of fluoride, Concentrations as low as 0 .02 mgl can be detected. The method is not suitable for waste waters and industrial effluents

ENVIRONMENTAL SIGNIFICANCE

Fluorides are important industrial chemicals with a number of uses but the largest uses are for aluminium production, drinking water fluoridation and the manufacture of fluoridated dental preparations.When fluorides are ingested by humans or laboratory animals, they are absorbed in the stomach and/or the intestine. Fluoride from soluble fluorides is almost completely absorbed (either as HF or F^-, depending on stomach acidity). However, when fluoride is bound to aluminium, calcium etc., its release and subsequent absorption may be reduced because this combination is less soluble. When fluorides in gaseous or particulate form are breathed in, the respiratory tract, they are partially or completely absorbed depending on how soluble they are or on how big the fluoride-containing particles are. Fluoride is then rapidly distributed in tissues. In humans and laboratory animals, fluorides mostly build up in bones and teeth, which retain about 99% of the total fluoride body burden. Fluoride is eliminated from the body primarily through the urine. Infants retain 80 to 90% of

fluoride ingested, while adults retain approximately 60%. However, the balance of fluoride in the body (i.e. the difference between the amount of fluoride ingested and the amount excreted) can be positive or negative. This physiological balance is determined by earlier fluoride exposure, the degree of accumulation in bone, the rate at which it is released from bone and the efficiency of the kidneys in excreting fluoride. When fluoride intakes are low excretion through urine can exceed intake.

PRINCIPLE

When a fluoride ion-selective electrode comes into contact with an aqueous solution containing fluoride ions, a potential difference develops between the measuring electrode and the reference electrode . The value of this potential difference is proportional to the logarithm of the value of the fluoride ion activity in accordance with the Nernst equation.

MATERIALS REQUIRED

APPARATUS REQUIRED

1. Minivolt meter
2. Fluoride ion selective electrode
3. Reference electrode
4. Measuring cells
5. Water bath
6. Wash Bottle
7. Magnetic stirrer
8. Polyethylene beaker

CHEMICALS REQUIRED

1. Fluoride Stock solution

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 4^0 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:

i. In general, the glass electrode, is not subject to solution interferences like color, high salinity, colloidal matter, oxidants, turbidity or reductants.

ii. 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.

iii. Before using, allow the electrode to stand in dilute hydrochloric acid solution for at least 2 hours.

iv. Electrodes used in the fluoride meter are highly fragile, hence handle it carefully.

PROCEDURE

Three major steps are involved in the experiment. They are

1. Preparation of Reagents
2. Testing of Sample

PREPARATION OF REAGENTS

1. Sodium Hydroxide 5 (M) –

- Dissolve cautiously 100 ± 0 .5 g of sodium hydroxide in water, cool and dilute to 500 ml.

2. Total Ionic Strength Adjustment Buffer (T1SAB) –

- Add 58 g of sodium chloride (NaCl) and 57 ml of glacial acetic acid to 500 ml of water in a 1 litre beaker .
- Stir until dissolved.
- Add 150 ml of the sodium hydroxide solution and 4 g of CDTA (trans-I.2-diaminocyclohexane-N,N,N' .N 'tetraacetic acid).
- Continue stirring until all the solids have dissolved and adjust the solution to pH 5 .2 with sodium hydroxide solution using a pH meter.
- Transfer to a 1000 ml one- mark volumetric flask. Make up to the mark with water and mix. Buffer Solution of pH 9.2

3. Fluoride, Stock Solution, 1000 mg/l

- Dry a portion of sodium fluoride and cool in a desiccator.
- Dissolve 2. 210 ± 0.001 g of the dried material in water contained in a 1000 ml one-mark volumetric flask, Make up to the mark with water and mix.
- Store the solution in a screw-capped polyethylene container .

4. Fluoride, working standard solution I

- 10 mg/l Pipette 10 ml of the fluoride stock solution into a 1000 ml-mark volumetric flask.
- Make up to the mark with water and mix . All standard solutions should be stored in plastic bottles and arc usable for one month .

5. Fluoride, working standard solution II

- 5 mg/l Pipette 5 ml of the fluoride stock solution into a 1000 ml-mark volumetric flask and make up to the mark with water .

6. Fluoride working standard solution III

- 1 mg/1 Pipette 100 ml of the working standard solution I into a 1000 ml-mark volumetric flask and make up to the mark with water

7. Fluoride working standard solution IV,

- 0.5 mg/l Pipette 100 ml of the working standard solution into a 1000 ml-mark volumetric flask and make up to the mark with water .

8. Fluoride working standard solution V

- 0.2 mg/l Pipette 20 ml of the working standard solution I into a 1000 ml - mark volumetric flask and make up to the mark with water.

MEASUREMENT

- Pipette 25 ml o f the buffer solution followed by 25 ml of the water sample into a measuring cell
- Ensure that the pH is 5 .2 ± 0.2; if necessary, adjust the pH with hydrochloric acid or sodium hydroxide solution using as little as possible for a series of determinations.
- Start the measurement with the lowest concentration and finish with the highest following the anticipated concentration of the samples.
- After measuring the high concentrations, recondition the electrode before measuring

the low concentrations.

- Measure all the solutions according to the following procedure. Wait until constant temperature (for example 25 ± 0.5 °C) is reached and carry out all the measurement at this temperature.
- Put a stirring bar into the measuring cell and place it on the magnetic stirrer.
- Insert the electrodes into the solution and fix them in place. Adjust the stirring rate to about 180 min/I to 200 min/I.
- When the potential does not change by more than 0.5 mV in 5 min , switch off the stirrer . After at least 15 s , record the value obtained .
- Rinse the stirring bar and the electrodes with the next solution to be measured. before starting the next measurement.

MEASUREMENT AFTER CONCENTRATION ENHANCEMENT

- If a water sample contains less than 0 .2 mg/l, proceed as follows:
    - Add 500 ml of the fluoride standard solution I to 25 ml of the sample using a piston pipette, and 25 ml of the buffer solution with a volumetric pipette; continue as described above. When calculating the result, subtract the amount of fluoride ions added from the total result.

CALCULATION

To determine the value of fluoride of the given water sample the readings obtained are required to be tabulated

TABLE

Sample No	Temperature of Sample (ºC)	Fluoride (ppm)
1.		
2.		
3.		

DETERMINATION OF FLUORIDE
DATA SHEET

Date Tested :

Tested By :

Project Name : Sample Number :

Sample Location

BH1 : Sample

Description :

Sample Location

BH2 : Sample

Description :

Sample Location

BH2 : Sample

Description :

TABULATION

Sample No	Temperature of Sample (ºC)	Fluoride (ppm)
1.		
2.		
3.		

Result:-

Ex.No.17 DETERMINATION OF AMMONIACAL NITROGEN

Aim

To determine the ammoniacal nitrogen present in the givJsamp1e.

Apparatus Required:

Burette with stand, Pipette, Conical flask, measuring flask.

Chemical required:

Oxalate, concentrated hydrochloric acid, phenolphthalein indicator, sodium hydroxide, ammonia solution.

Reagents Preparation:

Oxalate Solution:

Dissolve 630mg of oxalate in distilled water and make up to l00ml.

Phenolphthalein indicator:

Add 1g of phenolphthalein in 200 ml distilled water and dissolve it. Add 0.02N Sodium hydroxide solution drop wise until a faint pink colour appears.

Sodium hydroxide solution:

Dissolve 4g of sodium hydroxide in distilled water and make up to l00ml.

Standard Hydrochloric acid:

Dissolve 2 ml of HC1 in distilled water and make upto 100 ml.

Procedure:

Titration — I NaoH Vs Oxalic acid

1. Pipette 20ml of oxalic acid solution into the conical flask
2. Add one or two drops of phenolphthalein indicator.
3. Titrate against sodium hydroxide solution until the appearance of pink colour.
4. Repeat the titration for concordant values.

Titration —II NaoH Vs Ammonia

1. Take l7ml of distilled water in the conical flask and add lml of ammonia solution and 2 ml of hydrochloric acid.
2. Add one or two drops of phenolphthalein indicator.
3. Titrate against sodium hydroxide solution until the appearance of pink colour.
4. Repeat the titration for concordant values.

Titration — III NaoH Vs sample

1. Take 17m1 of distilled water in the conical flask and add 1ml of sample and 2 ml of hydr
2. Add one or two drops of phenolphthalein indicator.

3. Titrate against sodium hydroxide solution until the appearance of pink colour.
4. Repeat the titration for concordant values.

ENVIRONMENTAL SIGNIFICANCE

I. Excess of ammonia in the form of nitrogen leads to Eutrophication in lakes.

2. Consumption of Nitrogen greater than 2mg/I in drinking water may lead to mathemoglobonemia in children.

APPLICATION

1. Determination of ammoniacal nitrogen used for standardizing the drinking water supply.
2. The data is used in the treatment of waste water before it is subjected to water courses. It is also used to determine the extend of eutrophication and possible methods

17.8 OBSERVATION :

Source of sample :
Date of collection :
Time :
Temperature :
Tabulation

Titration – I NaoH Vs Oxalic acid

Sample details	Volume sample taken (ml)	Burette reading (ml)		Volume of NaoH used (ml)	End Point
		Initial reading	Final reading		
Oxalic acid					Appearance of pale pink colour

Concordant Value =_____ml

Titration – II NaoH Vs Sample

Sample details	Volume sample taken (ml)	Burette reading (ml)		Volume of NaoH used (ml)	End Point
		Initial reading	Final reading		
Ammonia solution					Appearance of pale pink colour

Concordant Value =_____ml

Titration – III NaoH Vs HCL						
Sample details	**Volume sample taken (ml)**	**Burette reading (ml)**		**Volume of NaoH used (ml)**		**End Point**
		Initial reading	**Final reading**			
Sewage						Appearance of pale pink colour
Concordant Value =_____ ml						

Calculation:

Titration I

Volume of oxalic acid×Normality of oxalic acid (0.1)
Normality of NaOH =

Volume of NaOH

Titration II

$$\text{Normality of Sample X} = \frac{\text{Volume of NaOH} \times \text{Normality of NaOH} \times 0.0091}{\text{Volume of sample}}$$

$$\text{Amount of Ammoniacal Nitrogen in mg/L} = \frac{X \times \text{Vol of NaOH}}{000\ \text{Volume of Sample}} \times 1$$

RESULT:

Amount of Ammoniacal Nitrogen present in the given sample is----------mg/L

Ex.No.18 EXPERIMENT ON DETERMINATION OF BIOCHEMICAL OXYGEN DEMAND

PREAMBLE:

"**How to determine biochemical oxygen demand in Water and Wastewater**". Test procedure is in accordance to IS: 3025 (Part 44) - Reaffirmed 2003.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

(1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 5210 B.

(2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 405.1.

AIM

To determine biochemical oxygen demand in the given water sample with the stipulations as per **IS: 3025 (Part 44) - Reaffirmed 2003.**

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.

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.

MATERIALS REQUIRED

APPARATUS REQUIRED

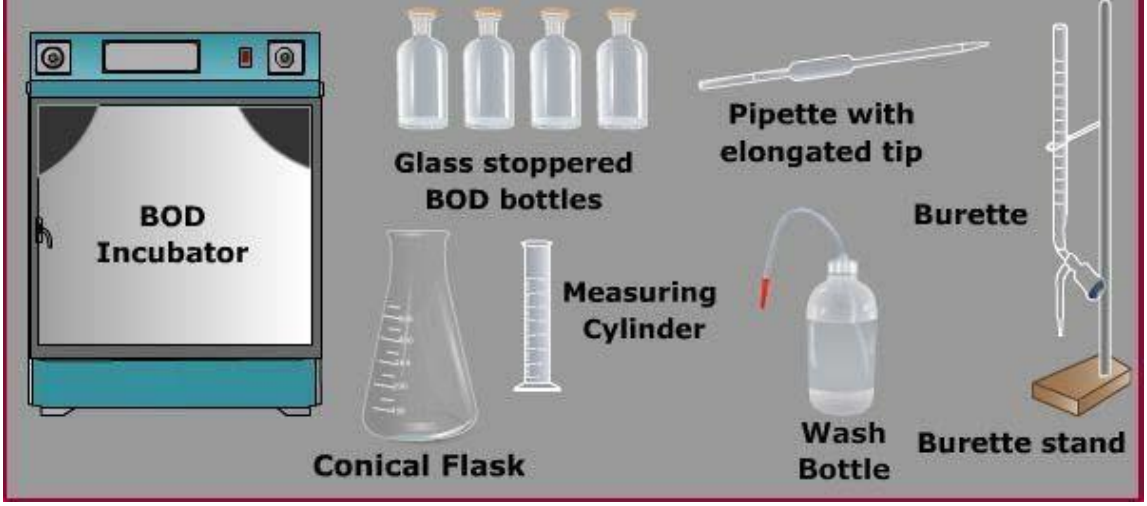
1. BOD Incubator
2. Burette & Burette stand
3. 300 mL glass stopper BOD bottles
4. 500 mL conical flask

5. Pipettes with elongated tips
6. Pipette bulb
7. 250 mL graduated cylinders
8. Wash bottle

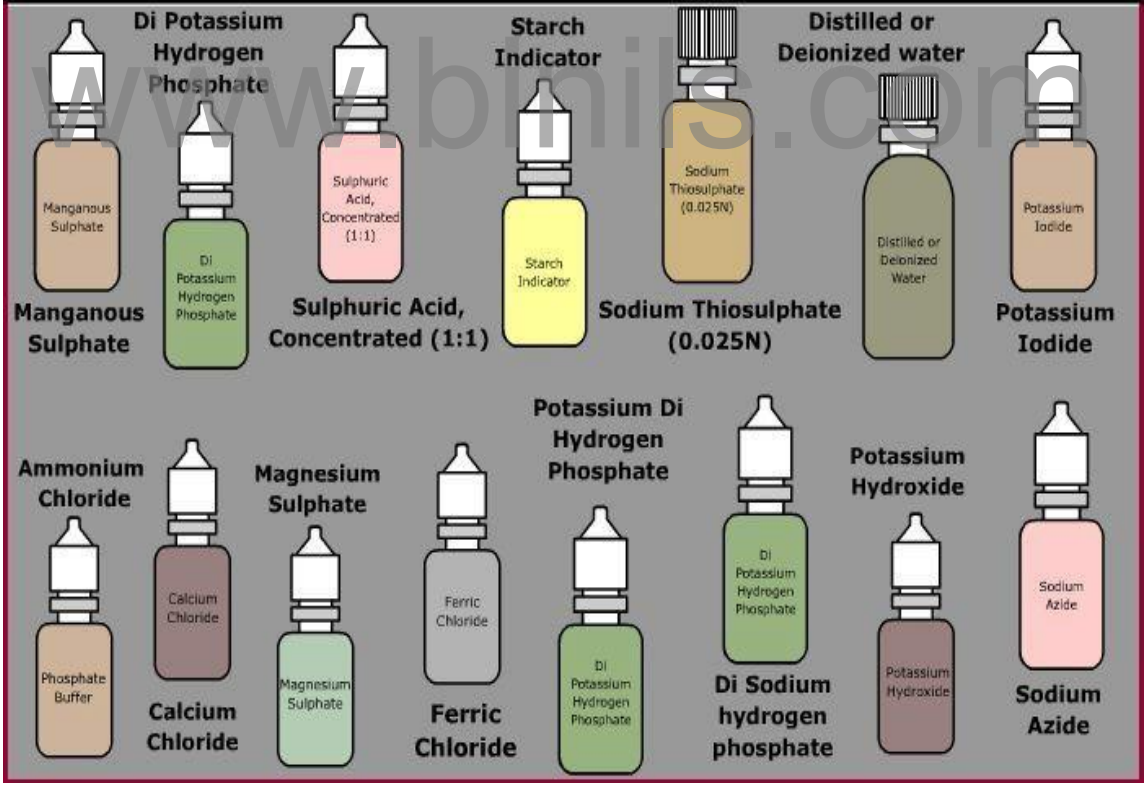
CHEMICALS REQUIRED

1. Calcium Chloride
2. Magnesium Sulphate
3. Ferric Chloride
4. Di Potassium Hydrogen Phosphate
5. Potassium Di Hydrogen Phosphate
6. Di sodium hydrogen phosphate
7. Ammonium Chloride
8. Manganous sulphate
9. Potassium hydroxide
10. Potassium iodide
11. Sodium azide
12. Concentrated sulfuric acid
13. Starch indicator
14. Sodium thiosulphate
15. Distilled or deionized

Apparatus Required

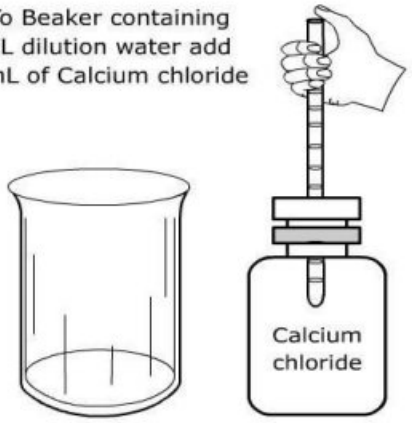


Chemicals Required



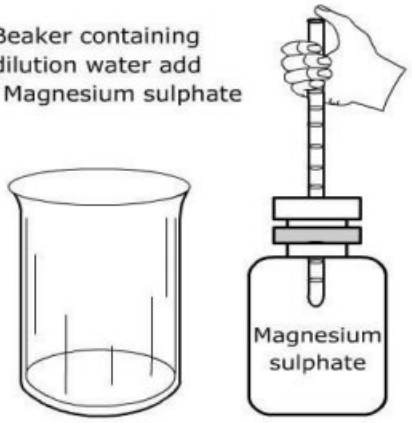
PROCEDURE CHART

To Beaker containing
5L dilution water add
5mL of Calcium chloride



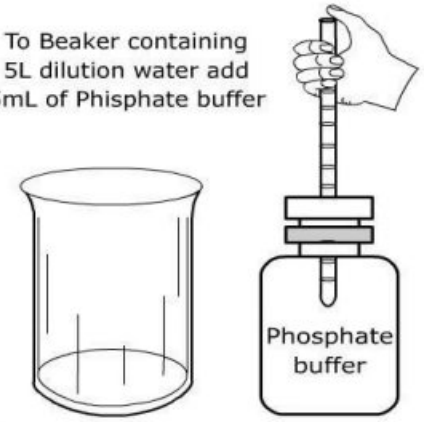
1

To Beaker containing
5L dilution water add
5mL of Magnesium sulphate



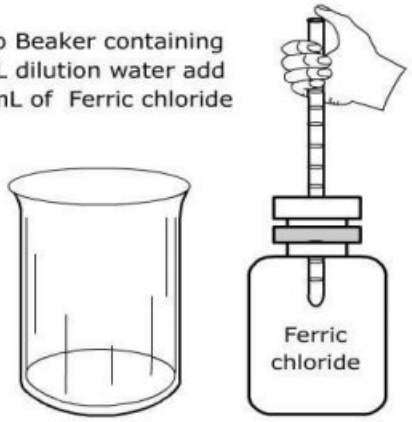
2

To Beaker containing
5L dilution water add
5mL of Phisphate buffer

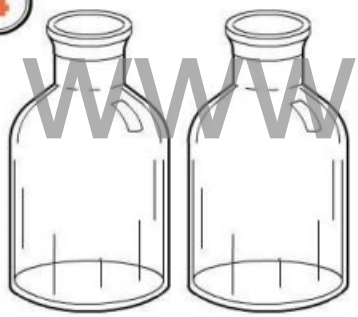


3

To Beaker containing
5L dilution water add
5mL of Ferric chloride



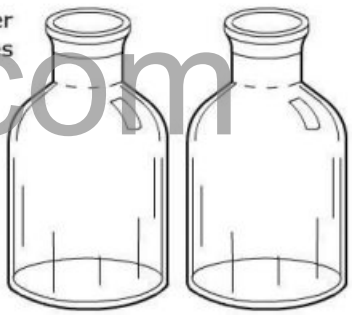
4



Add 30 mL of
field sample and
remaining 270mL
of dilution water
(10 times dilution)
to 2 BOD bottles

5

Take 300mL
of dilution water
in 2 BOD bottles
(blank)



Remaining
2 BOD bottles
of the blank and the
sample needs to be
kept in BOD incubator
at 20°C for 5 days.
After which the blank
and the sample needs
to be titrated
as per the procedure
given in DO chart.

7

6

Each one of the blank and the
sample needs to be titrated
immediately as per the procedure
given in DO chart.

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 can not 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

PRECAUTIONS

The following precautions should be observed while performing the experiment:

- Prepare dilution water 3 to 5 days before initiating BOD test to ensure that the BOD of the dilution water is less than 0.2 mg/L. **Discard dilution water if there is any sign of biological growth.**
- The sample should be adjusted to a pH between 6.5 and 7.5, using sulfuric acid for samples with pH in the alkaline side i.e., greater than 7.5 or sodium hydroxide for samples with pH in the acidic side i.e., less than 6.5.
- Add sodium sulfite (Na_2SO_3) to remove residual chlorine, if necessary. Samples containing toxic metals, arsenic, or cyanide often require special study and pretreatment.

- While still letting sample water flow down the tube, slowly pull the tube from the bottom of the bottle and fill the bottle to its brim. Check for bubbles. Carefully stopper the BOD bottle as described above.

PROCEDURE

For testing the given sample, first the reagents are required to be prepared.

PREPARATION OF REAGENT

a) Manganous Sulphate Solution

Dissolve Manganese Sulphate

$\rightarrow$ 480g of $MnSO_4.4H_2O$ (or)

$\rightarrow$ 400g of $MnSO_4.2H_2O$ (or)

$\rightarrow$ 364 g of $MnSO_4.H_2O$

in freshly boiled and cooled distilled water, filter the solution and make up to 1000 mL (One litre). In this experiment, we are using Manganese sulphate Mono hydrate.

Take 364g $MnSO_4.H_2O$ of and transfer it to the beaker. To dissolve the content, place it in the magnetic stirrer

Note: The solution should not give blue color by addition of acidified potassium iodide solution and starch.

b) Alkaline Iodide Sodium Azide Solution

To prepare this reagent we are going to mix three different chemicals

Dissolve either

→ 500 g of Sodium Hydroxide (or)
→ 700 g of Potassium Hydroxide
→ 135 g of Sodium Iodide (or)
→ 150 g of Potassium Iodide

To prepare this reagent, take 700 g of potassium hydroxide and add 150 g of potassium iodide and dissolve it in freshly boiled and cooled water, and make up to 1000 mL (One litre).

Dissolve 10 g of Sodium Azide (NaN_3) in 40 mL of distilled water and add this with constant stirring to the cool alkaline iodide solution prepared.

c) Sodium Thiosulphate stock solution

Weigh approximately 25 g of sodium thiosulphate $(Na_2S_2O_3.5H_2O)$ and dissolve it in boiled distilled water and make up to 1000 mL. Add 1 g of sodium hydroxide to preserve it.

d) Starch Indicator

Weigh approximately 2 g of starch and dissolve in 100 mL of hot distilled water. In case if you are going to preserve the starch indicator add 0.2 g of salicyclic acid as preservative.

e) Sulphuric Acid

f) Calcium Chloride solution

Weigh accurately 27.5 g of anhydrous calcium chloride and dissolve it in distilled water.

Take 100 mL standard measuring flask and place a funnel over it.

Transfer it to the 100 mL standard flask and make up to 100 mL using distilled water.

g) Magnesium Sulphate solution

Weigh accurately 22.5 g of magnesium sulphate and dissolve it in distilled water. Take 100 mL standard measuring flask and place a funnel over it.

Transfer it to the 100 mL standard flask and make up to 100 mL using distilled water.

h) Ferric Chloride solution

Weigh accurately 0.15 g ferric chloride and dissolve it in distilled water. Take 100 mL standard measuring flask and place a funnel over it.

Transfer it to the 100 mL standard flask and make up to 100 mL using distilled water.

i) Phosphate buffer solution

Weigh accurately 8.5g of Potassium Di Hydrogen Phosphate (KH_2PO_4) and dissolve it in distilled water.

Then add exactly 21.75 g of Di Potassium Hydrogen Phosphate (K_2HPO_4) and dissolve it.

To the same beaker 33.4 g of Di sodium hydrogen phosphate ($Na_2HPO_4\ 7H_2O$), is weighed and added.

Finally to the beaker containing all the salts, add accurately 1.7 g of Ammonium Chloride (NH_4Cl) and dissolve it.

Take 1000 mL standard measuring flask and place a funnel over it.

Transfer it to the 1000 mL standard flask and make up to 1000 mL using distilled water. The pH should be 7.2 without further adjustment.

j) Dilution Water

High quality organic free water must be used for dilution purposes.

The required volume of water (five litres of organic free distilled water) is aerated with a supply of clean compressed air for at least 12 hours. Allow it to stabilize by incubating it at 20ºC for at least 4 hours.

For the test we have taken five litres of organic free aerated distilled water, hence add 5mL each of the nutrients.

- Add 5mL calcium chloride solution
- Add 5mL magnesium sulphate solution
- Add 5mL ferric chloride solution and

- Add 5mL phosphate buffer solution

This is the standard dilution water. Prepare dilution water 3 to 5 days before initiating BOD test to ensure that the BOD of the dilution water is less than 0.2 mg/L.

TESTING OF SAMPLE

- Take four 300 mL glass stoppered BOD bottles (two for the sample and two for the blank).
- Add 10 mL of the sample to each of the two BOD bottles and the fill the remaining quantity with the dilution water. i.e., we have diluted the sample 30 times.
- The remaining two BOD bottles are for blank, to these bottles add dilution water alone.
- After the addition immediately place the glass stopper over the BOD bottles and note down the numbers of the bottle for identification.
- Now preserve one blank solution bottle and one sample solution bottle in a BOD incubator at 20ºC for five days.
- The other two bottles (one blank and one sample) needs to be analysed immediately.

Avoid any kind of bubbling and trapping of air bubbles. Remember – no bubbles!

- Add 2mL of manganese sulfate to the BOD bottle by inserting the calibrated pipette just below the surface of the liquid.
- Add 2 mL of alkali-iodide-azide reagent in the same manner.
- (The pipette should be dipped inside the sample while adding the above two reagents. If the reagent is added above the sample surface, you will introduce oxygen into the sample.)
- Allow it to settle for sufficient time in order to react completely with oxygen.
- When this floc has settled to the bottom, shake the contents thoroughly by turning it upside down.
- Add 2 mL of concentrated sulfuric acid via a pipette held just above the surface of the sample.
- Carefully stopper and invert several times to dissolve the floc.

- Titration needs to be started immediately after the transfer of the contents to Erlenmeyer flask.
- Rinse the burette with sodium thiosulphate and then fill it with sodium thiosulphate. Fix the burette to the stand.
- Measure out 203 mL of the solution from the bottle and transfer to an Erlenmeyer flask.
- Titrate the solution with standard sodium thiosulphate solution until the yellow color of liberated Iodine is almost faded out. (Pale yellow color)

Add 1 mL of starch solution.

- and continue the titration until the blue color disappears to colourless.
- Note down the volume of sodium thiosulphate solution added , which gives the D.O. in mg/L. Repeat the titration for concordant values.
- After five days, take out the bottles from the BOD incubator and analyse the sample and the blank for DO.
- Add 2mL of manganese sulfate to the BOD bottle by inserting the calibrated pipette just below the surface of the liquid.
- Add 2 mL of alkali-iodide-azide reagent in the same manner.
- If oxygen is present, a brownish-orange cloud of precipitate or floc will appear.
- Allow it to settle for sufficient time in order to react completely with oxygen.
- When this floc has settled to the bottom, shake the contents thoroughly by turning it upside down.
- Add 2 mL of concentrated sulfuric acid via a pipette held just above the surface of the sample.
- Carefully stopper and invert several times to dissolve the floc.
- Titration needs to be started immediately after the transfer of the contents to Erlenmeyer flask.
- Rinse the burette with sodium thiosulphate and then fill it with sodium thiosulphate. Fix the burette to the stand.
- Measure out 203 mL of the solution from the bottle and transfer to an Erlenmeyer flask.

- Titrate the solution with standard sodium thiosulphate solution until the yellow color of liberated Iodine is almost faded out. (Pale yellow color)
- Add 1 mL of starch solution and continue the titration until the blue color disappears to colourless.
- Note down the volume of sodium thiosulphate solution added, which gives the D.O. in mg/L. Repeat the titration for concordant values.

CALCULATION

For determining the Biochemical Oxygen Demand in the given water sample, the readings should be tabulated.

TABLE

Trial No.	Day	Volume of Sample (mL)	Burette Reading (mL)		Volume of Titrant (mL) ($Na_2S_2O_3$ solution used)	Dissolved Oxygen (mg/L)
			Initial	Final		
Blank						
1.						
2.						
Blank						
1.						
2.						

Burette Solution: Sodium

Thiosulphate Pipette Solution:

Sample

Indicator: Starch

End point : Disappearance of blue color

DETERMINATION OF BIOCHEMICAL OXYGEN DEMAND DATA SHEET

Date Tested :

Tested By :

Project Name :

Sample Number :

Sample Location :

Sample Description :

Trial No.	Day	Volume of Sample (mL)	Burette Reading (mL)		Volume of Titrant (mL)	Dissolved Oxygen (mg/L)
			Initial	Final		
Blank						
3.						
4.						
Blank						
1.						
2.						

Specimen Calculation:

Initial DO of the diluted sample, D_0 = mL
DO at the end of 5 days for the diluted sample, D_5 = mL
Blank correction = $C_0 - C_5$, BC = mL
Initial DO of the blank, C_0 = mL
DO at the end of 5 days for the blank, C5 = mL

Biochemical Oxygen Demand = $\frac{\{D0 - D5 - BC\} \times \text{Volume of the diluted sample}}{\text{Volume of sample taken}}$

Biochemical Oxygen Demand (mg/L) = mg/L

INTERPRETATION OF RESULTS

The BOD of the given sample of water = **90 mg/L.**

INFERENCE

On the basis of the BOD values, the characteristics of the water and the biological activity of the incubated microflora can be determined. Effluent with high BOD levels is discharged into a stream or river; it will accelerate bacterial growth in the river and consume the oxygen levels in the river. The oxygen may diminish to levels that are lethal for most fish and many aquatic insects. As the river re-aerates due to atmospheric mixing and as algal photosynthesis adds oxygen to the water, the oxygen levels will slowly increase downstream. The biological capacity of a sewage treatment plant can be tested by comparing the BOD value of a known control solution with the BOD derived from the treatment plant.

BOD detects only the destructible proportion of organic substances and as a general principle is therefore lower than the COD value, which also includes inorganic materials and those materials which cannot be biologically, oxidized.

EVALUATION

1. Biochemical oxygen demand (BOD) is an important measure of
   a) the oxygen using potential of water and wastewater
   b) oxygen content of water and wastewater
   c) an organism's natural level of oxygen requirement
   d) a measure of the biological activity of water and wastewater

2. In BOD test, dilution water is aerated
   a) for supplementing air
   b) for cooling the sample
   c) for super saturation
   d) for diluting the sample

3. Which of the following is added as nutrient
   a) Calcium chloride
   b) Calcium sulphate
   c) Magnesium chloride
   d) Magnesium phosphate

4. Seeding is the process of addition of

a) seeds
b) live microbes
c) cold water
d) nutrients

5. After the incubation period of BOD which is 5 days at 20°C,

a) all the organic content would be exhausted.
b) all organisms present will die
c) practical convenience
d) all the nutrients would be exhausted.

6. In a treatment plant when the influent BOD is 245 mg/L and the effluent BOD is 22 mg/L, the percentage of BOD removed is

a) 19%
b) 91%
c) 9%
d) 86%

7. The reaction that occurs between iodine and sodium thiosulphate result in_____.

a) Sodium iodide
b) Disodium iodide
c) Disodium thioiodide
d) Sodium thio iodide

8. Manganous hydroxide takes up dissolved oxygen in molecular form to form Manganous oxide.

a) Manganous oxide
b) Manganous di oxide
c) Manganic di oxide
d) Manganic oxide

9. Sulphuric acid is added to

a) reduce tetravalent manganese to trivalent manganese
b) reduce tetravalent manganese to divalent manganese
c) reduce tetravalent manganese to manganese
d) make acidic pH

10. The increased level of BOD in water indicate that

a) it is not fit for potable use
b) it is fit for potable use
c) it tastes better
d) it smells pleasant

ANNEXURE I

BACTERIOLOGICAL EXAMINATION OF WATER

OBJECTIVES

1. Carry out a presumptive test for the presence of coliform bacteria in a water sample
2. Determine the most probable number (MPN) of bacteria in a positive presumptive sample
3. Carry out a confirmed test to begin isolation of bacterial colonies
4. Carry out a completed test using a Gram stain and morphology
5. Write up a water use recommendations for a regulatory agency

INTRODUCTION

Fresh-water streams, lakes and ground water to be polluted when some condition makes the water unsafe for human recreation or consumption. We usually think of two forms of pollution: toxic chemicals or pathogenic microorganisms. Probably the largest single source of potentially pathogenic microbes is animal feces (including human), which contains billions of bacterial per gram. Although most intestinal microbes are non- pathogenic, some cause enteric disease. The organisms which cause typhoid fever (*Salmonella typhi*), cholera (*Vibrio cholera*), and bacterial dysentery (*Shigella flexneri*) are examples of enteric diseases caused by bacteria. In addition, some viral and protozoan pathogens are spread through water contaminated by feces.

How can we know if a water sample contains any of these pathogens? To test for each organism separately would be extremely costly and time-consuming. Therefore, a simple rule is followed: if a water sample contains any microorganism common to animal intestines, it should not be consumed, because it may contain enteric pathogens.

Water testing for microbiological safety rests on the ability of microbiologists to detect coliform bacteria. The word “coliform” refers to any bacterium that is like *Escherichia coli* in the following characteristics: 1) it is a small, gram-negative rod; 2) it does not contain spores; 3) it ferments lactose with the production of acid and gas; 4) it produces a green metallic sheen on EMB agar.

E. coli, which is found in large numbers in the feces of all animals, lives longer in water than most intestinal pathogens do. Therefore, if no *E. coli* are present, there should be no intestinal pathogens present in the water sample. For this reason, testing for coliform organisms is performed daily by municipal water departments and waste-water (sewage) treatment plants.

The bacterial examination of water to ascertain its potability (suitablity for drinking) has been standardized into three tests. The first, or presumptive test, is a screening test to sample water for the presence of coliform organisms. A series of lactose fermentation tubes are inoculated with the water sample. If the presumptive test is negative, no further testing is performed, and the water source is considered microbiologically safe for drinking. If, however, any tube in the series shows acid and gas, the water is considered unsafe and the confirmed test is performed on the tube displaying a positive reaction. The presumptive test is also designed to estimate the concentration of coliform organisms, called the most probably number (MPN) in the water sample. The confirmed test is a second screening procedure in which a gram-negative selective medium is used (like EMB). This also allows for the differentiation of coliform (producing a green metallic sheen) from non-coliform colonies. The completed test is performed on a typical, well- isolated colony to reaffirm gas production in lactose, and to determine the morphology and gram reaction of the isolate from a nutrient agar slant.

MATERIALS:

Presumptive Test and MPN

3 triple strength lactose tubes with brom-thymol blue
6 regular strength lactose tubes with brom-thymol blue
Sterile water collection bottles for community sampling (or “doped” water samples) 10 ml and 1 ml sterile pipets

Confirmed

Test 1 EMB
agar plate

Completed

Test

1 lactose tube with brom-thymol blue, 1 NA slant, Gram stain reagents

PROCEDURE

PRESUMPTIVE TEST AND MPN

1. Collect approximately 50 ml of water to be tested (from pools, streams, ocean, etc.) or you may use the “doped” samples provided in class. Record the source and date of community samples or the sample number of the “doped” sample to be tested.
2. Vigorously shake the water sample to be tested by moving it 25 times through a 12-inch arch.
3. Transfer 10 ml of the sample into each of the three, triple strength lactose tubes. Always use aseptic technique in the water inoculations and label the tubes with the amount of water sample tested.

4. Transfer 1 ml of the sample into each of three regular strength lactose tubes. Using the same pipet, transfer 0.1 ml to each of the three remaining regular strength lactose tubes.
5. Incubate all tubes at 37o C. until the next laboratory session.

CONFIRMED TEST

1. Examine the tubes from the presumptive test and determine if any has produced an acid/gas reaction. If so, this is a positive presumptive test. If no gas is present in any of the Durham tubes, this is a negative presumptive test.
2. From any tube showing 10% gas production or more, streak one loopful of the broth onto an EMB plate using the isolation streaking technique. Incubate the plate at 37o C. until the next laboratory session.
3. Determine the number of tubes positive for acid/gas in each of the three volume categories. Determine the MPN of your water sample by comparing these numbers to the MPN Determination chart accompanying this exercise. Complete the table for the presumptive test as shown below and enter it into your laboratory notebook.

COMPLETED TEST

1. Draw or describe your EMB plate in your laboratory notebook. Carefully examine the plate, looking for well-isolated coliform colonies. Typically, *E. coli* colonies appear with a metallic green sheen on EMB. From one of these colonies, set up your completed test by inoculating a lactose fermentation tube and a NA slant. Incubate them at 37o C. until the next laboratory session.

2. After this incubation, check the lactose tube for acid and gas production. If no gas is present, this is a negative completed test. Prepare a Gram stain from the NA slant. If the organism is a non-spore producing Gram negative rod and the lactose broth shows an acid/gas reaction, this is a positive completed test. Record your results in the table below in your laboratory notebook. Include a drawing of your Gram stain.