

GUDLAVALLERU ENGINEERING COLLEGE

(An Autonomous Institute with Permanent Affiliation to JNTUK, Kakinada)
Seshadri Rao Knowledge Village, Gudlavalleru – 521356, Krishna District (A.P.)

Department of Civil Engineering



WATER AND WASTEWATER ENGINEERING LAB



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GUDLAVALLERU ENGINEERING COLLEGE
SESHADRI RAO KNOWLEDGE VILLAGE: GUDLAVALLERU
DEPARTMENT OF CIVIL ENGINEERING

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Experiment No.1

Estimation of Total, Volatile, Settleable, Dissolved, Suspended Solids

Aim:

To determine the total solids, dissolved solids and settleable solids

Apparatus and equipment:

- a. Electrically heated temperature controlled oven
- b. Weighing balance
- c. Evaporating dish
- d. Pipettes
- e. Measuring cylinder (100mL)
- f. Muffle furnace
- g. Whatmann filter paper (No. 42)

Theory

The term 'solid' refers to the matter either filterable or non-filterable that remains as residue upon evaporation and subsequent drying at a defined temperature. Further categorization depends upon depends upon the temperature employed for drying and ignition. Different forms of solids are defined on the basis of method applied for their determination. Solids may affect water or effluent quality adversely in number of ways. Water with high dissolved solids may include an unfavourable physiological reaction in the transient consumer and generally are of inferior palatability. Highly mineralized waters are unsuitable for many industrial applications. High suspended solids in waters may be aesthetically unsatisfactory for such purposes as bathing. Analysis of total solids are important to decide upon the various unit operations and processes in physical and biological wastewater treatment and to assess its performance evaluation. For assessing compliance with regulatory agency, wastewater effluent limitations for various forms of solids act as indicating parameters.

A. Total solids

Principle

Residue left after the evaporation and subsequent drying in oven at specific temperature 103-105°C of a known volume of sample are total solids. Total solids include "Total suspended solids" (TSS) and "Total dissolved solids" (TDS). Whereas loss in weight on ignition of the same sample at 500°C, in which organic matter is converted to CO₂ volatilization of inorganic matter as much as consistent with complete oxidation of organic matter, are volatile solids.

Sample collection, preservation and storage

The water samples may be collected in resistant glass or plastic bottle. Water has considerable solvent property. There is possibility of increase in mineral content of sample, if water is collected and stored in non-

resistant glass bottle. The effect is pronounced with alkaline water. Exclude particles such as leaves, sticks, fish and lump of fecal matter in the sample. Begin analysis as soon as possible due to impracticality of preservation of sample.

Procedure

Total Solids

1. Take the empty weight of the thoroughly cleaned dish.(W₁)
2. Take 20 ml of a well-mixed sample in the same dish.
3. Evaporate the sample to dryness at 103-105°C in hot air oven.(4-5h)
4. Cool in desiccator, weigh and record the reading (W₂)
5. Take the same crucible and ignite the dish for 30 minutes in a muffle furnace maintained at 550°C.
6. Cool the dish in a desiccator and record final weight (W₃).
7. The concentration is to be calculated in percent by weight.

Dissolved solids

1. Take the empty weight of the dish (W₄)
2. Take 25 ml of sample and filter it using Whatmann filter paper (No. 42) using the filtration assembly.
3. Pour the filtrate into the dish and evaporate the sample to dryness at 103-105°C in hot air oven.
4. (4-5 h).
5. Cool in desiccator, weigh and record the reading (W₅)

Observation:

S.No	Details	Notations	Weight(g)
1.	Weight of empty crucible	W ₁	
2.	Weight of crucible with water sample after oven drying.	W ₂	
3.	Weight of crucible with water sample after taking it from muffle furnace.	W ₃	
4.	Weight of empty crucible	W ₄	
5.	Weight of crucible with filtrate after oven drying.	W ₅	

Calculation:

Total solids (TS) = (W₂-W₁)/volume of sample

Total dissolved solids (TDS)= (W₅-W₄) /volume of sample

Total suspended solids (TSS) = TS-TDS

Total fixed solids (TFS) = (W₃-W₁) / volume of sample

Total volatile solids = (TVS) = TS- TFS =

Total Settleable solids =

Results:

The solids present in the given wastewater are reported below

- Total solids =
- Dissolved Solids =
- Suspended solids =
- Fixed solids =
- Volatile solids =

Inference:

Experiment No.2

Determination of Chlorides

Aim: To find the amount of chlorides present in the given water sample.

Apparatus Required: Burette, Conical flask, Pipette, Measuring jar.

Theory:

Chlorides are generally present in water in the form of Sodium Chloride (Common Salt) and may be due to leaching of marine sedimentary deposits, pollution from the sea water, brine or industrial and domestic waste etc. Chloride occurs in all natural waters in widely varying concentrations. The chloride content normally increases as the mineral content increases. Upland and mountain supplies usually are quite low in chlorides, whereas river and ground waters usually have a considerable amount. Human excreta, particularly the Urine contain chloride in an amount about equal to the chlorides consumed with food and water. This amount average about 6gm of chlorides/person/day and increases the amount of Cl^- in sewage about 15mg/l above that of the carriage water.

Chlorides in reasonable concentrations are not harmful to humans. At concentrations above 250mg/l they give a salty taste to water which is objectionable to many people. For this reason, the US Public Health Service recommends that chlorides be limited to 250mg/l in supplies intended for public use. The sudden increase of chlorides in water of low chloride concentration, is indicative of organic pollution. A high chloride content also exerts a deleterious effects on metallic pipes and structures, as well as on agricultural plants.

Principle:

Silver Nitrate react with chloride to form very slightly soluble white precipitate of AgCl . At the end point when all the chlorides get precipitated free silver ions react with chromate to form reddish brown colour.

Reagents:

Silver Nitrate (0.014N), Potassium Chromate

Procedure:

- Take 20 ml of water sample in a conical flask.
- Add 1-2 ml Potassium chromate indicator solution, then colour of the sample changes to yellow.
- Titrate the sample against 0.014N silver nitrate solution until the colour changes from yellow to brick red
- Record the volume of silver nitrate used.
- Repeat the process until two successive consecutive readings.

Observations Table:

Sl. No	Volume of sample (ml)	Burette Reading (ml)		Volume of Silver Nitrate used (A-B)(ml)
		Initial Reading (A)	Final Reading (B)	

Calculation:

$$\text{Chlorides, mg/l} = \frac{\text{ml of AgNO}_3 \times \text{Normality of AgNO}_3 \times \text{Eq.wt of chlorine} \times 1000}{\text{ml of Sample}}$$

Result:

The amount of chlorides present in the given water sample is -----

Inference:

Experiment No.3

Determination of Optimum Coagulant dosage

Aim: To find out the optimum coagulant required to precipitate turbid particles present in the water.

Apparatus Required:

Jar test apparatus, Beakers – capacity 1000ml – 6nos, Turbidimeter, pH meter, Turbid water, pipettes, Graduated measuring cylinder – 1000ml

Reagents: Coagulant (Alum solution)

Theory:

Chemical Coagulation is an important process applied extensively in water treatment practice, particularly where surface supplies are involved. It is also used to some extent in domestic and industrial waste treatment.

Chemical coagulation of water is performed for several reasons. The major ones are removal of i) turbidity, organic and inorganic ii) colour, true, apparent iii) Harmful bacteria and other pathogens iv) algae and other plankton organisms v) taste and odour producing substances and vi) Phosphates, which serve as nutrients for growth of algae

Chemicals used for coagulation of water, sewage and industrial wastes react with water to form insoluble hydroxide precipitates.

Much of the suspended matter in water, is colloidal in nature. This is particularly true of substance that cause colour and turbidity. The colloidal matters are negatively charged, and for this reason, the salts of trivalent metals, usually iron and aluminium are used to coagulate them.

The usual coagulants used for coagulation are Aluminium Sulphate, Chlorinated Copperas, Ferrous sulphate and lime, Magnesium carbonate, Sodium Aluminate and Polyelectrolytes etc...

The reactions involved in chemical coagulation are essentially the same for both ferric and aluminium sulphates (usually called filter alum). Filter alum $[Al_2(SO_4)_3 \cdot 18H_2O]$ is most widely used. The various positive species which are formed may combine with negatively charged colloids to neutralize part of the charge on the colloidal particle

Principle:

Metal salts hydrolyze in the presence of the natural alkalinity to form metal hydroxides. The divalent cation can reduce the zeta potential while the metal hydroxides are good absorbents and hence remove the suspended particles by enmeshing them.

Procedure:

1. Take equal volume of sample in each jar.
2. Adjust the jars in such a way that moving paddles remain at the center of jars and merged in samples

3. Add increasing doses of coagulant of known strength to each jar (i.e., 2ml, 4ml, 6ml)
4. Now rotate the paddles with the help of driving unit at a constant speed of approximately 120 rpm for 2 to 3 mins
5. Reduce the speed to 40 to 60 rpm for a period of 30 mins
6. These jars are allowed to stand still for 15 mins.
7. Take the sample out of the beaker and test for turbidity in each trial
8. Plot the graph between dose of coagulant and turbidity
9. The least turbidity of the sample shows the Optimum dose of coagulant

Observations:

Volume of sample taken:

Jar No	ml of alum added	Dosage of alum (mg/L)	pH	Residual Turbidity	% Removal of Turbidity

Result:

The optimum dosage of coagulant required to remove turbidity in the given water sample is _____mg/L

Experiment No.4
DETERMINATION OF CHLORINE DEMAND

AIM: To find the residual chlorine available and chlorine demand in the given sample.

PRINCIPLE:

Orthotolidine is an organic compound that is oxidized agents to produce a yellow colored compound called "Holoquinone". Intensity of yellow color of Holoquinone is proportional to the amount of chlorine present.

INTERFERENCE:

Oxidizing agents such as ferric compounds, manganic salts, nitrites etc.

APPARATUS:

Chloroscope.

REAGENT:

Orthotolidine reagent.

PROCEDURE:

1. Take the water sample under question into one of the cylinders of the comparator and distilled water into the other.
2. Add 5 drops of Orthotolidine solution to both the cylinders and put them in the comparator.
3. The color which matches in both the cylinders directly gives the residual chlorine.

OBSERVATION AND RESULTS:

Amount of residual chlorine (mg/l)= mg/l

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 a bacterologically impeccable water.
- Active chlorine should be present in drinking water within the range of 0.1 to 0.2 mg/l. However excessive chlorine content may give out bad odor and may change even taste of water. Further chlorine is said to be earainogenous. Hence exceptduring epidemic 'super chlorination' not to be done.
- Determination of chlorine residuals is used universally in disinfection practice to controladdition of chlorine so as to ensure effective disinfection without taste
- 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.

RESULT:

Amount of residual chlorine (mg/l) is

Inference:

Experiment No.5

ESTIMATION OF SETTLEABLE SOLIDS USING IMHOFF CONE

AIM: To find out total settleable solids present in the given sample

PRINCIPLE:

The particles in suspension whose specific gravity greater than that of water will settle under quiescent conditions.

APPARATUS:

1. Imhoff cone
2. Holding device

ENVIRONMENTAL SIGNIFICANCE:

Total solids measurements can be useful as an indicator of the effects of runoff from construction, agricultural practices, logging activities, sewage treatment plant discharges, and other sources. Total solids also affect water clarity. Higher solids decrease the passage of light through water, thereby slowing more rapidly and hold more heat; this, in turn, might adversely affect photosynthesis by aquatic plants. Water will heat up affect aquatic life that has adapted to a lower temperature regime. As with turbidity, concentrations often increase sharply during rainfall, especially in developed watersheds. They can also rise sharply during dry weather if earth disturbing activities are occurring in or near the stream without erosion control practices in place. Regular monitoring of total solids can help detect trends that might indicate increasing erosion in developing watersheds. Total solids are related closely to stream flow and velocity and should be correlated with these factors. Any change in total solids over time should be measured at the same site at the same flow.

1. 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.

2. 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.

PROCEDURE:

1. Gently fill the imhoff cone with the thoroughly well mixed sample usually 1 liter and allow it to settle.
2. After 45 minutes gently rotate the cone between hands to ensure that all solids adhering to the sides are loosened.
3. Allow the solids to settle for 15 minutes more, to make up for a total period 1 hour
4. Read the volume of the sludge which has settled in the apex.
5. Express the results in ml settle able solids per liter of sample per hour

CALCULATION:

$$\text{Total Settle able Solids (ml/L)} = \frac{\text{ml of solids} * 1000}{\text{ml of sample}}$$

PRECAUTION:

1. The imhoff cone must be clean with a strong soap and hot water using the brush.
2. Wetting the cone with water before use helps in preventing adherence of solids to the sides.
3. The method is subjected to considerable inaccuracy if the solids contain large fragments.
4. The determination of total settle able solids should be carried out soon after sampling in order to avoid errors through flocculation

OBSERVATIONS & RESULTS:

Sample details	Volume of sample taken (ml)	Total settleable solids (ml/l/hr)

RESULTS:

Total settleable solids present in the given sample is =

Experiment No.6

DETERMINATION OF DISSOLVED OXYGEN WITH D.O. METER

AIM: To determine the Dissolved Oxygen present in the given water sample.

INTRODUCTION:

The reagent $MnSO_4$ reacts with the alkaline to form $Mn(OH)_2$ (white precipitate). If oxygen is present, it will oxidize $Mn(OH)_2$ to $MnO(OH)_2$ (brown precipitate). If H_2SO_4 is added the brown precipitate is dissolved to form $MnSO_4$ which immediately reacts with KI already added and liberates Iodine equivalent to the quantity of oxygen originally present in the sample. The amount of iodine is estimated by titration against sodium thiosulphate solution, using starch indicator.

EQUIPMENT AND REAGENTS:

1. Dissolved oxygen sampler
2. 300ml glass stopper BOD bottle
3. Manganese sulphide
4. Alkaline Iodide Azide reagent
5. Sulphuric acid (H_2SO_4)
6. Starch solution
7. Sodiumthiosulphate solution (0.025N)

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.

PROCEDURE:

1. Take a glass stopper BOD bottles of known capacity and fill it with sample to avoid air bubbles.
2. No air should be trapped in the bottle after stopper is placed. Open the bottle and add 2ml of Manganous sulphate and add 2ml of Alkali Iodide Azide using separate pipette.
3. Place the stopper and shake the bottle thoroughly until a brown precipitate formed.
4. Add 2ml of Conc. H_2SO_4 to dissolved the precipitate.
5. Take 203ml sample in a conical flask

6. Put a few drops of starch indicator and titrate against 0.025N Sodium thiosulphate solution.
7. Titrate till the colour changes from blue to colourless

PRECAUTIONS:

1. Disgard any subsequent return of colour
2. If the end point is overrun back titrate with biniodate (0.025) and effect the necessary correction
3. Avoid air bubbles while stoppering the bottle

OBSERVATION AND CALCULATION:

DO in mg/l = ml of $\text{Na}_2\text{S}_2\text{O}_3$ used

RESULT:

The Dissolved oxygen in the given sample is=

Experiment No.7

DETERMINATION OF BIOLOGICAL OXYGEN DEMAND

AIM: Determination of Biological Oxygen Demand in the given water sample

INTRODUCTION:

Biological Oxygen Demand is defined as the amount of oxygen required by micro-organisms while stabilizing biologically decomposable organic matter of water under aerobic conditions. The BOD test is widely used to determine

1. The pollution load of waste water.
2. The degree of pollution in lakes, streams at any time and their self purification capacity.
3. Efficiency of waste water methods.

APPARATUS:

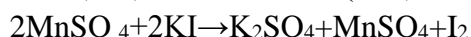
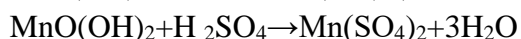
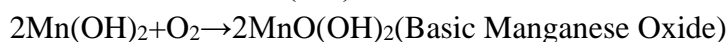
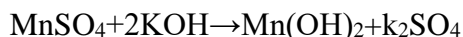
1. BOD bottles
2. Incubator
3. Conical flask
4. Analytical balance
5. Burette
6. Pipette

CHEMICALS AND REAGENTS:

1. Manganese Sulphate solution
2. Calcium Chloride
3. Ferric chloride
4. Starch Indicator
5. Sodium Thiosulphate
6. Alkaline Iodide Azide and Conc.H₂SO₄

PRINCIPLE:

- MnSO₄ is added to the water sample containing alkaline manganese hydroxide.
- It is oxidized to basic manganese oxide but the dissolved oxygen present in the water sample.
- When H₂SO₄ is added, the basic manganese oxide liberates iodine which is equivalent to the DO present in the sample.
- The liberated iodine is titrated with a standard hypo solution using starch as an indicator



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.

PROCEDURE:

1. Take BOD bottles of 300 ml capacity
2. Dilute the sample, if necessary
3. Fill the BOD bottle with diluted sample
4. Find out Initial DO of the given sample
5. Place another BOD bottle in the incubator at a constant temperature of 20°C for a period of 5 days
6. During incubation water is protected from air. This can be done by keeping the BOD bottles in incubator for 3 days or 5 days.
7. Take out the sample from incubator and let it come to room temperature
8. Determine the DO value of the sample

OBSERVATION:

	Sl.No	Sample Details	Capacity of BOD bottle for titration	Volume of sample taken	Burette reading		ml. of titrant used	D.O. in mg l
					Initial	Final		
For Initial D.O	1. 2.							
For Final D.O	1. 2.							

CALCULATIONS:

Initial DO = DO of sample on first day

Final DO = DO of sample after 5 days

BOD in the given sample is = (initial DO - final DO) / Dilution factor

RESULT:

The BOD in the given water sample is.....

Experiment no.8

DETERMINATION OF COD FOR GIVEN SAMPLE

Aim:

To determine the chemical oxygen demand (COD) in the given waste water sample.

Apparatus Required:

Refluxing Apparatus, Burette, Hot plate/Heating mantle

Principle:

The organic matter present in sample gets oxidized completely by $K_2Cr_2O_7$ in the presence of H_2SO_4 to produce CO_2 and H_2O . The excess of $K_2Cr_2O_7$ remaining after the reaction is titrated with $Fe(NH_4)_2(SO_4)_2$. The dichromate consumed gives the oxygen required for the oxidation of organic matter.

Reagents:

1. Standard Potassium Dichromate 0.25N
2. Sulphuric Acid with reagent (Conc. $AgSO_4 + H_2SO_4$)
3. Std. Ferrous Ammonium Sulphate titrant (0.1N)
4. Ferroin Indicator
5. Mercuric Sulphate crystal
6. Silver Sulphate

Procedure:

- Take 10 ml of sample.
- Place 5 ml of Conc Dichromate solution in a flask together with glassbeads.
- Add slowly 15 ml of H_2SO_4 containing $AgSO_4$ and mix thoroughly.
- Add pinch of mercurous sulphate ($HgSO_4$) and silver sulphate (Ag_2SO_4).
- Connect the flask to condenser. Mix the contents thoroughly before heating. Improper mixing may result in bumping and the sample may be blown out.
- Reflux for a minimum period of 2 hours. Cool and wash down the condenser with distilled water.
- Dilute the sample to make up to 40 ml with distilled and cool.
- Add 2-3 drops of Ferroin indicator. Mix thoroughly and titrate it against 0.1N $Fe(NH_4)_2(SO_4)_2$. Sharp colour changes from blue-green to wine red indicate the end point.
- Reflux the blank solution in the same manner using distilled water instead of sample.

Observations:

Sl.No	Volume of water sample (ml)	Burette Reading (ml)		Concurrent Burette Reading (ml)	Volume of Ferrous Ammonium Sulphate (ml)
		Initial Reading	Final Reading		

Calculation:

Quantity of Fe (NH₄)₂ (SO₄)₂ added for blank (A) =

Quantity of Fe (NH₄)₂ (SO₄)₂ added for sample (B) =

Chemical Oxygen Demand (COD) = (A-B) X N X 8 X 1000 X Dilution factor

Volume of Sample

Result:

The COD for the given water sample is -----

Inference:

The BIS standard for COD of wastewater effluent to be discharged into marine coast is 250 mg/l. The amount of COD obtained in the given sample is _____. So, this water **can/cannot** be disposed in the marine coast.

Experiment No.9

Determination of Fluorides

Aim:

To determine the fluoride concentration present in the given water sample.

Theory:

Fluorides is more common in ground water than in surface water. The main sources of fluoride in ground water are different fluoride bearing rocks. In rare instances the fluoride concentration of naturally occurring water may approach 10 mg/L. Such water should be defluoridated.

A fluoride concentration of approximately 1 mg/L in drinking water effectively reduces dental caries or tooth decay without any harmful effect on health. The actual concentration of fluoride in drinking water depends on the air temperature, because ambient air temperature influences the amount of water that people drink.

Apparatus:

Spectrophotometer for use at 570 nm, providing a light path of at least 1 cm or longer.

Nessler's tubes with capacity of 100 mL

REAGENTS:

- **Stock fluoride solution (0.221 gm/L):** Dissolve 221.0 mg anhydrous Sodium fluoride (NaF) in dissolved in distilled water and dilute to 1000 ml. 1 ml = 100 µg.
- **Standard fluoride solution:** Dilute 100 ml stock fluoride solution to 1000 ml with distilled water. (1 ml = 10µgF)
- **SPADNS solution:** Dissolve 958 mg SPADNS, [sodium 2 – (parasulfophenylazo)- 1,8-dihydroxy-3,6-naphthalene disulfonate] in distilled water and dilute to 500 ml. This solution is stable for 1 year if protected from sunlight.
- **Zirconyl-acid reagent:** Dissolve 133 mg zirconyl chloride octahydrate ($ZrOCl_2 \cdot 8H_2O$) in about 25 ml distilled water. Add 350 ml conc. HCl and dilute to 500 ml with distilled water.
- **Acid Zirconyl-SPADNS reagents:** Mix equal volumes of SPADNS solution and zirconyl acid reagent. The combined reagent is stable for at least 2 years.
- **Reference solution:** Add 10 ml SPADNS solution to 100 ml distilled water. Dilute 7ml conc HCl to 10 ml and add to the diluted SPADNS solution. The resulting solution, used for setting the instrument reference point (zero) of the spectrophotometer. This solution is very stable and may be reused indefinitely.
- **Sodium Arsenite Solution:** Dissolve 5.0 gm $NaAsO_2$ and dilute to 1000 mL with distilled water. (Caution: Sodium arsenite is toxic avoid ingestion).

Procedure:

Preparation of standard curve:

- Prepare fluoride standards in the range of 0 to 1.40 mg/L by diluting appropriate quantities of standard fluoride solution to 50 mL with distilled water (Given below Table).
- In each 50 mL of Nessler tube, add 10 mL mix solution (or 5 mL of SPANOS and 5mL zirconyl acid reagent). Mix well. Take absorbance immediately of the bleached colour at 570 nm using reference solution for setting zero absorbance.
- Plot standard graph between concentration and absorbance (optical density).

Observations:

SL.NO	CONCENTRATION	ABSORBANCE

Calculation:

Fluoride (mg/L) = (mg F determined photometrically/mL sample used) × 1000 × B/C

The ratio B/C applies only when a sample is diluted to a volume B, and a portion C taken from it for colour development.

Fluoride estimation in sample:

1. Take 50 mL filtered sample and add 10 mL of mix solution, mix well and read the absorbance immediately of the bleached colour at 570 nm using reference solution for setting zero absorbance. (Note: After adding the mix solution take reading immediately)
2. If sample contain residual chlorine, remove it by adding NaAsO₂ solution; [1 drop(0.05mL)=0.1mg Cl)]. Note: Sodium arsenite concentration of 1300 mg/L produce an error of 0.1 mg/L at 1 mgF/L).
3. If the transmission fall beyond the range of the standard curve, repeat the procedure using a smaller sample.

Result:

The amount of Fluoride present in given sample is

Inference:

Fluoride is found in all natural waters at some concentration. Seawater typically contains about 1 mg l⁻¹ while rivers and lakes generally exhibit concentrations of less than 0.5 mg l⁻¹. In groundwaters, however, low or high concentrations of fluoride can occur, depending on the nature of the rocks and the occurrence of fluoride-bearing minerals.

Experiment No.10

Determination of Sulphates

Aim:

To find the amount of Sulphates present in the given water sample.

Apparatus Required:

Conical Flask, Watt-man filter paper (No 42), crucibles, Measuring Jar, Beaker.

Principle:

Sulphate is precipitated as Barium sulphate in the hydrochloric acid medium by the addition of Barium Chloride solution. The reaction is carried out near the boiling temperature. The precipitate is filtered and then washed to remove the chlorides and ignited and weighed as Barium Sulphate.

Many substances interfere in performing this test. Suspended matter, silica, Nitrate and sulphate lead to the positive errors where the results are on the higher side. Alkali metal sulphates causes the low results. Presence of other metals such as iron and chromium also yield low results due to the formation of metal sulphate.

Reagents:

Methyl orange Indicator, Barium chloride solution, Concentrated Hydrochloric acid.

Procedure:

- Take 100 ml of sample in a conical flask. Add 2 drops of Methyl orange Indicator to the sample. Adjust pH between 4.5 — 5 by adding few drops of concHCl until the colour changes to pink.
- Add 1 ml of Conc.HCl heat at 40⁰ C until vapour appears and add Barium chloride Solution until the precipitate forms. This precipitate is heated at 90⁰ C allowing it to boil for 1 hour. Filter it, wash the precipitate with distilled water and filter it with dry ash less filter paper and ignite it in a crucible at 500- 800⁰ C for 1 hour
- Cool it in desiccator and weigh the precipitate of Barium sulphate.

Observations:

Sl.No	Description	Weight of crucible		Sulphates
		Empty weight (g)	Empty weight (g) + Filtrate	

Calculation:

$$\text{Amount of Sulphate} = \frac{\text{Mg. of Sulphate} \times 411.5}{\text{ml of Sample}}$$

Result:

The amount of Sulphates present in the given water sample is -----

Inference:

The presence of sulfate in drinking-water can also result in a noticeable taste; the lowest taste threshold concentration for sulfate is approximately 250 mg/litre as the sodium salt. Sulfate may also contribute to the corrosion of distribution systems.

Experiment No.11

Determination of Iron

AIM:

To determine the amount of ferrous iron present in the given 100ml Mohr's salt solution by using standardized KMnO_4 solution

APPARATUS:

Burette, Conical Flask, Pipette, Beaker, Glazed tile, measuring jar

CHEMICALS REQUIRED:

KMnO_4 (standardized KMnO_4 solution), given Mohr salt solution and dilute H_2SO_4 .

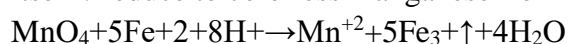
ENVIRONMENTAL SIGNIFICANCE:

Iron is believed to be the tenth most abundant element in the universe. Iron is also the most abundant (by mass, 34.6%) element making up the Earth; the concentration of iron in the various layers of the Earth ranges from high at the inner core to about 5% in the outer crust. Most of this iron is found in various iron oxides, such as the minerals hematite, magnetite, and taconite. The earth's core is believed to consist largely of a metallic iron-nickel alloy.

Iron is essential to almost living things, from micro-organisms to humans. World production of new iron is over 500 million tonnes a year, and recycled iron adds other 300 million tonnes. Economically workable reserves of iron ores exceed 100 billion tonnes. The main mining areas are China, Brazil, Australia, Russia and Ukraine, with sizeable amounts mined in the USA, Canada, Venezuela, Sweden and India.

THEORY:

KMnO_4 oxidizes ferrous iron in acidic medium and in cold condition. But KMnO_4 itself reduces to colorless manganese ion

**PROCEDURE:**

1. Pipette out 10 ml of given solution in a washed conical flask.
2. Titrate this solution against KMnO_4 taken in burette.
3. The light pink color shows the end point. Repeat the experiment to get at least two consequent readings.

OBSERVATION AND CALCULATION:

S.no	Vol Of Mohr's Solution	Burette Reading		Vol Of KMnO_4 Rundown
		Initial	Final	
1				
2				
3				

$$N_1V_1 = N_2V_2$$

N_1 = Normality of Mohr's Salt Solution

N_2 =Normality of KMnO_4 Solution

V_1 =Volume of Mohr's salt solution

V_2 =Volume of KMnO_4 Solution

$$N_1 = \frac{N_2 V_2}{V_1}$$

Therefore Strength of the given Fe^{+2} ions Solution $N_1 = \dots\dots$

RESULT: Amount of Fe^{+2} ions present in the given 100ml solution is

Wt of Fe^{+2} ion=Normality of Fe^{+2} ion

From Stage 2 *gram unit wt of Fe^{+2} ion*100/1000

Experiment No.12

Determination of phosphates

Aim

To determine the amount of phosphate present in the given wastewater sample.

Principle

In acidic conditions orthophosphate reacts with ammonium molybdate forming Molybdophosphoric acid, reduced further to molybdenum blue by stannous chloride. The intensity of the blue colour is directly proportional to the concentration of phosphate. The absorbance is noted at 690nm using spectrophotometer.

Apparatus required:

Spectrophotometer, Lab Glassware, Hot Plate, Nessler's Tube.

Reagents:

Ammonium molybdate reagent:

25g ammonium molybdate is dissolved in 175ml distilled water. 280ml concentrated sulphuric acid is added to 400ml distilled water and cooled. Molybdate solution is added and the mixture diluted to 1000ml.

Stannous chloride reagent:

2.5g fresh stannous chloride is dissolved in 100ml glycerol, heated on water bath and stirred with the glass rod to hasten dissolution.

Standard phosphate solution:

219.5 mg of dried AR potassium hydrogen phosphate is dissolved in distilled water and made up to 1000ml, where 1ml = 50.0 μ g. of phosphate. 10ml of the stock solution is made up to 1000ml to give 1ml = 0.05 mg. Standards of strength ranging from 0 (blank) to 0.05mg/L at intervals of 0.01mg is prepared by diluting the stock with distilled water

Procedure:

To 50ml of the filtered sample, 4ml of ammonium molybdate reagent and about 4-5 drops of stannous chloride reagent is added.

After about 10 min but before 12 min, the colour developed is measured photometrically at 690nm and calibration curve is prepared.

A reagent blank is always run with same treatment with distilled water as sample.

The value of phosphate is obtained by comparing absorbance of sample with the standard curve and expressed as mg/L

Observation:

Sl.NO	CONCENTRATION	ABSORBANCE

Calculation:

$$\text{Phosphates (mg/L)} = \frac{\text{Absorbance of sample} \times \text{Conc.of Std} \times 1000}{\text{Absorbance of Std} \times \text{Sample taken}}$$

Result

The amount of Phosphate present in the given sample is -----mg/l.

Inference:

Any specific permissible limit for phosphates is not available till date since it is a limiting nutrient. However, if the water is to be stored for a longer duration, it is good to have a phosphate content below 0.5 ppm. The water with phosphate content above 0.5 ppm can lead to eutrophication when stored for a longer duration.