

**NATIONAL INSTITUTE OF
TECHNOLOGY SRINAGAR**

Department of Civil Engineering

**Environmental Engineering Laboratory
Manual**

Vision of the Institute

To establish a unique identity of a pioneer technical Institute by developing a high-quality technical manpower and technological resources that aim at economic and social development of the nation as a whole and the region in particular keeping in view the global challenges.

Mission of the Institute

M1. To create a strong and transformative technical educational environment in which fresh ideas, moral principles, research and excellence nurture with international standards.

M2. To prepare technically educated and broadly talented engineers, future innovators and entrepreneurs, graduates with understanding of the needs and problems of the industry, the society, the state and the nation.

M3. To inculcate the highest degree of confidence, professionalism, academic excellence and engineering ethics in budding engineers.

VISION OF THE DEPARTMENT

To nurture Civil engineers with passion for professional excellence, ready to take global challenges and to serve the society with high human values.

MISSION STATEMENT OF THE DEPARTMENT

- (1) To provide facilities and infrastructure for academic excellence in the field of Civil engineering.
- (2) To inculcate in the student the passion for understanding professionalism, ethics, safety, sustainability and then actively contribute in the society.
- (3) To nurture creativity and encourage innovative solutions to real life challenging problems in Civil engineering students.
- (4) To prepare student for lifelong learning in global perspective.

PROGRAM EDUCATIONAL OBJECTIVES

PEO1: To prepare students to get employment, profession and/or to pursue post-graduation and research in Civil engineering discipline in particular and allied engineering fields in general.

PEO2: To prepare students to identify and analyse Civil engineering problems in an iterative approach that involves defining, quantifying, testing and review of the identified problem.

PEO3: To prepare students to plan, organize, schedule, execute and communicate effectively as an individual, a team member or a leader in multidisciplinary environment.

PEO4: To provide the students, an academic environment that makes them aware of excellence in field of Civil Engineering and enables them to understand significance of lifelong learning in global perspective.

EXPERIMENT--1

DETERMINATION OF pH OF A SAMPLE OF WATER

The term “pH” refers to the measurement of hydrogen ion activity in the solution. Since the direct measurement of the pH is very difficult, specific electrodes are needed for quick and accurate pH determination. pH is measured on a scale of 0 to 14, with lower values indicating high H⁺ (more acidic) and higher values indicating low H⁺ ion activity (less acidic). A pH of 7 is considered as neutral.

Every whole unit in pH represents a ten-fold increase in or decrease in hydrogen ion concentration. Most natural waters possess the pH values ranging from 5.0 to

8.5. Rain water have a pH value of 5.4 to 6.0 which then reacts with the soils and minerals causing the reduction in H⁺ ion concentration and thus the water may become alkaline with a pH of 8.0-8.5. More acid water (pH<5) and more alkaline (pH >9) and other immediate changes in the hydrogen ion concentration (pH) suggest that the quality of the water is adversely affected due to the introduction of some toxic contaminants in water bodies.

pH is measured using pH meter, which comprises a detecting unit consisting of a glass electrode, reference electrode, usually a calomel electrode connected by KCl Bridge to the pH sensitive glass electrode and an indicating unit which indicates the pH corresponding to the electromotive force is then detected. Before measurement, pH meter should be calibrated by using at least two buffers.

Precautions

1. The pH meter can be standardized by measuring the 7-pH buffer solution or any other solution of standard pH. Sometimes, the manufacturer of the pH meter may suggest other methods of standardizing, which too have to be followed.
2. The electrodes have to be inserted into the water so that it does not touch the bottom of the beaker. Bottom contact with damage may cause damage to the electrodes.
3. Any cause of slow response due to the polarization can be solved by washing the electrodes thoroughly.
4. Periodic check has to be conducted to check the electrodes

5. During the electrode storage, they have to be kept moist. And also follow the instructions of the manufacturer.

EQUIPMENT REQUIRED:-

1. pH meter
2. pH electrode filled with KCL solution
3. Buffer solutions of pH4 and pH 7
4. Clean beakers
5. Tissue papers
6. Distilled water
7. Thermometer

Procedure:-

- * Plug in the pH meter to power source and let it warm up for 5 to 10 minutes
- * Wash the glass electrode with distilled water and clean slowly with a soft tissue.
- * Note the temperature of water and set the same on the pH meter
- * Place the electrode in pH 7 buffer solution and set the value of 7 on the pH meter turning the Calibrate knob on the meter.
- * Take out the electrode, wash with DW and clean.
- * Dip the electrode in the pH 4 buffer solution. Adjust the value on the pH readout meter by the Slope switch . Repeat with pH 7 and pH4 buffers till a correct and stable reading is displaced.
- * While moving and cleaning the electrode, put the selector switch on standby mode.
Turn to pH mode for recording the pH.

- * Now place the electrode in the water sample whose pH is to be determined.
- * You can take a number of simultaneous readings for different samples until the power is on



pH Meter

EXPERIMENT NO-2

DETERMINATION OF TURBIDITY IN A WATER SAMPLE

AIM

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

5.2 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.

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.

Equipments 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.

Experimental Steps

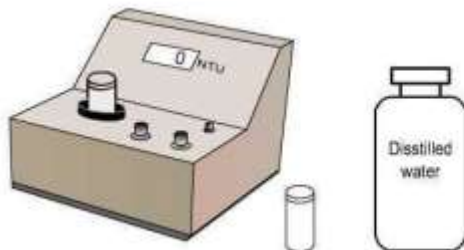
Switch on the Turbidity meter
(Atleast 30 min before the test)



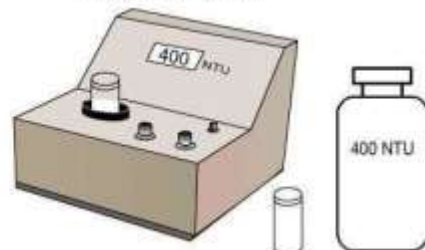
Prepare 400 NTU
solution



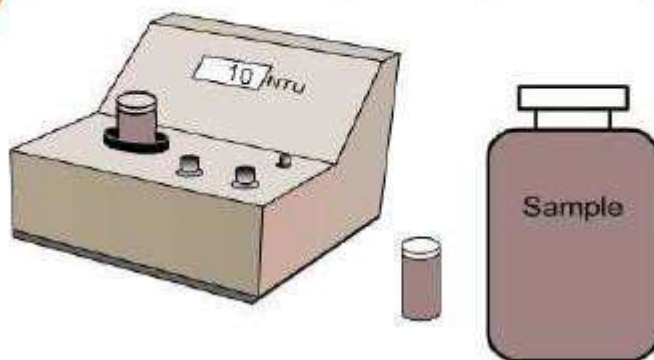
Calibrate the turbidity meter
to 0 NTU using distilled water
and by adjusting the
calibration knob



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



Read the Turbidity meter
by inserting the sample



NOTE:- IN STEP 3, USE SET ZERO KNOB TO ADJUST THE READING TO ZERO WITH DISTILLED WATER

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

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.

NOTE:- FOR MAKING SOLUTIONS OF LOWER TURBIDITY VALUES, USE THE NORMALITY EQUATION, $N_1V_1 = N_2V_2$ USING DISTILLED WATER FOR DILUTION PURPOSES

Example: - $N_1 = 4000$, N_2 required = 200 NTU for experiment, $V_1 =$ say 10ml, then $V_2 = \frac{N_1V_1}{N_2} = \frac{(4000 \times 10)}{200} = 400$ ml. Hence to make a solution of 200NTU, you need to dilute 10 ml of 4000 NTU to 400 ml.

EXPERIMENT NO-3

DETERMINATION OF TOTAL, SUSPENDED, AND DISSOLVED SOLIDS

AIM

The aim of the experiments is determination of total, suspended and dissolved solids in water.

APPARATUS REQUIRED

1. Balance 2. Beaker 3. Measuring Cylinder 4. Filter paper/ or Gooch Crucible 5. Funnel 6. Dropper

PROCEDURE:

(a) Measurement of Total Solids (TS)

(1) Take a clear dry glass beaker of 150 ML capacity (which was kept at 103°C in an oven for 1 hour) and put appropriate identification mark on it. Weight the beaker and note the weight.

(2) Pour 100ml. of the thoroughly mixed sample, measured by the measuring cylinder, in the beaker.

(3) Place the beaker in an oven maintained at 103°C for 24hours. After 24 hours, when whole of the water has evaporated, cool the beaker and weight. Find out the weight of solids in the beaker by subtracting the weight of the clean beaker determined in step (1)

(4) Calculate total solids (TS) as follows:

Total Solids in water= Difference of weight of the beakers / Volume of sample
X1000

(b) Measurement of Total Dissolved Solids (TDS)

(1) Same as above (step 1 of total solids).

(2) Take a 100 ml. of sample and filter it through a double layered filter paper or a Gooch Crucible and collect the filtrate in a beaker.

(3) Then repeat the same procedure as in steps (3) and (4) of the total solids determination and determine the dissolved solids contents as follows:

CALCULATION:

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

Also total solid (TS)= Suspended Solids + Total dissolved Solids (TDS)

EXPERIMENT-4

TO DETERMINE THE ALKALINITY IN A SAMPLE OF WATER

Alkalinity is a measure of the acid-neutralizing capacity of water and is usually determined by titration against sulfuric acid to the endpoint of the acid–base reaction.

Alkalinity has two types :- **Phenolphthalein alkalinity** denoted by (**P**) and the Total Alkalinity denoted by (**T**)

Phenolphthalein alkalinity measures the hydroxides and half the carbonates at a pH 8.3. **Total alkalinity** measures all carbonate, bicarbonate, and hydroxide **alkalinity** at a pH 4.5 (approximately).

DETERMINATION OF ALKALINITY

APPARATUS REQUIRED:

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

CHEMICALS REQUIRED

1. Standard 0.02N sulphuric acid
2. Phenolphthalein indicator
3. Methyl orange indicator

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

PROCEDURE:

1. Measure 100 ml of your sample into a 250 ML flask which has been thoroughly cleaned.
2. Add 03 drops of phenolphthalein indicator. If the color of the solution turns pink, titrate your sample with 0.02 N H₂SO₄ until color changes from pink to clear. Record the volume of acid used for the titration. Record the ml of N/50 sulphuric acid used as (P)
3. To the same sample, add 03 drops of methyl orange and continue to titrate with N/50 sulphuric acid until the first pink color appears. Note the volume of acid used as (T)

The various relations between (P) and (T) are given below:-

(a) $P=0$, alkalinity = $T \times 10$ mg/l (Bicarbonate Alkalinity)

(b) $P=T$, alkalinity= $T \times 10 \text{ mg/l}$ (Hydroxide Alkalinity)

(c) $P= 1/2 T$, Alkalinity = $T \times 10 \text{ mg/l}$ (carbonate alkalinity)

(d) $P < 1/2 T$, Alkalinity =($T-2P$) $\times 10 \text{ mg/l}$ (bi carbonate alkalinity)

(e) $P > 1/2 T$, Alkalinity = ($2P-T$) $\times 10 \text{ mg/l}$ (Hydroxide alkalinity)

TABLE FOR RECORDING THE OBSERVATIONS

S.No	Initial Reading (ml)	Final Reading (ml)	Vol of N/50 acid used	P(ml)	T (ml)	Alkalinity (mg/l)

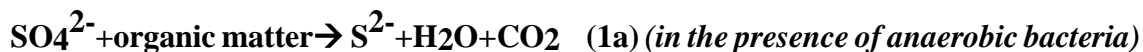
Note: - Take three readings for a sample

Experiment 5a: Sulfate Ions

Objective: To determine sulfate ion concentration in a water sample using method: 4500-SO₄²⁻ E. Turbidimetric Method)

Background:

Sulfate are found in appreciable quantity in all natural waters, particularly high in arid and semi arid regions where natural waters in general have high salt content. Sulfate salts are mostly soluble in water and impart hardness. Water with high concentrations has a bitter test. Sulfate may cause intestinal disorders. These ions can produce hydrogen sulfides as per following equation (1):



The sulfate data is used in determining applicability of different water types for their public and industrial applications. It indirectly indicates extent of problems that can arise due to reduction of sulfates to hydrogen sulfides. In addition, sulfate content of organic matter fed to anaerobic digester is important information as it gives idea of generation of hydrogen sulfides, which needs to be removed.

Procedure:

The turbidimetric method depends on the fact that barium sulfate formed following barium chloride addition to a sample (Equation 2) tends to precipitate in a colloidal form and this tendency is enhanced in the presence of an acidic buffer (consists of magnesium chloride, potassium nitrate, sodium acetate, and acetic acid). These precipitates need to be separated through filtration (using a filter) before sample is analyzed for sulfate concentration. This is a very rapid method and can be used for samples with sulfate concentration greater than 10 mg/L (samples can be diluted and then it can be analyzed).



Reagents:

1. Buffer Solution A: Dissolve 30 g magnesium chloride ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$), 5 g sodium acetate ($\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$), 1.0 g potassium nitrate (KNO_3), and 20 mL acetic acid (CH_3COOH ; 99%) in 500 mL distilled water and make up to 1000 mL.
2. ~~Buffer Solution B (required when the sample $\text{SO}_4^{2-} < 10$ mg/L):~~ Dissolve 30 g magnesium chloride, 5 g sodium acetate, 0.111 g sodium sulfate, and 20 mL acetic acid (99%) in 500 mL distilled water and make up to 1000 mL.
3. Dry Barium Chloride (BaCl_2) crystals
4. Standard Sulfate Solution: Dissolve 0.1479 g of anhydrous sodium sulfate in distilled water to make the volume 1 L. This solution contains 100 mg sulfate/L (i.e., 1 mL = 100 μg SO_4)

5. Prepare standards of various strengths (preferably from 0.0 to 40.0mg/L at the intervals of 5 mg/L by diluting this stock solution). Above 40 mg/L accuracy decreases and BaSO₄ suspensions lose stability.

Apparatus: Whatman No. 1 filter paper; Spectrophotometer; Magnetic stirrer

Steps:

1. Filter the sample through filter paper (Whatman No. 1) and take 50 mL of filtrate in an Erlenmeyer flask.
2. Add 20 mL buffer solution and mix in stirring apparatus. While stirring, add 0.15 g of barium chloride to the sample and stir the sample with the help of magnetic stirrer for about an hour.
3. Measure the absorbance against a distilled water blank (**DO NOT ADD BARIUM CHLORIDE TO IT.**) at 420 nm using spectrophotometer. Absorbance for the blank sample is taken to correct for sample color and turbidity.

<i>Sample Name</i>	<i>Turbidity (NTU)</i>	<i>Sample Name</i>	<i>Turbidity (NTU)</i>
Distilled water blank		Standard 1 (5ppm)	30
Sample 1		10ppm	60
Sample 2		15	89
		20	101
		25	129
		30	157
		35	191
		40	205

4. Process the standard solution of different strengths in similar way and record the absorbance for each solution. Plot a standard sulfate calibration curve on a graph paper from these absorbance values putting strengths (mg/L) on X-axis and absorbance @ 420 nm on Y-axis. Fit a best-fit linear model to the data. Express equation as:

$$\text{Absorbance value} = A + B \times \text{Sulfate concentration (in mg/L)} \quad (3)$$

5. Using the standard sulfate calibration curve (a linear-model; Equation 3), find out sulfate concentration in the given unknown sample in mg/L.

Sulfate concentration (mg SO₄²⁻/L) = (1000×mg SO₄²⁻)/(mL sample) (4)

Experiment 5b

Determination of chloride concentration in a sample of water

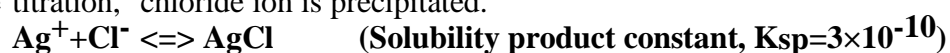
Background:

Chloride in the form of chloride (Cl^-) ion is one of the major inorganic anions in water and wastewater. The chloride concentration is higher in wastewater than in raw water because sodium chloride is a common article of diet and passes unchanged through the digestive system (Average estimate of excretion: 6 g of chlorides/person/day; additional chloride burden due to human consumption on wastewater: 15 mg/L). Along the sea coast chloride may be present in high concentration because of leakage of salt water into the sewage system. It also may be increased by industrial process. In potable water, the salty taste produced by chloride concentration is variable and depends on the chemical composition of water. Some waters containing 250 mg/L Cl^- may have a detectable salty taste if sodium cation is present. On the other hand, the typical salty taste may be absent in waters containing as much as 1000 mg/L when the predominant cations are calcium and magnesium. In addition, a high chloride contents may harm metallic pipes and structures as well as growing plants.

The measured chloride ions can be used to know salinity of different water sources. For brackish water (or sea water or industrial brine solution), it is an important parameter and indicates the extent of desalting of apparatus required. It also interferes with COD determination and thus it requires a correction to be made on the basis of amount present or else a complexing agent, such as HgSO_4 can be added. Further, chloride ions are used as tracer ions in column studies to model fate of different contaminants in soil and liquid media.

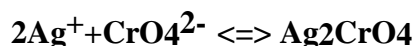
Lab Procedure

The Mohr Method uses silver nitrate for titration (normality: 0.0141) This corresponds to **1 mL of 0.0141 equals to 1 mg chloride in solution**. The silver nitrate solution is standardized against standard chloride solution, prepared from sodium chloride (NaCl). During the titration, chloride ion is precipitated.



The indicator (potassium chromate) is added to visualize the endpoint, demonstrating presence of excess silver ions. In the presence of excess silver ions, solubility product of silver chromate

exceeded and it forms a reddish-brown precipitate. This stage is taken as evidence that all chloride ions have been consumed and only excess silver ions have reacted with chromate ions:



The silver chromate produced is responsible for imparting brick red color to the solution

Apparatus: Burette, conical flask, pipette, measuring cylinder

Reagents: Potassium chromate indicator solution, standard silver nitrate as a titration agent..

Steps:

1. Take 50 ml of sample in a conical flask.
2. Add 1.0 ml indicator solution, (Potassium chromate)
3. The initial color of the mixture is slight yellow
4. Titrate with standard silver nitrate solution to brick red end point and note down volume of titrant used.
5. Calculate chloride ion concentration using the equation :-:

Chloride Ion Concentration (mg/L) = (ML of AgNO₃ used - 0.2) X 500 / ML of sample used

Precautions:

1. A uniform sample size must be used, preferably 50 ML, so that ionic concentrations needed to indicate the end point will be constant.
2. The pH must be in the range of 7 to 8 as silver ions are precipitated as AgOH at high pH levels and the chromate ions are converted to Cr₂O₇²⁻ at low pH values.
3. A definite amount of indicator must be used to provide a certain concentration of chromate ions, otherwise silver chromate may form too soon or not soon enough.
4. Caution should be made to notice indicator color change as it can vary person-to-person. The titrant should be added drop wise and the sample shaken constantly.
5. Take at least three readings for each sample.

Answer the questions also (for Submission with Lab report):

1. Does the measured chloride ion concentration exceed the maximum concentration criteria? Compare your values with IS:- 10050 to determine whether the determined chloride content is within permissible limits.
2. What is the permissible value of Chlorides for drinking water ?
3. What does excess of Chloride content in drinking water indicate ?

EXPERIMENT NO:-6
DETERMINATION OF ACIDITY

APPARATUS REQUIRED:

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

REAGENTS REQUIRED:

1. Sodium hydroxide titrant (0.02 N); 2. Phenolphthalein Indicator; 3
.Methyl Orange Indicator

PROCEDURE:

1. Take 50 ml sample in a conical flask and add 2-3 drops of methyl orange indicator solution.
2. Fill the burette with 0.02 N NaOH solution and titrate till the colour of solution just changes to faint orange colour, indicating the end point. Record the volume of titrant consumed as V1 in ml.
3. Calculate the methyl orange acidity using Equation: **Methyl orange acidity (or Mineral Acidity) = (V1×1000) / (Sample volume)**

When the 0.02 N NaOH solution, used in titration is not standardized, mineral acidity is calculated using following Equation:-

Methyl orange acidity= (V1×N×50×1000) / (Sample vol.) Where N is
the normality of NaOH

For phenolphthalein acidity test.

Add 2-3 drops of phenolphthalein indicator solution to water sample and continue the titration till the faint pink colour develops in the solution (i.e., the end point of titration). Record the volume of titration consumed as V2 (mL) and calculate total acidity or phenolphthalein acidity using equation:-

Total acidity (or Phenolphthalein Acidity)= (V2×N×50×1000) / (Sample vol.)

Record your observations in a tabular form as under:-

Sample No.	Initial Reading-ml	Final Reading-ml	Volume of 0.02N NaOH used (ml)	Indicator Used	Acidity(mg/l) (Mineral)
				Methyl Orange	
				Methyl Orange	
				Methyl Orange	

Sample No.	Initial Reading-ml	Final Reading-ml	Volume of 0.02N NaOH used (ml)	Indicator Used	Acidity(mg/l) (Total)
				phenolphthalein	
				phenolphthalein	
				phenolphthalein	

NOTE:- To be adopted for all titrations

1. Add all titrant very slowly, drop by drop
2. Shake the flask containing the sample thoroughly
3. Note all readings accurately on the burette.
4. Titrate against a white background to note the colour change.

EXPERIMENT NO-7

Determination of Hardness in a sample of water:-

Background: Hard Water: Hard waters are generally considered to be those waters that require considerable amounts of soap to produce foam and that also produce scale in water pipes, heaters, boilers and other units in which the temperature of water is increased. Hard water are appropriate for human consumption similar to that as soft waters, however it produces adverse actions with soap and thus their use for cleaning purposes is unsatisfactory and thus their removal from water is required. Hardness of waters varies from place to place. In general, surface waters are softer than ground waters. Waters are commonly classified based on degree of hardness

Classification of hardness types

Hardness (mg/L) Degree of hardness

0-75	Soft
75-100	Moderately hard
150-300	Hard
>300	Very hard

Hardness is most commonly measured by titration with an EDTA solution. A titration involves adding small amounts of a solution to a **water** sample until the sample changes color. You can titrate a sample for total **hardness** using a burette .

Total hardness is defined as the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate in mg/L. When hardness (numerically) is greater than the sum of carbonate and bicarbonate alkalinity, amount of hardness equivalent to the total alkalinity is called “Carbonate hardness”. . When the hardness is numerically equal to or less than the sum of carbonate and bicarbonate alkalinity all of the hardness is carbonate hardness and there is no non carbonate hardness. The hardness may range from zero to hundreds of milligrams per litre in terms of calcium carbonate, depending on the source and treatment to which the water has been subjected.

Ethylenediamine tetra-acetic acid and its sodium salts (EDTA) form a chelated soluble complex when added to a solution of certain metal cations. If a small amount of a dye such as Eriochrome black T is added to an aqueous solution containing calcium and magnesium ions at a pH of 10 ± 0.1 , the solution will become wine red. If EDTA is then added as a titrant, the calcium and magnesium will be complexed. After sufficient EDTA has been added to complex all the

magnesium and calcium, the solution will turn from wine red to blue. This is the end point of the titration.

Apparatus

1. Burette
2. Pipette
3. Erlenmeyer flask
4. Bottle etc.

Reagents

1. Standard EDTA titrant (0.01 M)
2. Eriochrome black T indicator
3. Ammonia buffer solution

Procedure

1. Dilute 25 mL of sample (V) to about 50 mL with distilled water in an Erlenmeyer flask.
2. Add 1 mL of buffer solution.
3. Add two drops of indicator solution. The solution turns wine red in colour.
4. Add the standard EDTA titrant slowly with continuous stirring until the last reddish tinge disappears from the solution. The colour of the solution at the end point is blue under normal conditions.
5. Note down the volume of EDTA added (V1).

$$\text{Hardness as CaCO}_3 = \frac{1000 V_1}{V} = \dots\dots\dots \text{mg / L}$$

EXPERIMENT-8

TO DETERMINE ODOUR AND TASTE IN A WATER SAMPLE

CAUSES AND SIGNIFICANCE

Taste and odour can originate from natural inorganic and organic chemical contaminants and biological sources or processes (e.g. aquatic microorganisms), from contamination by synthetic chemicals, from corrosion or as a result of problems with water treatment (e.g. chlorination). Taste and odour may also develop during storage and distribution as a result of microbial activity.

Taste and odour in drinking-water may be indicative of some form of pollution or of a malfunction during water treatment or distribution. It may therefore be an indication of the presence of potentially harmful substances. The cause should be investigated and the appropriate health authorities should be consulted, particularly if there is a sudden or substantial change.

There are a number of diverse organisms that often have no public health significance but which are undesirable because they produce taste and odour. As well as affecting the acceptability of the water, they indicate that water treatment and/or the state of maintenance and repair of the distribution system are insufficient.

Actinomycetes and fungi can be abundant in surface water sources, including reservoirs, and they can also grow on unsuitable materials in the water supply distribution systems, such as rubber. They can produce geosmin, 2-methyl isoborneol and other substances, resulting in objectionable tastes and odours in the drinking-water.

Blooms of cyano bacteria and other algae in reservoirs and in river waters may cause odour and taste.

High concentrations of chloride give a salty taste to water. Concentrations in excess of 250 mg/l are increasingly likely to be detected by taste, but some consumers may become accustomed to low levels of chloride-induced taste.

Most individuals are able to taste or smell chlorine in drinking-water at concentrations well below **5 mg/l**, and some at levels as low as **0.3 mg/l**. Hence in the chlorination process, the residual chlorine at the consumer end is not to exceed **0.2 mg/l**

CLASSIFICATION OF ODOURS

(a) No odour (b) Ammoniacal bad eggs (sulphide) (c) chlorine (bleach)
(d) earthy (e) fruity (f) fuel
(g) milky (h) oily (i) soapy (j) Fishy and some other (this should be specified)

TASTE

(a) No Taste (b) Sweet (c) Salty (d) Sour (e) Bitter

Apparatus Required:-

1. 500 ML clear flasks
2. Watch glasses or stopper
3. Heating device with temperature readout

Procedure :- For Cold Odour:-

Fill the 500 ML flask with around 200 ML of sample. Close the stopper. Shake the flask well at room temperature. Open the flask and smell at the mouth of the flask. Report the odour in terms of the classifications mentioned above

Procedure for Hot Odour:-

The intensity of the cold odour may be very low and cannot be detected. For this purpose, heat the flask to around 58-60 degrees C. Remove the stopper and smell at the mouth of the flask.

The odours can be detected by a group of observers as single observation may not be correct. See how many agree to a common odour.

Odour Intensity:-

The intensity of the odour can be determined on its strength. Numbers are assigned as follows:-

0--No odour

1--Very

Faint 2--

Faint

3--

Distinct

4--

Decided

5--Very Strong

Add your results in a tabular form as indicated below.

Sample Number	Date	Source Name	Odour Classification	Cold Odour intensity	Hot Odour intensity	Remarks

As far as taste is concerned, it should be noted only when the full potability of water is ascertained, i.e. it should be free from all impurities (Physical, Chemical and Biological)

Sample Number	Date	Source Name	Classification of Taste	Remarks

NOTE:- Potable water should be colorless, tasteless and odourless. Refer :- IS:- 10050:- Specifications for Drinking Water

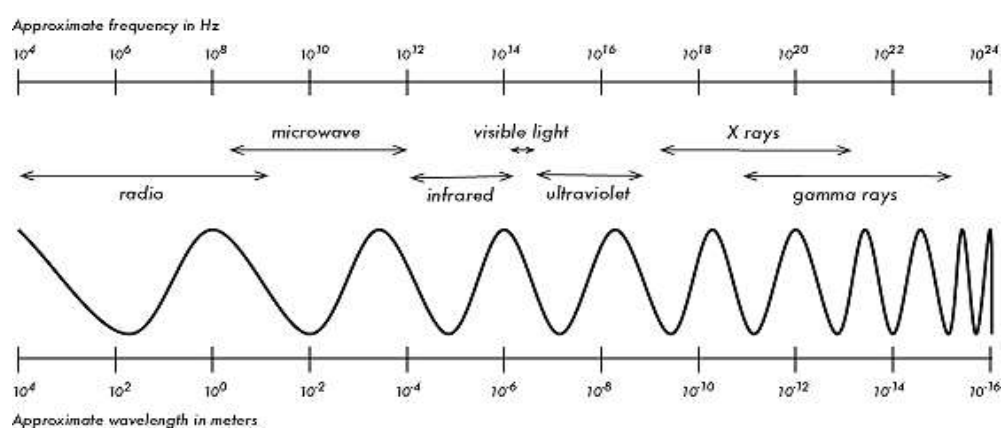
Experiment 10

AIM To determine the Na, Ca, K in a sample of water

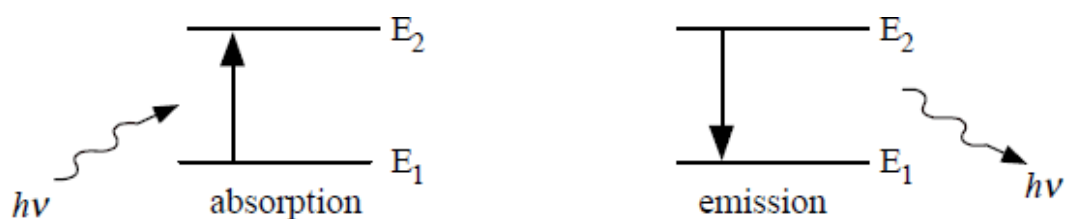
INTRODUCTION

Atomic spectroscopy is based on the absorption, emission or fluorescence process of light by atoms or elementary ions. Information for atomic scale is obtained in two regions of the electromagnetic radiation (EMR) spectrum. These regions are UV/VIS and the X-ray.

Electromagnetic Spectrum:



When sample interacts with light, absorption process occurs. Ground state electrons of the sample atom tend to move to the excited states with the energy of absorbed light. This process can also be called excitation. Apart from light, heat can cause excitation. Since excited state is unstable, electrons want to return back to the ground state. When an excited electron turns back to its ground state a radiation is emitted that is equal to the energy difference between excited and ground states. The emitted light is monochromatic and it has the same wavelength as the light absorbed in the excitation process. Depending on the excitation technique, absorbed or emitted light is measured. If excitation source is flame, emitted radiation is measured. On the other hand, absorption is measured when lamp is used for excitation. Both are directly proportional with the number of atoms in the sample.



Flame photometry or flame emission spectroscopy is an atomic emission technique. There is no need for light source. Flame serves both as an atomizer and excitation source. It is suitable for qualitative and quantitative determination of several cations, especially for metals that are easily excited to higher energy levels at flame temperature. These metals are Na, K, Ca,

In this technique, first aerosols are formed from sample solution by a jet of compressed gas. This process is called nebulization. The design of the nebulizer is shown in Figure 1. Then the flow of the gas carries the aerosols into a flame where atomization takes place. Atomization is the conversion of sample aerosols into an atomic vapor by flame. When a sample is atomized, a substantial fraction of the metallic constituents are reduced to gaseous atoms and also depending on the temperature of the flame a certain fraction of these atoms are ionized. Then, electrons of the formed atoms are excited to upper state. Light is emitted at characteristic wavelengths for each metal as the electron returns to the ground state.

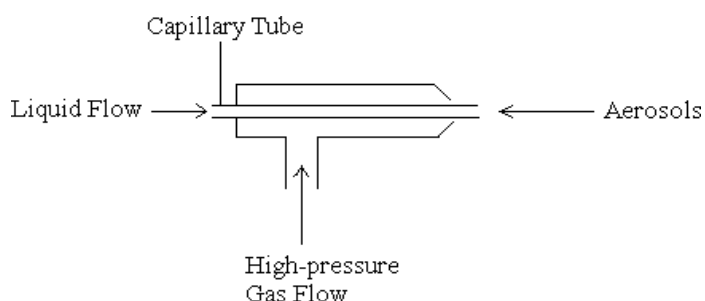


Figure 1. Design of a nebulizer

Flame is formed by two components: fuel and oxidant. Temperature of the flame changes depending on the fuel and oxidant types and their proportions. In flame photometer generally natural gas is used as a fuel and air is the oxidant. Table 1 lists the different types of fuel, oxidant and the temperature of the flame.

Table 1. Flame components and temperatures

FUEL	OXIDANT	TEMPERATURE, °C
Natural Gas	Air	1700-1900
Natural Gas	Oxygen	2700-2800
Hydrogen	Air	2000-2100
Hydrogen	Oxygen	2550-2700
Acetylene	Air	2100-2400
Acetylene	Oxygen	3050-3150
Acetylene	Nitrous Oxide	2600-2800

Flame consists of three important regions. These are the primary combustion zone, interconal region and outer cone (secondary combustion zone). The appearance and the relative sizes of these regions can be changed with the fuel-oxidant ratio. Regions of the flame are shown in Figure 2.

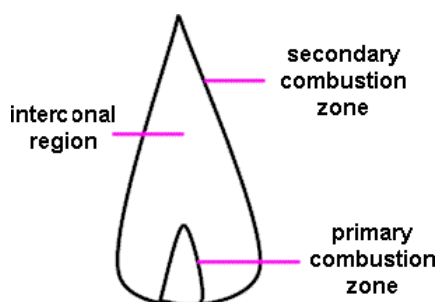


Figure 2. Schematic appearance of flame

The primary combustion zone of the flame is blue in color. In this region, there is no thermal equilibrium. Therefore, it is not used in flame spectroscopy. The interconal region is rich in free atoms and is the most widely used region for the spectroscopy. In The outer cone the products of the inner core are converted to stable molecular oxides.

In flame photometer there are three fundamental systems which are emission, λ -selection and recording. The general flow diagram is shown in Figure 3.

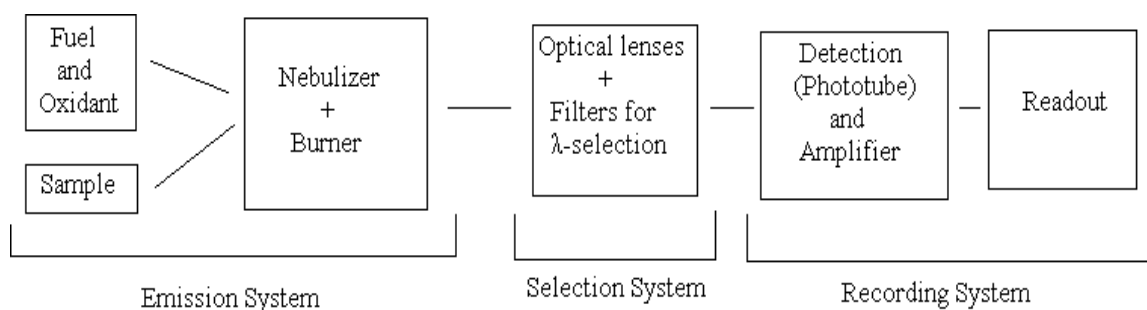


Figure 3. A general scheme of a Flame Photometer

Emission System: This consists of the flame, which is the source of emission.

λ -Selection System: This includes the whole optical system of wavelength selection. In flame photometer the wavelength selector is filter. The radiation emitted by the excited atoms is selected by using a filter which transmits an emission line of one of the elements while absorbing the others. There are two types of filters. These are absorption and interference filters. Absorption filters are restricted to visible region of the spectrum but interference filters are used in UV, VIS and IR regions of the spectrum.

Absorption filters are less expensive than the interference filters and they have been widely used for band selection in the visible region. These filters function by absorbing certain portions of the

spectrum and transmitting the band of wavelengths belonging to the analyte element. The most common type consists of colored glasses.

Interference filters rely on optical interference to provide relatively narrow bands of radiation. They consist of a transparent dielectric layer (CaF₂ or MgF₂) that occupies the space between two semi-transparent metallic films. This array is sandwiched between two plates of glass.

Recording System: This part consists of all the means of detection (phototubes or photomultiplier tubes), the electronic devices of amplifying and electrical apparatus for measuring and direct recording.

The instrument that is used in this experiment is a JENWAY PFP7 model which is a low temperature, single channel emission Flame Photometer designed for the routine determinations of Na, K, Ca, . It is a direct reading digital instrument designed for use in clinical, industrial and educational applications.

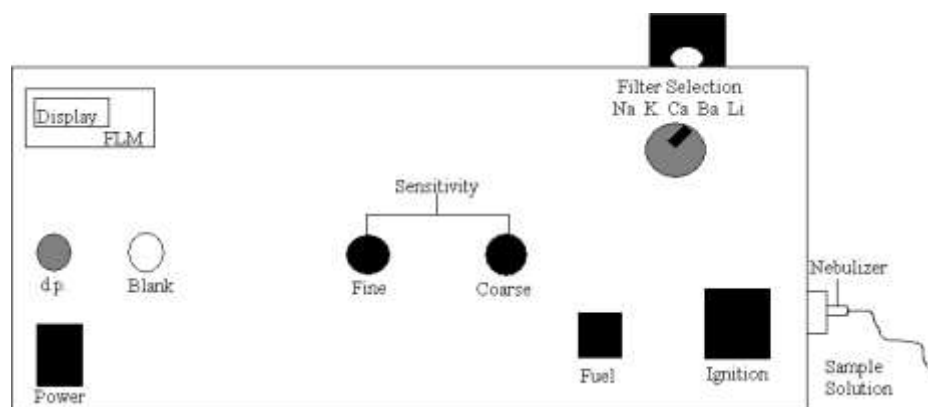


Figure 4. Front Panel Controls for the instrument

Calibration Curve:

In flame photometry emitted light intensity from the flame is directly proportional to the concentration of the species being aspirated. The graph below shows that the direct relationship between the emission and concentration is true only at relatively low concentrations of mg/L level (up to 50 mg/L).

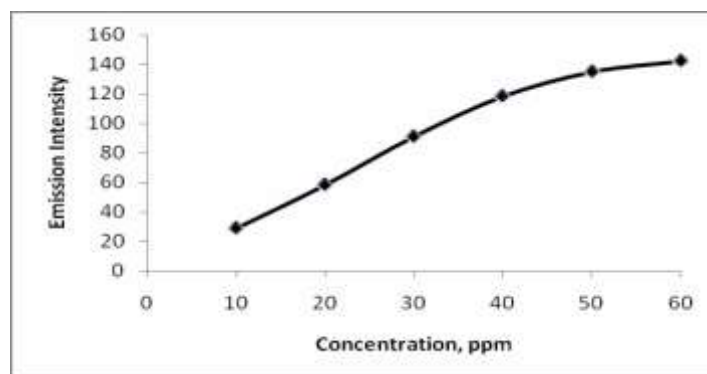


Figure 5. Calibration curve

If the samples being analyzed lie on the linear part of the curve then user can take direct concentration readings from the digital display. However, if the concentration of sample is above the linear part, then user has to dilute sample so that it lies on the linear part of the curve.

A calibration curve is obtained by using standard solutions containing known concentrations of the elements to be determined. The concentration range covered by the calibration curve depends on the expected concentration so that the sample readings fall somewhere inside the calibration curve. Once the calibration curve has been plotted, the scale reading for the sample solution is compared with the curve to find the concentration.

It is important to emphasize that each element has its own characteristic curve and separate curves must be constructed.

REAGENTS AND APPARATUS

- Calcium Carbonate, $\text{Ca}(\text{NO}_3)_2$
- Sodium Chloride, NaCl (in the balance room)
- 100 mL volumetric flask
- 5 mL pipette

PROCEDURE

A. Flame Photometric Determination of Calcium in tap water

- 1) Prepare 100.0 mL of 1000 mg/L Ca stock solution from $\text{Ca}(\text{NO}_3)_2$.
- 2) Prepare 100.0 mL of 10.0, 20.0, 30.0, 40.0 and 50.0 mg/L standard Ca solutions from 1000 mg/L Ca stock solution.
- 3) Select Ca as analyte from the instrument.
- 4) Adjust the fuel/oxidant ratio and burner position by aspirating 30.0 mg/L standard Ca solution.
- 5) Measure the emission intensities of all standard Ca solutions (including the blank solution) and sample solution after necessary dilutions.
- 6) Make 10 replicate measurements of 10.0 mg/L Ca standard solution to calculate Limit of detection (LOD).
- 7) Draw the calibration plot and find the concentration (in mg/L) of Ca in beverages and tap water.
 - *After finishing measurements, draw calibration curve and discuss the applicability of it to the unknown samples by looking at correlation coefficient and dynamic range and if necessary, prepare more solutions.*

B. Flame Photometric Determination of Sodium in tap water

- 1) Prepare 100.0 mL of 1000 mg/L Na stock solution from NaCl .

- 2) Prepare 100.0 mL of 10.0, 20.0, 30.0, 40.0 and 50.0 mg/L standard Na solutions from 1000 mg/L Na stock solution.
- 3) Select Na as analyte from the instrument.
- 4) Adjust the fuel/oxidant ratio and burner position by aspirating 30.0 mg/L standard Na solution.
- 5) Measure the emission intensities of all standard Na solutions (including the blank solution) and sample solution after necessary dilutions.
- 6) Make 10 replicate measurements of 10.0 mg/L Na standard solution to calculate Limit of detection (LOD).
- 7) Draw the calibration plot and find the concentration (in mg/L) of Na in beverages and tap water.
 - *After finishing measurements, draw calibration curve and discuss the applicability of it to the unknown samples by looking at correlation coefficient and dynamic range and if necessary, prepare more solutions.*

Enroll No:-

Section:

Date:

REPORT SHEET FLAME PHOTOMETRY

Part A: Determination of Calcium		Sodium	
Sample	Emission	Sample	Emission
blank		blank	
10 ppm		10 ppm	
20 ppm		20 ppm	
30 ppm		30 ppm	
40 ppm		40 ppm	
50 ppm		50 ppm	
Tap water		Tap water	
Sour cherry juice		Sour cherry juice	

Part A: Determination of Calcium	
Line Equation ($y=mx+n$)	
R ²	
Limit of Detection	
CCa in tap water (ppm)	
CCa in sour cherry juice	

Part B: Determination of Sodium	
Line Equation ($y=mx+n$)	
R ²	
Limit of Detection	
CNa in tap water (ppm)	
CNa in sour cherry juice	

Experiment 11

Determination of percentage of ammonia nitrogen present in a given sample of water.

SCOPE AND APPLICATION

1. This method covers the determination of ammonia in drinking, ground, surface, and saline waters, domestic and industrial wastes.
2. The applicable range is 0.01-2.0 mg/L NH₃ as N.
3. Higher concentrations can be determined by sample dilution.

PROCEDURE

1. Take 5 ml filtered sample and make up to 50 ml in nessler's tube.
2. Add 1 drop of EDTA.
3. Add 2 ml of Nessler's reagent.
4. Take reading in Spectrophotometer.
5. Multiply by 10 to get actual concentration in mg/L.

DATA ANALYSIS AND CALCULATIONS

1. Prepare a calibration curve by plotting instrument response against standard concentration. Compute sample concentration by comparing sample response with the standard curve.
2. Report results in mg NH₃ -N/L.