CANARA ENGINEERING COLLEGE

(Affiliated to Visvesvaraya Technological University, Belgaum)
SPONSERED BY CANARA HIGH SCHOOL ASSOCIATION
BENJANAPADAVU – 574219
BANTWAL TALUK, D.K. DISTRICT, KARNATAKA

DEPARTMENT OF CHEMISTRY

LABORATORY MANUAL IN ENGINEERING CHEMISTRY

I / II SEMESTER – B.E

Compiled By

THE HOD & FACULTY

Dept. of Chemistry

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CONTENTS

LABORATORY DISCIPLINE

Laboratory work requires great care, keen observation, intelligence and systematic working. Moreover, as student may have to work in groups, a code of conduct is also very essential for safe working in the laboratory. While working in chemical laboratory, the students must follow all the rules laid down by the teacher along with the followings.

1.   While coming to the laboratory for practical, students must have a practical record book, lab manual book, a hand kerchief and other equipments as directed by the teacher.

2.   Always come prepared for the experiment.

3.   Listen to the teachers instructions carefully.

4.   Plan the experiment work so that it should finish within stipulated time.

5.   Keep your work table clean and tide.

6.   Set the apparatus systematically.

7.   Be economical in the use of chemicals, gas and water. Use little reagent as it is convenient to perform the experiment. Turn off the water tap and gas tap immediately after use.

8.   Handle the glass apparatus very carefully. In case of breakage, report it to the lab instructor.

9.   Dismantle the experiment set up carefully, clean the apparatus and keep neatly.

10. Do your experiments honestly with caring for the final result. Record the observations in an observation note book.

11. Dispose off all the waste materials in their proper place.

GENERAL INSTRUCTIONS

1. Keep your working table clean and tidy.

2. Keep your apparatus scrupulously clean.

3. Keep the reagents in their proper place after use. Never return any used / unused material to the reagent bottle.

4. Keep the gas taps and water taps closed when not in use.

5. Do not walk barefooted in the laboratory.

6. Do turn out to be a better analyst, understand the theory behind the reactions you carry out.

7. Use the specified quantities of reagents.

8. Do not use wet hand during weighing.

9. Read the label of the bottle carefully before using the chemicals from it. Otherwise serious accidents may occur in a few instances.

10. Before leaving the lab, clean the apparatus, replace them in the proper place.

ENGINEERING CHEMISTRY LABORATORY

(As per choice based Credit System (CBCS) scheme)

(Effective from the academic year 2018-2019)

SEMESTER –I/II

Laboratory Code	18CHEL16/18CHEL26	IA Marks	40
Number of Lecture Hours/week	3(1 hr tutorial +2 hrs lab)	Exam marks	60
Total Number of Lecture hours	42	Exam Hours	03

Course objectives:

- To provide students with practical knowledge of quantitative analysis of materials by classical and instrumental methods for developing experimental skills in building technical competence.

Course outcomes:

On completion of this course, students will have the knowledge in,

- Handling different types of instruments for analysis of materials using small quantities of materials involved for quick and accurate results, and

- Carrying out different types of titrations for estimation of concerned in materials using comparatively more quantities of materials involved for good results.
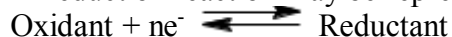
EXPERIMENT NO. 1

ESTIMATION OF FAS BY POTENTIOMETRIC METHOD

AIM:
Estimation of FAS using standard potassium dichromate solution by Potentiometric titration

PRINCIPLE:
A reversible oxidation – reduction reaction may be represented as follows.

$$\text{Oxidant} + n\text{e}^- \rightleftharpoons \text{Reductant}$$

The potential developed when an inert electrode like platinum is immersed in a solution containing both oxidant and reductant given by the expression.

$$E = E_0 + \frac{0.0591}{n} \log \frac{[\text{Oxidised state}]}{[\text{Reduced state}]} , \text{at } 25^\circ C$$

If the concentration of the oxidant and reductant are equal, E becomes equal to E_0, which is known as standard potential. From the above expression, it is evident that a ten fold change in the ratio of the concentration of the oxidant to reductant will change the potential of the redox system by $0.0591/n$ volts.

During the oxidation of a reducing agent and the reduction of the oxidizing agent, the ratio of the oxidant to reductant changes and therefore the potential changes. The change in the potential is more rapid in the vicinity of the end point of a redox reaction. Thus a titration involving ferrous ion with potassium dichromate may be followed potentiometrically and it is found that there is a sudden change in the potential at the equivalence point.

The indicator electrode is usually a bright Platinum–wire or foil (Pt- electrode) and the reference electrode is saturated calomel electrode.

The reaction between the ferrous ammonium sulphate (FAS) and potassium dichromate in sulphuric acid medium is as follows.

$$6\ FeSO_4 + 7\ H_2SO_4 + K_2Cr_2O_7 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + 3Fe_2(SO_4)_3 + 7H_2O$$

A known volume of ferrous ammonium sulphate solution acidified with sulphuric acid is treated against a standard solution of potassium dichromate and redox potential of the system is measured with reference to calomel electrode at various stages of addition of potassium dichromate. Then a plot of $\Delta E / \Delta V$ verses potassium dichromate is used to find out equivalence point. Known volume of potassium dichromate corresponding to the equivalence point, it is possible to find out the concentration of ferrous ion and thereby the amount of ferrous ammonium sulphate present in the given solution.

PROCEDURE:
Make up the Mohr's salt (FAS) solution kept in a 250ml standard flask to the mark with distilled water and shake well. Pipette out 25ml of this solution into a 100ml beaker. Add 1 test tube of dil. H_2SO_4. Immerse calomel electrode and platinum electrodes into it and connect to a potentiometer. Measure the potential. Add $K_2Cr_2O_7$ from the burette in increments of 0.5ml and measure the potential after each addition.

Initially the emf increases slowly and at the equivalence point there will be a sudden jump in the emf. Record at least 4-6 readings after the equivalence point.

Plot a graph of $\Delta E/\Delta V$ against volume of $K_2Cr_2O_7$. The volume of $K_2Cr_2O_7$ corresponding to the peak of the curve indicates the volume for the oxidation of ferrous

ammonium sulphate. From this determine the normality of FAS and the amount of FAS in the given solution.

RESULT:

      Normality of FAS in the given solution = -------------------- N

      Amount of FAS per litre =--------------------- g.

OBSERVATIONS AND CALCULATIONS:

[Given standard flask number =   ]

Volume of FAS solution pipetted out V_{FAS}=-------------ml
Normality of $K_2Cr_2O_7$, $N_{K_2Cr_2O_7}$ = -------------------------------- N

Volume of $K_2Cr_2O_7$ (ml)	Emf, E (V)	ΔE ($E_2 - E_1$)	ΔV ($V_2 - V_1$)	$\Delta E / \Delta V$
0.0		--------	----------	-----------
0.5				
1.0				

Graph:

From the graph,

Volume of $K_2Cr_2O_7$ at equivalence point $V_{K_2Cr_2O_7}$ = ---------------- ml.

Normality of Mohr's salt solution = $\dfrac{N_{K_2Cr_2O_7} \times V_{K_2Cr_2O_7}}{V_{FAS}}$ N

=

= .... N

Weight of FAS in litre of the solution, = N_{FAS} X Eq. wt of FAS

= X 392

= ....g.

RESULT:

Normality of FAS in the given solution = -------------------- N

Amount of FAS per litre = --------------------- g.

EXPERIMENT NO. 2

CONDUCTOMETRIC ESTIMATION OF ACID MIXTURE

AIM:

To determine the concentration of a strong acid and a weak acid in a mixture by conductometric titration with a strong base.

PRINCIPLE:

In conductometric titrations, the conductance of the solution is measured during titration, before and after the equivalence point, and the variation of conductance of the solution during the titration is utilized to locate the end point.

In a mixture of strong acid and a weak acid, the strong acid undergoes complete dissociation as it is a strong electrolyte and the weak acid remains almost undissociated. When a mixture of strong acid and a weak acid is titrated against a strong base, the strong acid is consumed first by the base. After the neutralization of the strong acid is completed, then only the weak acid is neutralized. When the strong acid is neutralized by the base, H^+ ions of the acid are consumed and are replaced with metal ions in the solution. For example for HCl + NaOH,

$$H^+ + Cl^- + Na^+ + OH^- \rightarrow Na^+ + Cl^- + H_2O$$

Since highly conducting H^+ ions are replaced by weakly conducting Na^+ ions during the titration, the conductance of the solution sharply decreases. When the weak acid is neutralized, the poorly conducting acid is converted into a salt. The salt undergoes complete dissociation, and therefore, the conductance increases gradually. After the neutralization of the weak acid is also completed, further addition of NaOH increases the conductivity sharply in proportional with the amount of NaOH added, as more and more Na^+ and OH^- ions are added into the solution. Therefore if the conductance of the solution is plotted against volume of the base added we will get three straight lines. The points of intersection of the lines give the equivalence points corresponding to the strong acid and the total acid respectively, and the equivalence point for the weak acid can be deduced.

The solution taken in the burette should be at least ten times more concentrated than the analyte solution to avoid the effect of dilution on the variation of conductance.

PROCEDURE:

Pipette out 50ml of the given acid mixture solution into a 100ml beaker. Clean the conductance cell with distilled water, dry it with filter paper strips and dip in the acid mixture. Connect the conductivity cell to the conductometer and select a suitable range for conductance, so that, maximum number of digits is displayed on the display of the conductometer. Fill a burette with the given strong base, NaOH. Add 0.5ml of the base into the beaker at a time, stir well and note the conductance of the solution after each addition. The titration should be continued till the decreasing trend of conductance changes to a gradually increasing trend, and then to a relatively sharply increasing trend; and at least 5-6 readings should be taken after that. The points of intersection of the curves give the equivalent points, the first point of intersection corresponding to the strong acid and the second corresponding to the total acid in the solution, respectively.

RESULT:

Equivalence volume for the Strong acid, V_1 = --------------- ml.

Concentration of the strong acid in the given solution = --------------N

Equivalence volume for weak acid, V_3 = ------------------ ml.

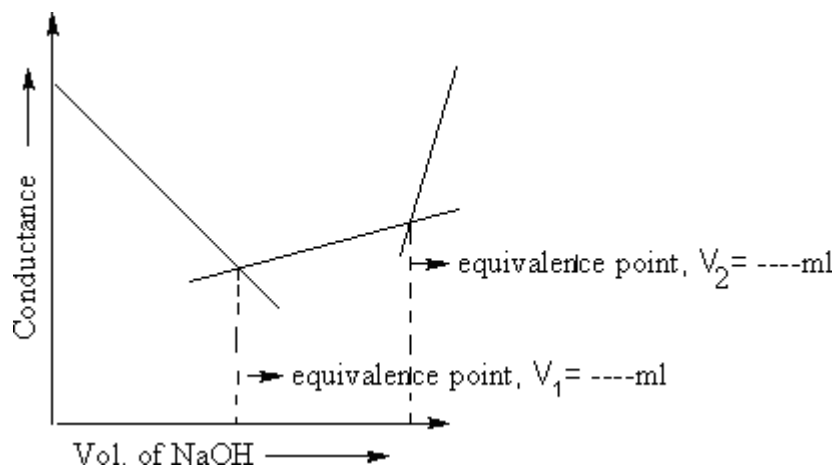Concentration of the weak acid in the given solution = ------------- N

OBSERVATIONS AND CALCULATIONS:

[Given standard flask number =   ]

Concentration of the given base, N_{NaOH}, N_1 = ------------------- N
Volume of the acid mixture pipetted out = 50 cm^3

Vol. of NaOH (cm^3)	Conductance	Vol. of NaOH (cm^3)	Conductance
0.0			
0.5			
1.0			

GRAPH:

Equivalence volume for the strong acid from the graph, $V_1 =$ .... cm^3

$\therefore$ Concentration of the strong acid $= \dfrac{V_1 \times N_1}{50}$

$=$

$=$...................N.

Equivalence volume for the total acid from the graph, $V_2 =$ .... cm^3

$\therefore$ Equivalence volume for the weak acid, $V_3 = (V_2 - V_1)$

$=$

$=$ .... cm^3

$\therefore$ Concentration of the weak acid $= \dfrac{(V_2 - V_1) \times N_1}{50}$

$=$

$=$ ....N

RESULT:

Equivalence volume for the Strong acid, $V_1 =$ -------------- ml.

Concentration of the strong acid in the given solution = --------------N

Equivalence volume for weak acid, $V_3 =$ ------------------ ml.

Concentration of the weak acid in the given solution = ------------- N

EXPERIMENT NO. 3

VISCOSITY COEFFICIENT OF A GIVEN LIQUID

AIM:

To determine the viscosity coefficient of given liquid using Ostwald's viscometer.

PRINCIPLE:

Viscosity arises due to internal friction between the moving layers of the molecules. A liquid flowing through a cylindrical tube of uniform diameter is expressed to move in the form of molecular layers. A layer close to the surface is almost stationary, while that at the axis of the tubes moves faster than any other, exerts a fraction on its nearest moving layer backwards. This property of liquid by which it retards or opposes motion between layers is called viscosity.

The coefficient of viscosity is defined as the tangential force per unit are required to maintain a unit viscosity gradient between any two successive layers of a liquid, situated at unit distance apart. The coefficient of viscosity of liquid is given by Poiseuille's formula.

$$\eta = \frac{\pi P r^4 t}{8 V l}$$

Where,

V = volume of the liquid   r = radius of the tube

l = length of the tube η = coefficient of viscosity of the liquid

t = time of flow   P = pressure difference between the two ends of the tube

Suppose equal volumes of two different liquids are allowed to flow through the same tube under identical conditions. Then, $\eta_1 / \eta_2 = t_1 d_1 / t_2 d_2$

Where t_1 and t_2 are the time taken by the definite volume of the standard liquid and sample liquid to flow through certain distance in the capillary tubes respectively d_1 and d_2 are densities of the standard liquid and sample liquid respectively, η_1 and η_2 are the viscosity coefficient of the standard liquid and sample liquid respectively.

By measuring the time taken by standard liquid (t_1) and the sample liquid (t_2) to flow through a certain distance in the capillary, and knowing the densities of the standard liquid (d_1) and the sample liquid (d_2) and coefficient of viscosity of the standard liquid (η_1), the viscosity of the sample liquid (η_2) can be calculated.

PROCEDURE:

Take a clean and dry viscometer and insert in water bath by fixing vertically to a stand. Pipette out a known volume (say 10ml) of the given organic liquid into the wider limb. Using a rubber tube, suck the liquid little above the upper mark of the narrow limb. Now allow the liquid to flow freely through the capillary. When the level of the liquid just crosses the upper mark, start a stop clock. Stop it when the liquid crosses the lower mark. Determine the time of flow of the liquid repeatedly.

Now remove the liquid from the viscometer, rinse with acetone and dry in an oven. Repeat the above procedure by taking same volume of distilled water. Determine the average time of flow and the coefficient of viscosity of the liquid using the relation $\eta_1 / \eta_2 = t_1 d_1 / t_2 d_2$

RESULT:

Viscosity coefficient of the liquid =--------------------- Ns / m²

OBSERVATION AND CALCULATIONS:

[Given liquid number =   ]

Sample	Time taken (in Seconds)			Average time (sec)
	Trial - 1	**Trial - 2**	**Trial - 3**	
Liquid				$t_{liquid} =$
Water				$t_{water} =$

Lab temperature   = -------------------^{0}C

Time taken by water (t_{water})   =--------------------- sec

Time taken by sample liquid (t_{liquid}) =---------------------sec

Density of water (d_{water})   = --------------------kg /m³

Density of liquid (d_{liquid})   = -------------------- kg / m³

Viscosity coefficient of water (η_{water}) = ----------------- X 10⁻³ Ns /m²

Therefore viscosity coeffcient of the liquid $\eta_{liquid} = \dfrac{\eta_{water} \times t_{liquid} \times d_{liquid}}{t_{water} \times d_{water}}$

$=$

$=$ Ns /m²

EXPERIMENT NO. 4

COLORIMETRIC ESTIMATION OF COPPER

AIM:
To estimate the amount of copper in the given solution by colorimetric method.

PRINCIPLE:
Colorimetry is one of the quantitative analytical techniques used to determine the concentration of the coloured species or chemical species, which is capable of forming a stable, coloured product, when treated with a suitable reagent under suitable conditions. In order to determine a chemical species by colorimetric method, the chemical species under investigation must be in the form of solution and it must confirm to Beers law and Lamberts law. Thus colorimeter is governed by two laws namely Beers and Lamberts law. The law which relates the absorption of light to the depth or thickness of the coloured liquid is called Lambert law and the other which is concerned with light absorption in relation to the concentration is known as Beers law.

The absorption of the light by a coloured species present in the absorbing medium depends upon the wavelength of the incident light, the thickness of the absorbing medium and the concentration of the coloured species. Each and every coloured species absorbs light to a maximum extent at a particular wavelength and is known as wavelength of maximum absorption. When a monochromatic light of intensity I_0, traverses through a transparent coloured medium, a part of light is absorbed (I_a), a part is reflected (I_r), and the remaining part is transmitted (I_t).

Then, $I_0 = I_a + I_r + I_t$, for air- glass interface

Ir is negligible, therefore, $I_0 = I_a + I_t$.

Lambert's law:
The law states that each layer of equal thickness absorbs an equal fraction of the light which transverses it. Thus when a ray of monochromatic light passes through an absorbing medium its intensity decreases exponentially as the thickness of the medium increases arithmetically

Beer's law:
Beers law is concerned with light absorption in relation to the solution concentration. It states that the intensity of a ray of monochromatic light decreases exponentially as the concentration of the absorbing medium increases arithmetically..

If I_0 is the intensity of incident light and I_t is the intensity of transmitted light then, the fraction of the light transmitted through the solution is I_t / I_0, is known as transmittance (T).

Then $\log 1/T = \log I_0/I_t$ is called absorbance (A) or optical density. The relation between absorbance (A), concentration (c) expressed in moles per dm^3, and the path length (t) expressed in cm is given by Beer – Lamberts law.

$$A = \log I_0 /I_t = \varepsilon. \ c. \ t.$$

Where is ε the molar absorptive coefficient, a constant for a given substance at a given wavelength. If the path length (t) is kept constant then, the absorbance is directly proportional to the concentration. Hence a plot of absorbance versus concentration gives a straight line.

Copper in the form of cupric ions when treated with ammonium produces a blue colored Cuprammonium complex. The complexation of Cu^{++} with ammonia molecules is as shown below.

$$Cu^{++} + 4NH_3 \longrightarrow [Cu (NH_3)_4]^{++}$$
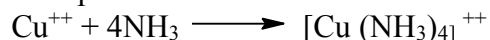
The cuprammonium complex has a maximum absorbance at 620nm.

A series of standard solutions of copper containing varying amounts of Cu^{++} are treated with an optimum amount of ammonia solution and diluted to a definite volume. The absorbance of the standard samples is measured at 620nm using a colorimeter and the absorbance are plotted against concentration to get the calibration curve (Beers Curve). A known volume of the test solution is also treated with the same amount of ammonia and diluted to the same volume. The absorbance of the test sample is measured at 620nm. Then the concentration of copper in the test solution is evaluated using the calibration curve.

PROCEDURE:

Take given copper sulphate solution in a burette. Transfer 5, 10, 15, 20 and 25ml of this solution into separate 50ml standard flasks. Add 5ml of 1:1 NH_3 to each of these flasks. Make up all these solutions to the mark with distilled water and shake well. Prepare a blank solution by diluting 5ml of 1:1 ammonia in another 50ml standard flask and mix well.

Take the test solution in another 50ml standard flask and add 5ml of ammonia to it. Dilute it up to the mark and mix well.

Take blank solution in a solvent cell and place in a photoelectric colorimeter which is set at 620nm wavelength. Adjust the instrument to zero absorbance. Then measure the absorbance of each of the standard solution and also of the test solution. Plot a graph of absorbance versus volume of copper sulphate and determine the concentration of copper in the test solution using the calibration curve.

RESULT:

Volume of the Cu^{++} in the test sample = ------------------- ml.

Wt. of Cu^{2+} ions present in the test solution = --------------- mg.
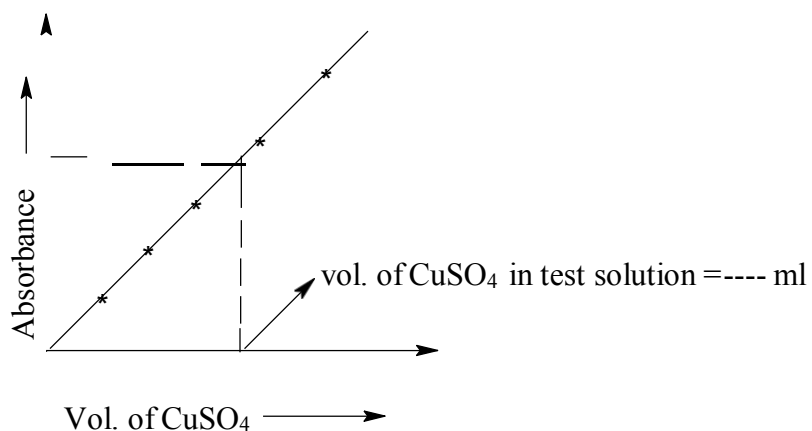
OBSERVATIONS AND CALCULATIONS:

[Given standard flask (test solution) number =   ]

Given 1ml of copper sulphate solution contains = 1.020 mg of copper.
Wavelength of maximum absorbance, λ_{max} = 620nm

Flask No.	Volume of copper sulphate solution (ml)	Weight of Cu^{2+} (mg)	Optical density or Absorbance
1	5		
2	10		
3	15		
4	20		
5	25		
	Test solution		

Graph:

vol. of CuSO₄ in test solution =---- ml

Vol. of CuSO₄ ⟶

From the graph,

Volume of Cu^{2+} in the test sample =------------------- ml.

Therefore weight of Cu^{2+} in the test solution = Volume of Cu^{2+} X 1.020 mg

$$=$$

$$= \rule{2cm}{0.4pt} mg.$$

RESULT:

Volume of the Cu^{++} in the test sample =------------------- ml.

Wt. of Cu^{2+} ions present in the test solution =---------------- mg.
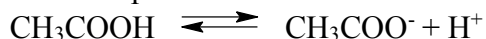
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EXPERIMENT NO. 5

pKa VALUE OF A WEAK ACID

AIM:
　　　　To determine the pKa value of a weak acid (acetic acid) using pH meter.

PRINCIPLE:
　　　　Organic acids such as Formic acid, Acetic acid, Oxalic acid etc. are considered as weak acids because they ionize feebly in water producing little H^+ ions. The dissociation of a weak acid such as CH_3COOH can be represented as follows.

$$CH_3COOH \rightleftharpoons CH_3COO^- + H^+$$

Applying the law of mass action, the equilibrium constant or dissociation constant is given by,

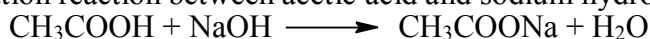$$Ka = \frac{[CH_3COO^-][H^+]}{[CH_3COOH]}$$

　　　　Since the dissociation of weak acid is partial, the concentration of ionic species is very much low when compared with that of unionized acid and therefore the value of K_a is very low. The dissociation constant of acetic acid and formic acid are 1.82×10^{-5} and 1.778×10^{-4} respectively at 25°c.
　　　　The dissociation constant is a measure of strength of an acid. Greater the dissociation constant, greater is the strength of an acid. It is convenient to express the strength of a weak acid in terms of pKa, a numerical value obtained by taking logarithm of Ka and changing the sign. i.e., $pKa = -\log_{10}Ka$.
　　　　Higher the value of Ka, smaller is the value of pKa. Therefore an acid with lower pKa value is relatively stronger than the one with higher pKa value.

　According to Hendersons equation, $pH = pKa + \log \dfrac{[salt]}{[acid]}$

Consider the neutralization reaction between acetic acid and sodium hydroxide.

$$CH_3COOH + NaOH \longrightarrow CH_3COONa + H_2O$$

　　　　In the above neutralization reaction, CH_3COOH is converted into CH_3COONa (salt). At half neutralization point, half of the acetic acid is in the form of CH_3COONa and hence concentration of acid and salt are same. Therefore the pH at half neutralization point is equal to pKa of week acid.

PROCEDURE:
　　　　Make up the weak acid solution kept in a 250ml standard flask to the mark with distilled water and shake well. Pipette out 50ml of this solution into a 100ml beaker. Immerse the glass electrode + calomel electrode assembly into it. Connect the electrode to a pH meter and measure the pH. Add NaOH from the burette to the beaker in increments of 0.5ml and measure pH after each addition. Initially pH increases slowly and at the equivalence point there will be a sudden jump. Record at least 4-6 readings after the equivalence point.
　　　　Plot a graph of $\Delta pH / \Delta V$ against volume of NaOH and determine the equivalence point. Plot another graph of pH against volume of NaOH and determine the half equivalence point. The pH at half equivalence point gives the pKa value of week acid.
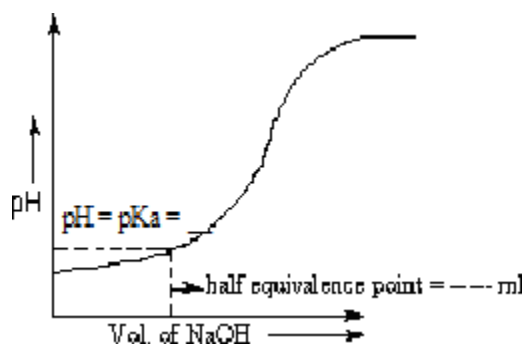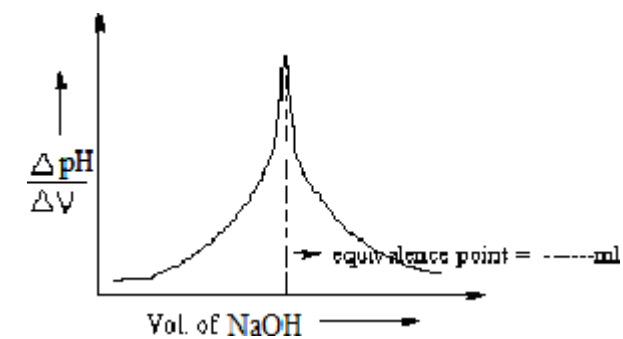
RESULT:

       pKa value of weak acid =--------------------- .

<u>OBSERVATIONS AND CALCULATIONS</u>:

[Given standard flask number =   ]

Volume of NaOH (ml)	pH	Δ pH (pH$_2$ – pH$_1$)	Δ V (V$_2$ - V$_1$) ml	Δ pH / ΔV
0.0		--------------	-----------------	----------------
0.5				

Graphs:

$$\frac{\Delta pH}{\Delta V}$$

equivalence point = ------ml

Vol. of NaOH

pH

pH = pKa =

half equivalence point = ---- ml

Vol. of NaOH

From the graph, equivalence point, V   =-------------------ml.

Therefore, half equivalence point,   V/2 =----------------- ml

At V/2, pH = pKa = ----------------

RESULT:
     pKa value of weak acid =------------------- .

EXPERIMENT NO. 6
FLAME PHOTOMETRY

AIM:

To estimate the sodium and potassium concentration in the given sample of water by flame photometry.

PRINCIPLE:

Emission of characteristic radiation by an element and the correlation of the emission intensity with the concentration of the element form the basis of flame photometry.

When a solution containing the sample element or ion is aspirated into the flame, a series of changes take place at the flame. First, the solvent gets evaporated leaving behind the salt in the flame. The salt then gets evaporated into vapors of the salt, which further undergo dissociation into the constituent atoms. Some of the metal atoms formed in the flame may absorbheat energy from the flame and get electronically excited to their higher energy level. Being unstable at the excited level, the atoms fall back to their ground state by emitting the energy difference between the excited and ground state, in the form of light radiation. The intensity of the light radiation emitted is proportional to the number of atoms in the excited state, which in turn is proportional to the number of atoms in the flame or the concentration of the solution fed into the flame. Thus concentration of the solution is related to the intensity of the emitted radiation. By measuring the intensity of the emitted radiation by a suitable detector device, the concentration can be determined. As different metals emit their characteristic radiations atdifferent wave lengths, they do not interfere with each other, even when they are present together. The different radiations emitted by the different metal atoms can be resolved by using adispersive unit, detected and measured for analysis. The sequence of changes taking place at the flame, when metal salt solution is aspirated may be summarized as follows.
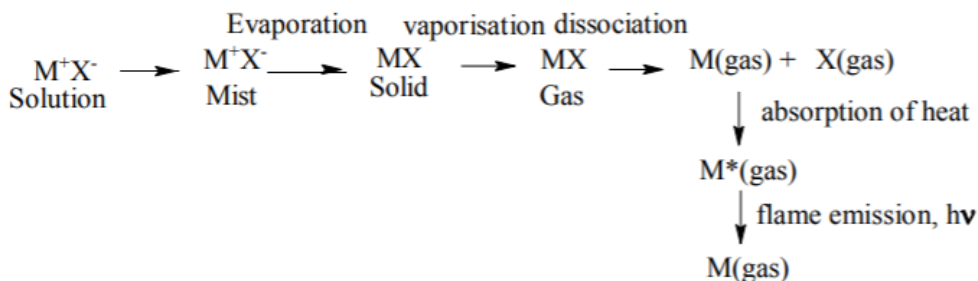
The intensity of the emitted radiation, measures as detector response is related to the concentration by an expression similar to Beer's relation,

$$E = k \, \alpha \, c,$$

Where E is the detector response, k is a constant, α is the efficiency of atomic excitation and c is the concentration of the solution.

The quantitative analysis by flame photometry can be carried out by calibration curve method. For this purpose a series of standard solutions of the analyte metal are taken, aspirated into the flame and the emission of each solution is measured in a flame photometer. Then the calibration curve is obtained by plotting the emission intensity against the concentration of the

standard solutions. The sample solution is properly diluted so that its emission is in the range of standards, and its emission is measured.

PROCEDURE:
For Sodium:
     A stock solution of sodium chloride provided has concentration of 100 ppm. (100 mg / litre) of sodium. Transfer exactly 3.0 ml, 6.0 ml, 9.0 ml, 12.0 ml, 15.0 ml, 18.0 ml and 21.0 ml of stock solution using a burette into seven separate 50ml volumetric standard flasks and dilute all the above solutions and the test solution with deionized water up to the mark. Shake well all the solutions for uniform concentration. Take the first standard solution in a small beaker and aspirate into the flame of the flame photometer. Measure the flame emission intensity of the standard by the flame photometer using sodium filter (598nm) in the photometer. Repeat the same for all the standard solutions with distilled water being sprayed to the flame in between the trials. Plot the calibration curve by taking emission response along the Y-axis and volume of the solution along the X-axis. Then aspirate the given test sample into the flame and note down the value of emission response. From the calibration curve compute the volume of Na in the test sample.
For Potassium:
     Repeat the same procedure with potassium chloride solution (100ppm).

RESULT:

For Sodium:
     Volume of unknown solution given   =_____**ml**
     Concentration of unknown solution   =_____**ppm**
For Potassium:
     Volume of unknown solution given   =_____**ml**
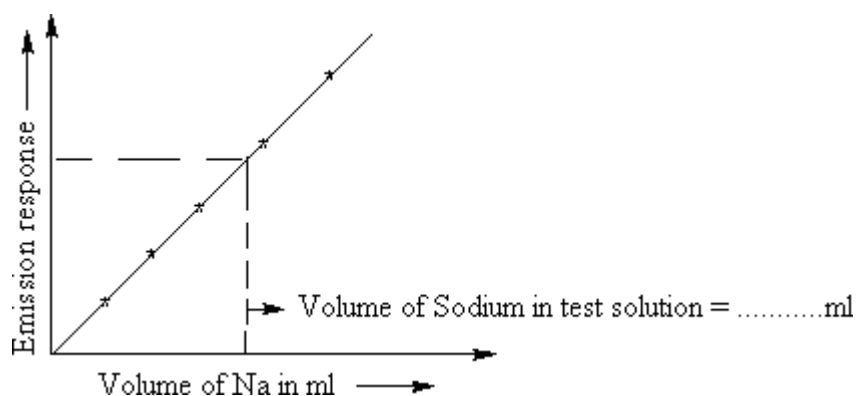   **Concentration of unknown solution**   =_____**ppm**

OBSERVATIONS AND CALCULATIONS:

For Sodium:
Concentration of the stock solution = 100 ppm of sodium = 100 mg / dm^3 of sodium

$$\text{Mass of sodium per cm}^3 = \frac{100\text{mg}}{1000\text{cm}^3} = 0.1 \text{ mg / cm}^3$$

Sl. No.	Volume of sodium solution taken (cm^3)	Mass of sodium (mg)	Concentration of sodium (ppm)	Emission Intensity from Flame Photometer
1.	3.0			
2.	6.0			
3.	9.0			
4.	12.0			
5.	15.0			
6.	18.0			
7.	21.0			
8.	Test Solution			

Graph:

Volume of test solution (from graph) =   cm^3
Mass of sodium in test solution = ... ml X 0.1mg

             = ... mg.

Concentration of Sodium in test solution   = $\dfrac{\text{Mass of Na in test solution x 1000mg}}{50}$

            = $\underline{\hspace{4cm}}$ mg/dm^3 or ppm.

For Potassium:
Concentration of the stock solution = 100 ppm of potassium
                 = 100 mg / dm^3 of potassium

Mass of potassiumper cm^3 = $\dfrac{100mg}{1000cm^3}$ = 0.1 mg / cm^3

Sl. No.	Volume of Potassium solution taken (cm^3)	Mass of potassium (mg)	Concentration of potassium (ppm)	Emission Intensity from Flame photometer
1.	3.0			
2.	6.0			
3.	9.0			
4.	12.0			
5.	15.0			
6.	18.0			
7.	21.0			
8.	Test Solution			

Graph:

Volume of test solution (from graph) =   cm³

Mass of potassium in test solution = ...ml X 0.1mg

                     = ...mg.

Concentration of Potassium in test solution   = Mass of K in test solution x 1000mg

                                                               50

                        = _____ mg/dm³ or ppm.

RESULT:

For Sodium:

       Volume of unknown solution given   =_____ml

       Concentration of unknown solution =_____ppm

For Potassium:

       Volume of unknown solution given   =_____ml

       Concentration of unknown solution =_____ppm

EXPERIMENT NO. 7

ESTIMATION OF TOTAL HARDNESS OF WATER

AIM:
To estimate the total hardness of the given sample of water using standard EDTA solution.

PRINCIPLE:
Hardness of water is due to the presence of dissolved salts (bicarbonates, chlorides & sulphates) of calcium, magnesium and other metal ions (less frequently iron & aluminium). Hard water does not give lather easily with soap, as the hardness causing ions react with soap to form insoluble soap. Temporary hardness is due to the bicarbonates of calcium and magnesium [$Ca(HCO_3)_2$ and $Mg(HCO_3)_2$], which can be removed by boiling. On boiling, the bicarbonates are converted into insoluble carbonates of calcium and magnesium.

$$Ca(HCO_3)_2 \longrightarrow CaCO_3 + CO_2 + H_2O$$

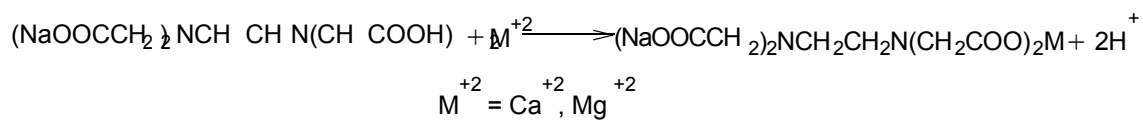$$Mg(HCO_3)_2 \longrightarrow MgCO_3 + CO_2 + H_2O$$
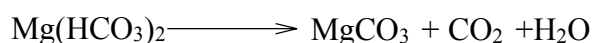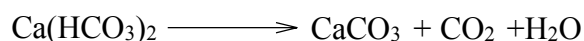
Temporary hardness can also be removed by Clarks process using CaO (Chemical method). Permanent hardness is due to the presence of other soluble chlorides and sulphates of calcium, magnesium and other metals, which can be removed by following chemical treatments.
(i). Lime-soda method using lime (CaO) and soda (Na_2CO_3).
(ii). Zeolite method using natural sodium aluminium silicates ($Na_2Al_2Si_2O_8$).
(iii) Ion exchange or demineralization process using synthetically prepared organic complex compounds called resins, capable of replacing all the cations by H^+ ions and anions by OH^- ions. The sum of temporary hardness and permanent hardness is known as total hardness. The hardness is expressed in parts of $CaCO_3$ equivalents (i.e. mg/dm^3 or mg / litre). EDTA is **ethylene diamine tetra acetic acid**. The structure is
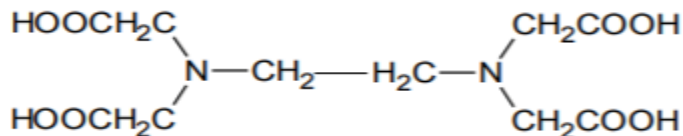
Hardness of water is determined by titrating a known volume of water sample with EDTA, using Eriochrome black –T (EBT) as indicator, in the presence of ammonium hydroxide – ammonium chloride buffer (pH 10). Ethylene diamine tetra acetic acid (EDTA) or its disodium salt is a strong complexing agent, reacts quantitatively with the metal ions to form a soluble, stable, 1:1 complex.

$$(NaOOCCH_2)_2NCH_2CH_2N(CH_2COOH)_2 + M^{+2} \longrightarrow (NaOOCCH_2)_2NCH_2CH_2N(CH_2COO)_2M + 2H^+$$

$$M^{+2} = Ca^{+2}, Mg^{+2}$$

When a few drops of the indicator EBT are added, it forms a wine red colored complex with the metal ions.

$$M^{2+} + EBT \longrightarrow M\text{ - EBT complex}$$
$$(\text{wine red})$$

24
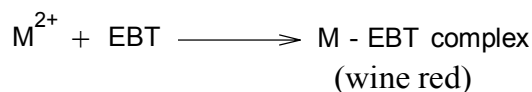
When the solution is titrated, EDTA reacts, preferentially with free metal ions present in the solution. Near the end point, when the free metal ions are exhausted in the solution, further addition of EDTA dissociates the M – EBT complex, consumes the metal ions and releases free indicator, which is blue in color. Therefore the colour change is from wine red to blue.

The indicator EBT shows the colour change at pH range 9-11. Therefore the pH of the solution is maintained around 10 using NH_4OH - NH_4Cl buffer.

EDTA being insoluble in water, it is generally used as a disodium salt which readily dissolves in water. A standard solution of EDTA is prepared by dissolving a known weight of EDTA in distilled water and making up to the mark in a 250ml standard flask.
Molecular weight of disodium salt of EDTA = 372.24.

$$\text{Molarity of EDTA solution} = \frac{\text{Weight taken}}{\text{Molecular weight}} \times 4$$

The structure of EBT is
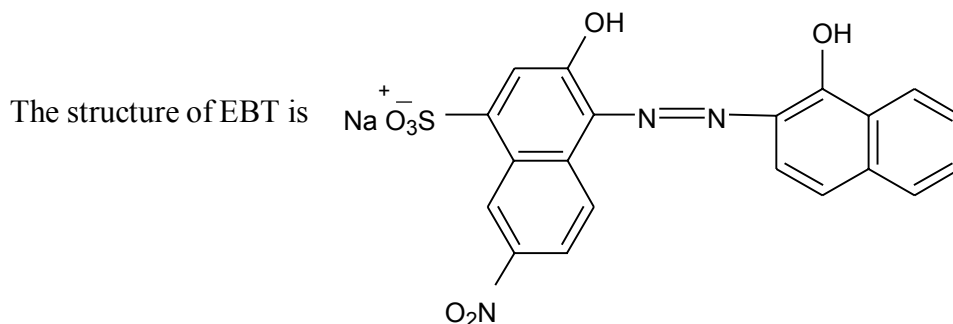
PROCEDURE:

PREPRATION OF STANDARD SOLUTION OF EDTA:

Weigh accurately about 0.8g of EDTA given in a weighing bottle and transfer in to a 250ml standard flask. Dissolve completely in distilled water. If necessary, add 2ml of NH_3. Makeup the solution to the mark using distilled water and shake well for uniform concentration. Takeit in a 50ml burette.

ESTMATION OF HARDNESS OF WATER:

Pipette out 25ml of given hard water into a clean conical flask. Add 2ml of NH_4OH – NH_4Cl buffer and 2 drops of EBT indicator. Titrate the resulting wine red colored solution against EDTA until clear blue solution is obtained. No tinge of reddish blue should remain at the end point. Repeat the titration to get agreeing values.

RESULT:

Hardness of water is = --------------------mg /dm³ or mg /litre or ppm of CaCO₃

OBSERVATIONS AND CALCULATIONS:

Weight of EDTA dissolved in 250ml standard flask   W = ---------------- g.

Molarity of EDTA solution = $\dfrac{\text{Weight taken}}{\text{Molecular weight}} \times 4$

i.e., $M_{EDTA} = \dfrac{W \times 4}{372.24}$

$= \dfrac{ \times 4}{372.24}$

$= \underline{} M$

ESTMATION OF TOTAL HARDNESS OF WATER:

Burette   : Standard EDTA solution
Conical flask **:** 25ml hard water + 2ml of NH$_4$OH - NH$_4$Cl buffer
Indicator   **:** 2 drops of Eriochrome Black – T
End point   **:** Change of color from wine red to blue

Trial No.	1	2	3
Final Burette reading (ml)			
Initial burette reading (ml)			
Volume of EDTA (ml)			

Agreeing value V_{EDTA} = ----------------------ml

Molarity of CaCO$_3$ in hard water = $\dfrac{M_{EDTA} \times V_{EDTA}}{V_{\text{Hard water}}}$

i.e., $M_{CaCO3} = \dfrac{}{25} M$

i.e., $M_{CaCO3} = \dfrac{}{} M$

Therefore amount of $CaCO_3$ per litre of hard water = M_{CaCO3} X Mol.wt. of $CaCO_3$

$$= \qquad X\ 100$$

$$= \text{-------------} \ g\ /dm^3 (\mathbf{Z})$$

Therefore hardness of water = $\mathbf{Z}$ X 1000 mg/dm^3

$$=$$

$$= \text{--------------} \ mg/dm^3 \ or \ mg/L \ or \ ppm \ of \ CaCO_3$$

RESULT:

Hardness of water is = -------------------- mg /dm³ or mg /litre or ppm of CaCO₃

EXPERIMENT NO. 8

ESTIMATION OF CALCIUM OXIDE IN CEMENT SOLUTION

AIM:

To estimate calcium oxide present in the given sample of cement solution.

PRINCIPLE:

Cement contains compounds of calcium aluminium, magnesium, iron, and insoluble silica. When dissolved in acid silica remains undissolved. On treating with ammonia, aluminium and iron can be precipitated as their hydroxides and separated. The provided solution of cement therefore contains calcium and magnesium ions.

To estimate the calcium content in the given solution, known volume of it is titrated with standard EDTA solution. In the presence of magnesium, calcium can be titrated with EDTA by using Patton and Reeder's indicator at pH values between 12 and 14. The indicator combines with calcium ions forming red colored complex.

$$Ca^{2+} + Indicator \longrightarrow Ca\text{- indicator complex.}$$
$$\text{(Wine red)}$$
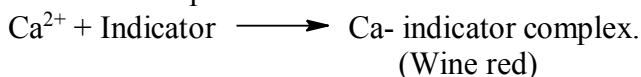
Near the end point, when free calcium ions are exhausted in the solution, further addition of EDTA dissociates Ca-indicator complex, consumes the calcium ions and releases free indicator, which is blue in colour. Therefore the colour change is wine red to blue.

The standard EDTA solution is prepared by dissolving a known weight of disodium salt of EDTA in a known volume of the solution.

Molecular weight of disodium salt of EDTA = 372.24

PROCEDURE:

PREPARATION OF STANDARD EDTA SOLUTION:

Weigh accurately about 0.8g of EDTA given in a weighing bottle and transfer in to a 250ml standard flask. Dissolve completely in distilled water. (If necessary, add 2ml of NH_3). Make up the solution to the mark using distilled water and shake well for uniform concentration. Take it in a 50ml burette.

ESTIMATION:

Pipette out 25ml of given cement solution into a clean conical flask. Add 5ml of 1:1 of glycerol with constant shaking of the contents of the flask followed by 5ml of diethyl amine buffer (so as to bring the pH of the solution to 12). Add 1 test tube full of 4 N sodium hydroxide and mix the solution thoroughly. Add 2 test tubes full of ion exchange water and 2 drops Patton and Reeder's indicator. Titrate the resulting solution in the conical flask against EDTA solution taken in the burette until wine red colour changes to clear blue. Repeat the titration to get agreeing values.

RESULT:

Weight of calcium oxide in a litre of the given cement solution = --------------- g.

OBSERVATIONS AND CALCULATIONS:

Preparation of standard solution of EDTA:

Weight of EDTA dissolved in 250ml,   W = -------------------- g

$$\text{Molarity of EDTA solution} = \frac{\text{Weight of EDTA in 250ml}}{\text{Molecular weight of EDTA}} \times 4$$

$$M_{EDTA} = \frac{W \times 4}{372.24}$$

$$= \frac{\rule{2cm}{0.4pt} \times 4}{372.24}$$

$$= \rule{2cm}{0.4pt} M$$

Estimation of CaO:

In Burette   **:** Standard EDTA solution
In Conical flask **:** 25ml of cement solution + 5 ml of diethyl amine buffer + 5ml of 1:1 of glycerol
                            + 1 test tube of 4N sodium hydroxide + 2 test tube of ion exchange water.
Indicator   **:** 2 drops of Patton and Reeder's indicator
End point   **:** Change of colour from wine red to blue

Trial No.	1	2	3
Final Burette reading (ml)			
Initial Burette reading (ml)			
Volume of EDTA (ml)			

Agreeing value, V_{EDTA} = -------------------- ml

Molarity of CaO in cement solution $M_{CaO} = \dfrac{M_{EDTA} \times V_{EDTA}}{V cement}$

$$M_{Cao} = \dfrac{}{25}$$

$= \underset{\cdots\cdots\cdots\cdots\cdots}{} M$

Therefore amount of CaO present in a litre of the cement solution

$= M_{CaO} \times$ Mol.wt. of CaO

$= \underset{\cdots\cdots\cdots\cdots}{} \times 56.08$

$= \underset{\cdots\cdots\cdots\cdots}{} g$

RESULT:

Weight of calcium oxide in a litre of the given cement solution = ---------------- g.

EXPERIMENT NO. 9

ESTIMATION OF COPPER IN BRASS

AIM:

To estimate the percentage of copper in given sample of brass, using standard sodium thiosulphate solution.

PRINCIPLE:

Brass is an alloy of copper and zinc. When dissolved in Conc. Nitric acid, both the metals get converted into their nitrates.

$$Cu + 4HNO_3 \longrightarrow Cu(NO_3)_2 + 2H_2O + 2NO_2 \uparrow$$

(Brown fumes)
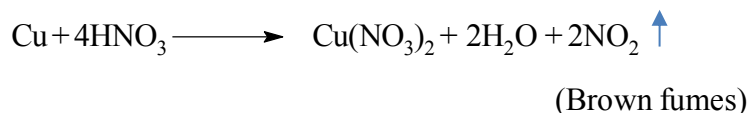
When brass solution is treated with excess of potassium iodide solution, cupric ions oxidize potassium iodide to liberate equivalent quantity of iodine.

$$2 Cu (NO_3)_2 + 4KI \longrightarrow Cu_2I_2 + 4KNO_3 + I_2$$
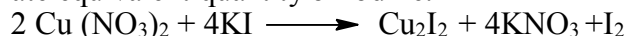
The liberated iodine is treated with sodium thiosulphate solution using starch as indicator.

$$I_2 + 2Na_2S_2O_3 \longrightarrow 2NaI + Na_2S_4O_6$$

(Sodium Tetrathionate)
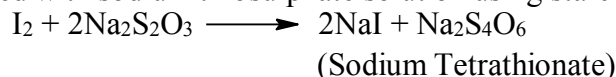
The oxides of nitrogen present in the brass solution are destroyed by adding urea. The presence of nitrogen oxides will be responsible for the liberation of extra iodine from KI, as they are also good oxidizing agents.

The nitric acid present in the brass solution is neutralized by adding NH_4OH solution till a pale blue precipitate of $Cu(OH)_2$ is obtained. Otherwise, being an oxidizing agent, nitric acid may also liberate iodine from KI. The $Cu(OH)_2$ precipitate is dissolved in dilute acetic acid. Other mineral acids are not preferable as they will bring down the pH to a very small value, at which the liberation of I_2 from KI by Cu^{2+} in not quantitative.

Starch reacts with iodine to form a blue coloured complex. At the end point, when free iodine is exhausted in the solution, addition of sodium thiosulphate dissociates the starch iodine complex; added quantity consumes iodine and liberates starch, there by discharging the blue colour. For the brass solution the colour change is from blue to white as Cu_2I_2 precipitate is present in the solution.

Starch indicator is added near the end point and not in the beginning for the following reasons.

1. At high concentration of I_2, starch may form an insoluble starch – iodine complex.
2. The iodine also gets adsorbed on colloidal starch particles in the solution. The adsorbed iodine cannot be liberated by the addition of sodium thiosulphate, and the amount of iodine getting adsorbed is more when iodine concentration is high.

Thus the error is minimized by adding starch near the end point. During the liberation of I_2 from KI, Cu^{2+} gets reduced to Cu^+ with change in oxidizing state by 1. Thus equivalent weight of Cu = atomic weight = 63.54

PROCEDURE:

PREPARATION OF BRASS SOLUTION:

Weigh accurately the given brass foil and transfer into a 250ml beaker, add ¼ test tube of conc. nitric acid. Dissolve the brass and dilute the resulting solution with 1 test tube of water.

Add about 1g of urea and boil for 1-2 minutes. Cool the solution to room temperature. Transfer the contents to a 250ml standard flask using funnel, make up to the mark with distilled water and shake well for uniform concentration.

ESTIMATION:

Pipette out 25ml of brass solution into a clean conical flask. Add NH_4OH solution in drops until bluish white precipitate persists. Dissolve the precipitate by adding acetic acid in drops followed by the 5ml of acetic acid in excess. Also add 10ml of 10% KI to the conical flask and titrate the liberated iodine against standard $Na_2S_2O_3$ solution taken in the burette until the solution in the flask changes to pale yellow. Now add 2ml of starch indicator. The solution turns dark blue. Continue the titration by adding the sodium thiosulphate ($Na_2S_2O_3$) solution from the burette slowly and carefully, until the blue colour just disappears and a milky white solution remains. Repeat the titration to get agreeing values.

RESULT:

Percentage of copper in the given sample of brass = ----------------.

OBSERVATIONS AND CALCULATIONS:

PREPRATION OF BRASS SOLUTION:

Weight of brass W = --------------- g

ESTIMATION:

In burette	: $Na_2S_2O_3$
In conical flask	: 25ml of brass solution + NH_4OH in drops + Dilute acetic acid + 10ml of 10% KI solution.
Indicator	: Starch solution near the end point
End point	: Dark blue to milky white

Trial No.	1	2	3
Final burette reading (ml)			
Initial burette reading (ml)			
Volume of Na₂S₂O₃ added (ml)			

Agreeing value, $V_{Na_2S_2O_3}$ = ... cm^3

Normality of $Na_2S_2O_3$ solution, $N_{Na_2S_2O_3}$ =...................... N

Strength of Cu in brass solution $N_{Cu^{2+}}$ = $\dfrac{N_{Na_2S_2O_3} \ X \ V_{Na_2S_2O_3}}{V_{Cu^{2+}}}$

$N_{Cu^{2+}}$ = $\dfrac{}{25}$

= ....N

Therefore, weight of Cu present in 250ml of the brass solution = $\dfrac{N_{Cu^{+2}} \ X \ Eq.wt. \ of \ Cu}{4}$

= $\dfrac{ X \ 63.54}{4}$

= ....g.

Therefore, percentage of Cu in brass = $\dfrac{Weight \ of \ Cu \ in \ 250ml \ of \ the \ brass \ solution \ X \ 100}{Weight \ of \ brass}$

= $\dfrac{ X \ 100}{}$

= ------------ .

RESULT:
   Percentage of copper in the given sample of brass =---------------.

EXPERIMENT NO. 10

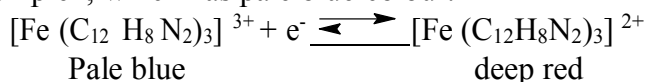## DETERMINATION OF COD OF INDUSTRIAL WASTE WATER

AIM:

To estimate the chemical oxygen demand (COD) of waste water.

PRINCIPLE:

Chemical oxygen demand (COD) parameter has been introduced to measure the total oxidisable impurities present in the sewage. This includes both biologically oxidisable and biologically inert but chemically oxidisable impurities in the water. It is the amount of oxygen equivalent to use while oxidizing the chemically oxidisable impurities in the water, with a strong chemical oxidant, $K_2Cr_2O_7$ in acid medium. It is expressed in mg dm^{-3}or ppm.

A known volume of the waste water sample is refluxed with excess of $K_2Cr_2O_7$ solution in sulphuric acid medium and in the presence of Ag_2SO_4 and $HgSO_4$. $K_2Cr_2O_7$ oxidizes all oxidisable impurities. Ag_2SO_4 catalyses the oxidation of straight chain organic compounds, aromatic compounds and pyridine. $HgSO_4$ avoids the interference of Cl^- ions forming soluble complex with them. In the absence of $HgSO_4$, Cl^- precipitates silver ions as AgCl. The amount of unreacted $K_2Cr_2O_7$ is determined by titration with standard Mohr's salt (ferrous ammonium sulphate) solution. The amount of dichromate solution consumed corresponds to COD of water sample. To calculate the COD, a blank titration without the water sample is carried out.
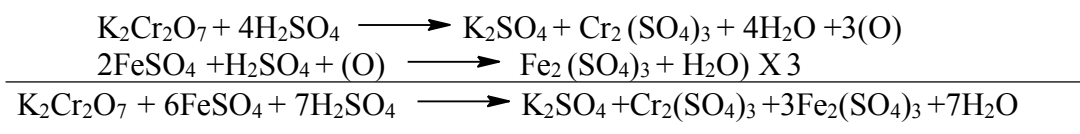
Ferroin (1, 10 – phenanthroline iron (II) complex.) is used as the indicator in the titration to detect the end point. The indicator is intensely red colour. Strong oxidizing agent oxidizes the indicator to iron (III) complex, which has pale blue colour.

$$[Fe\ (C_{12}\ H_8\ N_2)_3]^{\ 3+} + e^- \ \rightleftarrows\ [Fe\ (C_{12}H_8N_2)_3]^{\ 2+}$$
$$\text{Pale blue} \qquad\qquad\qquad \text{deep red}$$

As long as $K_2Cr_2O_7$ is present in the solution, the indicator is in the oxidized form imparting blue colour to the solution. The solution becomes blue green as the titration continues due to the formation of Cr (III) ions as result of reduction of $Cr_2O_7^{2-}$ ions. When $K_2Cr_2O_7$ is completely exhausted in the solution, the oxidized form of the indicator gets reduced to the reduced form, imparting the red brown colour to the solution. Therefore the end point is marked by the colour changes from blue green to red brown. The end point is sharp only at high acid concentration and therefore the solution is kept at a very high acidity of H_2SO_4.

A standard solution of Mohr's salt is prepared by dissolving a known weight of the salt in a known volume of the solution. A test tube of dil. H_2SO_4 acid is added during dilution to prevent the hydrolysis of the salt is in the solution.

The reaction between Mohr's salt and dichromate can be represented as follows.

$$K_2Cr_2O_7 + 4H_2SO_4 \longrightarrow K_2SO_4 + Cr_2\ (SO_4)_3 + 4H_2O + 3(O)$$
$$2FeSO_4 + H_2SO_4 + (O) \longrightarrow Fe_2\ (SO_4)_3 + H_2O)\ X\ 3$$

$$K_2Cr_2O_7 + 6FeSO_4 + 7H_2SO_4 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + 3Fe_2(SO_4)_3 + 7H_2O$$

Equivalent weight of Mohr's salt = its molecular weight = 392.

PROCEDURE:

PREPARATION OF STANDARD MOHR'S SALT SOLUTION:
  Weigh accurately the given Mohr's salt and transfer it into a clean standard flask. Add about 1 test tube of dil. H_2SO_4 to the flask. Dissolve the salt, make up to the mark with distilled water and shake well for uniform concentration. Take this solution in a burette.

ESTIMATION:
  Pipette out 25ml of waste water into a 250ml conical flask. Add 1g of $HgSO_4$ followed by 2 test tubes of silver sulphate / sulphuric acid solution to it. Also pipette out 25ml of given potassium dichromate solution into it. Add a porcelain piece, attach the flask with a reflux condenser and reflux the mixture for half an hour. Cool the flask to room temperature; wash the inner side of the condenser with distilled water into the same flask.
  Now add 1 test tube of 1:1 sulphuric acid and 1-2 drops of ferroin indicator. Titrate the mixture with standard Mohr's salt solution till the colour changes from bluish green to dark red. Repeat the titration to get agreeing values.

BLANK TITRATION:
Repeat the above procedure by taking 25ml of distilled water in place of waste water.

RESULT:
  COD of the given sample of waste water =--------------------ppm or mg/ dm^3.

OBSERVATIONS AND CALCULATIONS:

Weight of Mohr's salt transferred to the 250ml standard flask, W = ---------------- g.

$$\text{Normality of Mohr's salt solution} = \frac{W \times 4}{\text{Eq. wt. of Mohr's salt}}$$

$$N_1 = \frac{\quad\quad\quad}{392}$$

$$\underline{\quad\quad\quad\quad\quad}N$$

Volume of Mohr's salt consumed in blank titration, V_2 =------------ ml

ESTIMATION:

Solution in the burette   **:** Standard Mohr's salt solution.
Solution in the conical flask **:** 25ml refluxed solution + 1 test tube of 1:1 H_2SO_4
Indicator   **:** 1-2 drops of Ferroin indicator.
End point   **:** Change of colour from bluish green to red.

Trial No.	1	2	3
Final burette reading (ml)			
Initial burette reading (ml)			
Volume of Mohr's salt solution (ml)			

Agreeing value V_1 =------------------------- ml

Therefore volume of $K_2Cr_2O_7$ solution consumed by oxidisable impurities in terms of volume of Mohr's salt solution = $(V_2 - V_1)$

$$=$$

$$= \underline{\hspace{3cm}} ml$$

1000ml of 1N Mohr's salt solution   = 8 g of oxygen

Therefore 1ml of 1NMohr's salt solution   = 8 / 1000 g of oxygen

Therefore (V_2-V_1) ml of N_1 normal Mohr's salt solution, W_2 = $\dfrac{8 \times (V_2-V_1) \times N_1}{1000}$ mg of Oxygen.

$$= \dfrac{8 \times (V_2-V_1) \times N_1 \times 1000}{1000} \text{ mg of Oxygen.}$$

$$= 8 \times (V_2-V_1) \times N_1 \text{ mg of Oxygen.}$$

$$=$$

$$= \text{------------ mg of Oxygen}$$

Therefore, COD of Waste water $= \dfrac{W_2 \times 1000}{25}$ $=$ $\underline{\hspace{2cm}}$

$$= \text{------------ mg /dm}^3$$

RESULT:

         COD of the given sample of waste water =--------------------ppm or mg/ dm³.

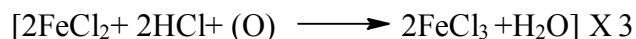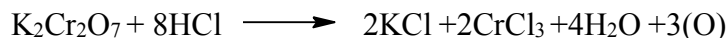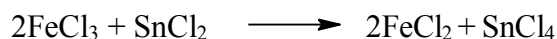# EXPERIMENT NO. 11

ESTIMATION OF IRON IN HAEMATITE

AIM:

To estimate the amount of iron present in the given haematite ore solution.

PRINCIPLE:

Haematite is an oxide ore of iron, existing as ferric oxide (Fe_2O_3). On treating with HCl, Fe_2O_3 dissolves in the acid as ferric chloride.

$$Fe_2O_3 + 6HCl \longrightarrow 2FeCl_3 + 3H_2O$$
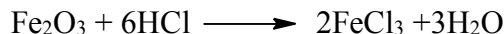
Since iron in $FeCl_3$ is already in the highest possible stable oxidation state (Fe^{3+}) it cannot be oxidized further. Therefore it is first reduced to ferrous state and then titrated with potassium dichromate solution, which is an oxidizing agent. Ferric chloride is reduced by stannous chloride in the presence of hydrochloric acid.

$$2FeCl_3 + SnCl_2 \longrightarrow 2FeCl_2 + SnCl_4$$

$$K_2Cr_2O_7 + 8HCl \longrightarrow 2KCl + 2CrCl_3 + 4H_2O + 3(O)$$

$$[2FeCl_2 + 2HCl + (O) \longrightarrow 2FeCl_3 + H_2O] \text{ X } 3$$

$$\overline{K_2Cr_2O_7 + 14HCl + 6FeCl_2 \longrightarrow 2KCl + 2CrCl_3 + 6FeCl_3 + 7H_2O}$$
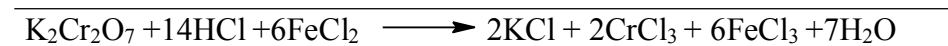
From the equation, 2 moles of $FeCl_2 \equiv$ 2moles of $FeCl_3 \equiv$ 1atom of oxygen.
Therefore the equivalent weight of iron = its atomic weight = 55.85

A slight excess of stannous chloride is added to ensure complete reduction of $FeCl_3$ to $FeCl_2$. The excess of stannous chloride added is destroyed by adding strong solution of mercuric chloride ($HgCl_2$). Since $SnCl_2$ can reduce potassium dichromate.

$$SnCl_2 + 2HgCl_2 \longrightarrow SnCl_4 + Hg_2Cl_2 \downarrow$$
$$\text{Silky white}$$
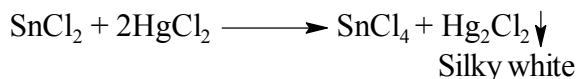
A silky white precipitate of mercurous chloride should be obtained. A black precipitate of finely divided mercury may be produced if too much $SnCl_2$ is present or mercuric chloride solution is added slowly. Therefore the trial must be discarded if a black precipitate is produced or no precipitate is formed on the addition of mercuric chloride because the complete reductionof ferric ions to ferrous ions is not ensured.

Potassium ferricyanide is used as external indicator to detect the end point of the titration. Potassium ferricyanide reacts with ferrous ions to produce an intense deep blue color to the formation of ferrous cyanide complex.

$$2K_3[(Fe(CN)_6] + 3Fe^{2+} \longrightarrow Fe_3[Fe(CN)_6]_2 + 6K^+$$
$$\text{Ferrous ferricyanide}$$
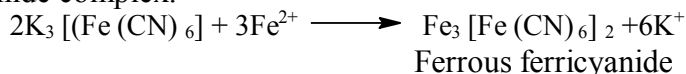
Therefore when a drop of titrated solution is treated with a drop of potassium ferricyanide solution, blue color is developed as long as ferrous ions are present in the solution. At the end point when ferrous ions are completely converted into ferric ions a drop of titrated solution does not develop blue color with the indicator.

A standard solution of $K_2Cr_2O_7$ is prepared by dissolving a known weight of the salt in known volume of water.

PROCEDURE:

PREPARATION OF STANDARD $K_2Cr_2O_7$ SOLUTION:

   Weigh the given $K_2Cr_2O_7$ crystals and transfer it into a 250ml standard flask. Dissolve the crystals in distilled water. Dilute the solution up to the mark and shake well for uniform concentration.

ESTIMATION OF IRON:

   Pipette out 25ml of given haematite ore solution into a clean 250ml conical flask. Add 5ml of conc. HCl and heat to boiling. To the hot solution, add $SnCl_2$ solution drop by drop with swirling until the yellow colour disappears completely. Add 2 more drops in excess. Cool the solution and add 5ml of $HgCl_2$ solution and shake well. A silky white precipitate should be obtained. (If black precipitate or no precipitate is obtained, discard the product).

   Take $K_2Cr_2O_7$ solution in a burette. Place the drops of freshly prepared $K_3[(Fe(CN)_6)]$ solution on the dry surface of a wax paper or white porcelain tile. Add a small quantity of potassium dichromate solution to the conical flask and shake well. With the help of a glass rod bring a drop of the titrated solution in contact with the indicator drop. The colour of the indicator drop turns blue. The addition of $K_2Cr_2O_7$ solution and testing with the indicator drop is continued until the titrated solution does not develop blue colour with the indicator. This is the end point of the titration. Repeat the titration for concordant values.

RESULT:
Weight of iron per litre of the given solution = ------------------ g.

<u>**OBSERVATIONS AND CALCULATIONS:**</u>

PREPARATION OF STANDARD $K_2Cr_2O_7$ SOLUTION:

Weight of $K_2Cr_2O_7$ crystals transferred to the standard flask, W = ---------------- g

$$\text{Normality of } K_2Cr_2O_7, \ N_{K_2Cr_2O_7} = \frac{\text{Wt. of } K_2Cr_2O_7 \ X \ 4}{\text{Eq.wt. of } K_2Cr_2O_7}$$

$$= \frac{\text{---------} \ X \ 4}{49.03}$$

$$= \text{----------------} N$$

ESTIMATION OF IRON:

Burette   **:** $K_2Cr_2O_7$
Conical flask **:** 25ml of ore solution + ½ test tube of conc. HCl, boil + $SnCl_2$, cool + 5ml of
      $HgCl_2$ solution
Indicator **:** Potassium ferricyanide (external Indicator)
End point **:** No development of blue color

Trial No.	1	2	3
Final burette reading (ml)			
Initial burette reading (ml)			
Volume of K₂Cr₂O₇ added (ml)			

Agreeing value, $V_{K_2Cr_2O_7}$ = ------------- ml

Normality of haematite solution $N_{haematite}$ = $\dfrac{N_{K_2Cr_2O_7} \times V_{K_2Cr_2O_7}}{V_{haematite}}$

$N_{haematite}$ = $\dfrac{}{25}$

= ....N

Therefore weight of iron per liter of the solution = $N_{haematite}$ X Eq.wt. of Fe

= X 55.85

= ....g.

RESULT:

Weight of iron per litre of the given solution = ------------------ g.

EXPERIMENT NO. 12

ESTIMATION OF CHLORINE IN BLEACHING POWDER

AIM:

Estimation of percentage of available chlorine in the given sample of bleaching powder

PRINCIPLE:

Bleaching powder, also known as chlorinated lime having a smell of chlorine and is readily soluble in water. It consists of a mixture of calcium hypochlorite $Ca(OCl)_2$ and calcium chloride $CaCl_2$, and some amount $Ca(OH)_2$. $Ca(OCl)_2$ is responsible for the bleaching action of bleaching powder . On treatment with glacial acetic acid, it liberates chlorine gas (Cl_2).

$$Ca (OCl)_2 + 2CH_3COOH \rightarrow Ca(CH_3COO)_2 + H_2O + Cl_2$$
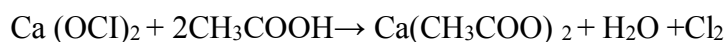
The amount of chlorine liberated by the action of an acid on bleaching powder $Ca(OCl)_2$ is termed as available chlorine.
The liberated chlorine oxidizes KI in presence of acid and liberate out an equivalent amount of iodine.

$$Cl_2 + 2KI \rightarrow 2KCl + I_2$$
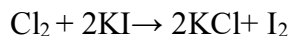
The liberated iodine is titrated with sodium thiosulphate solution using starch as indicator.

$$I_2 + 2Na_2S_2O_3 \longrightarrow 2NaI + Na_2S_4O_6$$
$$\text{(Sodium Tetrathionate)}$$
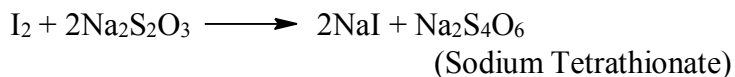
The chlorine content of bleaching powder varies from 35 – 40%.

PROCEDURE:

Weigh accurately the given bleaching powder sample and transfer into a clean 250 ml standard flask. Dissolve the powder in distilled water. Makeup to the mark with distilled water and mix well.
Pipette out 25ml of the turbid liquid into a conical flask. Add 1 test tube full of water, 5ml of glacial acetic acid and 10 ml of10% KI solution. Titrate the liberated iodine against standard $Na_2S_2O_3$ solution taken in the burette until the solution in the flask changes to pale yellow. Now add 2ml of starch indicator. The solution turns dark blue. Continue the titration by adding the sodium thiosulphate ($Na_2S_2O_3$) solution from the burette slowly and carefully, until the blue colour just disappears. Repeat the titration to get agreeing values.

RESULT:

Percentage of available chlorine in the given sample of bleaching powder = _____

OBSERVATIONS AND CALCULATIONS:

Mass of bleaching powder transferred to std. flask W=_____g

ESTIMATION:

In burette: $Na_2S_2O_3$ solution

In conical flask: 25 ml of Bleaching powder suspension + 1test tube of distilled water +5ml Glacial Acetic Acid + 10ml 10% KI.

Indicator: Starch solution is near the end point of the titration

End Point: Dark blue to colorless

Trial No.	1	2	3
Final burette reading (ml)			
Initial burette reading (ml)			
Volume of Na₂S₂O₃ added (ml)			

Agreeing value, $V_{Na_2S_2O_3}$ = -------------------- ml (X)

Normality of sodium thiosulfate solution = N_1 = _____

Volume of sodium thiosulfate solution required for 25 ml of bleaching powder suspension **X**=----------- ml

1000 ml of 1N $Na_2S_2O_3$ solution = 35.46 g of chlorine.

Therefore 1ml of N_1 $Na_2S_2O_3$ = 35.46 X N_1 / 1000 = "a" gm of chlorine

$$a = \frac{35.46 \ X}{1000} =$$

Amount of chlorine present in 25 ml of bleaching powder suspension = a X **X** = Z g

$$=$$
$$=$$

Therefore amount of chlorine in the 250ml of the solution = Z x10 g

$$=$$

Percentage of chlorine in the given sample of bleaching powder = $\frac{Z \ x10 \ x \ 100}{W}$

$$=$$

RESULT:

Percentage of available chlorine in the given sample of bleaching powder =_____

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EXPERIMENT NO. 13
# DETERMINATION OF ALKALINITY(OH^- , CO_3^{2-} , HCO_3^-) OF WATER SAMPLE

AIM:

To determine the type & amount of alkalinity present in the given sample of water, using, standard HCl solution.

PRINCIPLE:

The alkalinity of water is due to the presence of hydroxyl ions (OH^-) from NaOH or KOH, carbonate ions (CO_3^{2-}) from sodium carbonate or potassium carbonate and bicarbonates (HCO_3^-) from sodium bicarbonate or potassium bicarbonate or temporary hardness (calcium bicarbonate and magnesium bicarbonate). Therefore total alkalinity is the sum of alkalinity due to OH^- , CO_3^{2-} & HCO_3^-.

Alkalinity of water may be due to OH^- , CO_3^{2-}, HCO_3^- or combination of OH^- & HCO_3^- or by CO_3^{2-} & HCO_3^-. But OH^- & HCO_3^- doesn't exist together because they combine to form CO_3^{2-}.
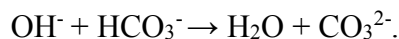
$$OH^- + HCO_3^- \rightarrow H_2O + CO_3^{2-}.$$

The alkalinity of water can be estimated by titrating water with a standard acid using phenolphthalein and methyl orange as indicators.
When titrated with acid, the reactions taking place are as follows:

i) $OH^- + H^+ \rightarrow H_2O$

ii) $CO_3^{2-} + H^+ \rightarrow HCO_3^-$

iii) $HCO_3^- + H^+ \rightarrow H_2O + CO_2$

Titration of the water sample with a standard acid up to phenolphthalein end point marks the completion of reactions(i) and (ii) only. The amount of acid consumed corresponds to hydroxides and one half of carbonates present. Titration of the water sample with a standard acid using methyl orange end point marks the completion of all the three reactions above. Hence, the amount of the total acid consumed corresponds to the total alkalinity and the amount of acid consumed after the phenolphthalein end point corresponds to one half of carbonate and all the bicarbonates. By knowing P & M values alkalinity due hydroxides, carbonates & bicarbonates ions can be calculated from the following table

Values of P&M	Alkalinity due to		
	OH^-	CO_3^{2-}	HCO_3^-
P=0	0	0	M
P= M	M	0	0
P= ½ M	0	2P	0
P<1/2 M	0	2P	M-2P
P> ½ M	2P-M	2(M-P)	0

PROCEDURE:

(i) Preparation of standard hydrochloric acid solution:
The given volume of HCl in 250ml standard flask is diluted up to the mark with distilled water and shaken well for uniform concentration.

(ii) Estimation:
Pipette out 25ml of the water sample into a conical flask. Add 2-3 drops of phenolphthalein indicator and titrate against the standard acid solution till colour changes from pink to colourless. Note down the titre value. Then add 2-3 drops of methyl orange indicator and continue the titration till colour changes from yellow to orange. Note down the final titre value. Repeat the titration for agreeing values.

RESULT:

    i)   **Alkalinity due to OH⁻**   =   **………….. ppm**

    ii) **Alkalinity due to CO_3^{2-}** =**...................ppm**

    iii) **Alkalinity due to HCO_3^-** =**.................. ppm**

    iv) **Total alkalinity of given water sample = ... ppm**

OBSERVATIONS AND CALCULATIONS:
Volume of water sample taken = 25ml

Concentration of the standard hydrochloric acid solution = N_{HCl} =.....................N

Estimation:

Solution in the burette   **:** Standard HCl solution.
Solution in the conical flask **:** 25ml water sample
Indicators & End point   **:** 2-3 drops of phenolphthalein- colour changes from pink to colourless + 2-3 drops of methyl orange – colour change from yellow to orange.

Trial No.		1	2	3
Final burette reading (ml)	**P**			
	M			
Initial burette reading (ml)				
Volume of HCl solution (ml)	**V_1(P)**			
	V_2(M)			

Agreeing Value: V_1 = ...ml

V_2 = ...ml

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I. Phenolpthalein alkalinity (P)

Normality of water, $N_p = \dfrac{V_1 \times N_{HCl}}{25}$

$= \dots\dots\dots N$

Phenolphthalein alkalinity in terms of $CaCO_3$ Equivalents $= N_p \times$ Eq.wt. of $CaCO_3$ g/lt

$= N_p \times 50 \times 1000$ mg/lt

$=$

$P = \dots\dots\dots\dots$ mg/litre or ppm

II. Methyl orange alkalinity or Total alkalinity (M)

Normality of water, $N_M = \dfrac{V_2 \times N_{HCl}}{25}$

$= \dots\dots\dots N$

Methyl orange alkalinity in terms of $CaCO_3$ equivalents $= N_M \times$ Eq. wt. of $CaCO_3$ g/lt

$= N_M \times 50 \times 1000$ mg/litre

$=$

$M = \dots\dots\dots\dots$ mg/litre or ppm

III. On comparing P and M values,

Since   …….,   alkalinity is due to …………………ions.

Therefore alkalinity due to OH^- $=$ ………. $=$ ..………… $=$ ………. ppm

Alkalinity due to CO_3^{-2} $=$ ……… $=$ ……….. $=$ ………....ppm

Alkalinity due to HCO_3^- $=$ ……….. $=$ ..……… $=$ ………. ppm

RESULT:

   i) **Alkalinity due to OH^-** $=$**.................ppm**

  ii) **Alkalinity due to CO_3^{2-}** $=$**.................ppm**

 iii) **Alkalinity due to HCO_3^-** $=$**................. ppm**

 iv) **Total alkalinity of given water sample M** $=$**..............ppm**

VIVA – VOCE QUESTIONS WITH ANSWERS
INSTRUMENTAL ANALYSIS

EXPERIMENT NO.1
ESTIMATION OF FAS BY POTENTIOMETRIC METHOD

1. What is potentiometric titration?
The estimation of substance in solution by measurements of emf is known as potentiometric titration.

2. What are the electrodes used in the determination of FAS potentiometrically?
Calomel electrode – Reference electrode (acts as anode)
Platinum electrode – Indicator electrode (acts as cathode)

3. Give the principle of potentiometric titration?
The principle involved in the potentiometric titration is that the measurements of emf between two electrodes (reference electrode and indicator electrode). While titration is in progress, change in emf is measured. The equivalence point of the reaction is found by sudden change in the graph of $\Delta E / \Delta V$ against volume of titrant.

4. What is an indicator electrode?
The electrode whose potential depends on the concentration of the ion to be determined is called the indicator electrode.

5. What is the determining factor in the oxidation reduction reaction?
The potential at the electrode is due to the redox reaction $Fe^{3+} + e^- \rightleftharpoons Fe^{2+}$. Its magnitude depends on the ratio between the concentration of the two ions $[Fe^{3+} / Fe^{2+}]$. When oxidant is added, the reaction changes and the potential of the redox electrode also changes.

6. What is the reaction that occurs between FAS and $K_2Cr_2O_7$ in potentiometric titration?
$K_2Cr_2O_7$ oxidizes ferrous to ferric in acidic media and itself gets reduced to chromic ion.
$$6\ FeSO_4 + 7\ H_2SO_4 + K_2Cr_2O_7 \longrightarrow K_2SO_4 + Cr_2(SO_4)_3 + 3Fe_2(SO_4)_3 + 7H_2O$$

7. Why sulphuric acid is added to FAS during the emf measurement?
This reaction is carried out in acidic medium. Hence to maintain the acidic conditions, sulphuric acid is added.

8. Why HCl and HNO_3 are not selected to maintain the acid medium instead of dilute H_2SO_4?
Chloride of HCl gets oxidized to chlorine and HNO_3 itself is a good oxidizing agent. Hence these two acids are not used to maintain the acid medium.

9. The colour of the Mohr's salt solution gradually changes into green during titration. Why?
When titration starts, dichromate ions are gradually reduced to chromic ions. Hence the solution appears green due to chromic ions.

$$Cr_2O_7{}^{2-} + 14H^+ + 6e^- \longrightarrow 2Cr^{3+} + 7H_2O$$
$$\text{Green}$$
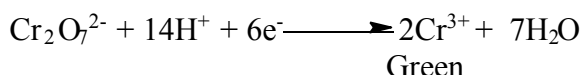
10. What are the advantages of potentiometic titration?
- Turbid, fluorescent or coloured solutions can be estimated.
- Mixture of solutions or very dilute solutions can be determined.
- Without indicator, the solutions can be analysed more accurately, since in these titrations, the end point is indicated with sharp change in emf.

EXPERIMENT NO. 2

CONDUCTOMETRIC ESTIMATION OF ACID MIXTURE

1. State ohm's law?

Ohm's law states that the current, I (ampere) flowing in a conductor is directly proportional to the applied electromotive force, E (volt) and inversely proportional to the resistance, R (ohm) of the conductor.

$$I = E/R$$

Where I is current, E is the potential across the electrodes (emf) and R is the resistance.

2. What is conductance?

The conductance (C) of a solution is the inverse of its resistance(R).

$$C = \frac{I}{R}$$

The conductance of a sample decreases with its length and increases with its cross sectional area.

3. What is the unit of conductance?

The unit of conductance is ohm^{-1} or siemens.

4. What is conductivity?

The reciprocal of resistivity is called conductivity. The SI unit of conductivity is siemens per meter (Sm^{-1}).

5. Mention the different types of conductivities.

They are three types.

        1. Specific conductivity.
        2. Equivalent conductivity
        3. Molar conductivity.

6. What type of conductivity is measured during the conductometric titration?

The specific conductivity is measured.

7. What is specific conductivity?

It is the conductivity of the solution measured between two electrodes of area $1cm^2$ and kept 1cm apart.

8. What is equivalent conductivity?

It is the conductivity of the solution, which contains 1g equivalent of solute, when placed between two electrodes of 1cm apart.

9. What is molar conductivity?

It is the conductance of the solution, which contains 1g molecular weight of a substance when placed between two electrodes at 1cm apart.

10. List out the factors that determine the conductance of solution?

There are three factors determining the conductance of a solution.

   a. Mobility of ions- higher the mobility, higher is the conductance.
   b. Number of ions – more the number of ions, more is the conductance.
   c. Temperature – as the temperature increases, conductivity increases.

11. What is the principle involved in the conductometric titration?

The basic principal of the conductometric titration is the replacement of ions of particular conductance by ions of different during the titration.

$$HCl + NaOH \longrightarrow NaCl + H_2O$$

$$CH_3COOH + NaOH \longrightarrow CH_3COONa + H_2O$$
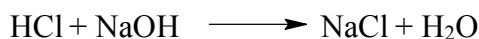
12. What are the advantages of conductometric titration?

   a. The method is accurate for dilute as well as concentrated solutions.
   b. It can also be employed for coloured solutions.
   c. Very weak acids like H_3BO_3, phenol can also be titrated conductometrically.
   d. Mixture of acids (like strong and weak acid) can also be titrated conductometrically.

13. Why the conductance does not touch the X- axis?

The conductance does not reach the X – axis, i.e., it does not become zero because of Na^+ ions present towards the end possess low mobility.

14. Explain the variation of conductance on adding NaOH.

On adding a strong base like NaOH, conductance of acid decreases due to the replacement of highly mobile H^+ ions by less mobile Na^+ ions. The conductance falls till strong acid is neutralized completely. Further, the weak acid starts reacting. When the weak acid is neutralized, the poorly conducting acid is converted into a salt. The salt undergoes complete dissociation, and therefore, the conductance increases gradually. When the neutralization of the weak acid is also completed, further addition of NaOH increases the conductivity sharply in proportional with the amount of NaOH added, as more and more Na^+ and OH^- ions are added into the solution.

15. What is a cell?

A device which produces an emf and delivers an electric current as a result of a chemical reaction is called a cell.

16. What is a cell constant?

Length per unit area of the electrode system is called cell constant.

$$K = \frac{l}{a}$$

EXPERIMENT NO. 3
VISCOSITY COEFFICIENT OF A GIVEN LIQUID

1. What is viscosity?

Viscosity arises due to internal friction between moving layers of molecules. A moving layer exerts a drag or friction on its nearest moving layers backwards. This property of a liquid by which it opposes motion between the layers is called viscosity.

2. What is density of a liquid?

Density = mass / volume

3. What is the SI unit of viscosity coefficient?

The SI unit of viscosity is Nsm^{-2}.

4. What is the law based on the viscous flow of liquids through capillary tubes?

The law based on the viscous flow of liquids through capillary tubes is Poiseuille's law.

$$\eta = \pi Pr^4 t / 8vl \qquad \text{Where,}$$

v = volume of the liquid $\qquad$ r = radius of the tube

l = length of the tube $\qquad$ η = coefficient of viscosity of the liquid

t = time of flow $\qquad$ P = pressure difference between the two ends of the tube

5. What is viscosity coefficient of a liquid?

The viscosity coefficient of a liquid is defined as the tangential force per unit area required to maintain a velocity gradient between two successive moving layers of a given liquid.

6. How viscosity varies with temperature?

Viscosity of a liquid is inversely proportional to temperature.

7. Why acetone is used to dry the viscometer?

Acetone dries the viscometer quickly due to its volatile nature.

8. Why do you require lab temperature to determine viscosity?

The physical constants like density, viscosity of a liquid vary with temperature. Hence lab temperature is required to determine viscosity.

9. How is the viscosity of a liquid related to its mobility?

Viscosity of a liquid is inversely proportional to its mobility.

EXPERIMENT NO. 4
COLORIMETRIC ESTIMATION OF COPPER

1. What is colorimetry?
Chemical analysis through measurements of absorption or transmittance of light radiation in the visible region of the spectrum (400 – 750nm) with respect to a known concentration is termed colorimetry.

2. What is photoelectric colorimeter?
It is an electrical instrument which measures the amount of light absorbed using a photocell.

3. Why filters are used in colorimetric experiment?
The filters are used in colorimetric experiment for selecting desired spectral region.

4. What is wavelength?
The distance between any two successive peaks or troughs of wave is called wavelength. It is represented by λ.

5. What is wave number?
It is the reciprocal of wavelength.

6. What is the frequency?
It is the number of waves passing through a point per second. It is represented by υ.

7. State Beer's law?
When a monochromatic light is passed through a transparent medium, the intensity of transmitted light decreases exponentially as the concentration of the absorbing medium increases arithmetically.

8. State Lambert's law?
When a monochromatic light is passed through a transparent medium, the intensity of transmitted light decreases exponentially as the thickness of the absorbing medium increases arithmetically.

9. State Beer- Lambert's law?
The amount of light absorbed is directly proportional to concentration of the solution.
$$A = \log I_o / I_t = \varepsilon ct$$
Where ε is molar absorption coefficient, c = concentration, t = path length.

10. What is calibration curve?
It is the plot of optical density (absorbance) against concentration of the solution.

11. What is transmittance?
It is the ratio of amount of light transmitted (I_t) to the amount of light incident (I_o).

12. What is absorbance or optical density?
Absorbance or optical density is given by
$$A = \log I_o / I_t = \log 1 / T.$$

13. Can you use different cuvets?
Different cuvets cannot be used as optical density depends on path length of the solution.

14. Why ammonia is added to copper sulphate in the colorimetric determination of copper?
Ammonia is added to get cuprammonium sulphate $[Cu(NH_3)_4]SO_4$ which is a dark blue complex, whose absorbance can be measured.

15. What is blank solution?
A blank solution is identical in all respects to the test solution except the metal ions.

16. Why estimation of copper is done at 620 nm wavelength?
At 620 nm wavelength, the complex shows a maximum absorbance.

17. Why blank solution used in colorimetric estimation?
It is used to nullify the absorbance caused by reagents, distilled water etc (other than test solute).

18. What are the advantages of colorimetric determination?
- More accurate results are obtained at low concentrations when compared with titrimetric or gravimetric analysis.
- The method can be used for estimation of metals like Cu, Fe, Ni etc.

EXPERIMENT NO. 5
pKa VALUE OF A WEAK ACID

1. What is a weak acid and mention any three weak acids?
The acid which undergoes partial dissociation in the solution is called weak acid. It ionizes incompletely when it is dissolved in water.
Eg: Acetic acid, formic acid, oxalic acid etc.

2. What is pKa of weak acid?
$$pKa = -\log_{10} Ka$$
pKa indicates the strength of weak acid, where Ka is the dissociation constant of the weak acid. If Ka value of acid is less, pKa value is higher. Therefore acid is weaker.

3. What does pH of a solution mean?
pH of a solution is the negative logarithm to the base 10 of molar concentration of hydrogen ion.
i.e., $pH = -\log_{10}[H^+]$.

4. Why glass electrode is called ion selective electrode?
Glass electrode responds only to certain specific ions namely H^+ ions. Ignoring all other ions, it develops a potential. Hence glass electrode is called ion selective electrode.

5. Explain, how pH and pKa are related?
According to Henderson, the relation between pH and pKa is

$$pH = pKa + \log_{10} \frac{[\text{salt}]}{[\text{acid}]}$$

At half equivalence point, [salt] = [acid]. Therefore pH = pKa, i.e., pH at half equivalence point gives the pKa value of the weak acid.
.

6. Which electrodes are used in the measurement of pH for the determination of pKa?
Glass electrode (indicator electrode) and calomel electrode (reference electrode) are used in the determination of pKa of a weak acid. In this case, glass electrode act as an anode and calomel electrode acts as cathode.

7. Why pH increases suddenly after the equivalence point?
At equivalence point, entire weak acid is neutralized by base. At this stage, on continuing the titration there will be a sudden increase in pH due to the excess of hydroxyl ions.

8. What are strong acids?
The acids which ionize completely into ions in aqueous solution are called strong acids. Eg: HCl, H_2SO_4, HNO_3.

9. What is Ka?
Ka is dissociation constant of weak acid.

$$Ka_{(CH3COOH)} = \frac{[CH_3COO^-][H^+]}{[CH_3COOH]}$$

10. How pH becomes equal to pKa at half equivalence point?
At half equivalence point, [salt] = [acid]

$$pH = pKa + \log_{10} \frac{[\text{salt}]}{[\text{acid}]}$$

Since log [salt] / [acid] = 0, pH = pKa.

11. How pKa and strength of weak acid are related?
Higher the pKa value, lower will be the strength of weak acid.

12. Explain pH scale?
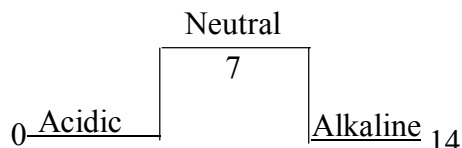
EXPERIMENT NO. 6
FLAME PHOTOMETRY

1. What is the basis for flame photometry?

Emission of characteristic radiation by an element and the correlation of the emission intensity with the concentration of the element form the basis of flame photometry.

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2. What is the characteristic line observed by sodium ion?
The yellow line of sodium at wave length of 598 nm is observed.

3. How flame emission readings vary with concentration?
The flame emission reading is directly proportional to the concentration of the metal ion.

4. What is meant by 1000 ppm of sodium?
1.0g of sodium in 1000 ml of water is called 1000 ppm of sodium.
5. Why distilled water is necessary for flame photometry?
The distilled water is pure and free from metal ions. Hence it is necessary for flame photometry.

VOLUMETRIC ANALYSIS

1. What is volumetric analysis?
The amount of substance present in a solution can be determined by a procedure using the measurement of volumes of solutions, and is referred to as volumetric analysis or titration.

2. Define Normality (N)?
It is the number of gram equivalents of the solute dissolved in one litre of the solution.
Normality = mass per dm^3 / equivalent weight.

3. Define Molarity (M)?
It is the number of gram molecular weight (moles) of the solute dissolved in one litre of the solution.
Molarity = mass per dm^3 / molecular weight.

4. Define Molality (M)?
It is the number of moles of the solute dissolved in one Kg of the solvent.

5. Define basicity of an acid?
The no. of replaceable hydrogen present in one molecule of an acid is called basicity of an acid. Eg: Basicity of HCl, HNO_3, CH_3COOH is one.
　　　　Basicity of H_2SO_4, $H_2C_2O_4$ (oxalic acid) is two.
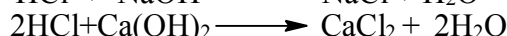　　　　Basicity of H_3PO_4 is three.

6. Define equivalent weight of an acid?
Equivalent weight of an acid = Molecular weight / Basicity of the acid

7. Define acidity of a base?
The no. of molecules of a monobasic acid required to neutralize one molecule of a base is called acidity of a base. Eg: $HCl + NaOH \longrightarrow NaCl + H_2O$
　　　　　　　　　　$2HCl + Ca(OH)_2 \longrightarrow CaCl_2 + 2H_2O$
The acidity of NaOH is 1 and acidity of $Ca(OH)_2$ is 2.
8. Define equivalent weight of a base?
Equivalent weight of a base = Molecular weight / Acidity of a base.

9. What are indicators?

Indicators are the substances which indicate the completion of a chemical reaction by change in colour.

10. Define equivalent weight of an oxidizing agent?

Equivalent weight of an oxidizing agent is that mass of the oxidizing agent which contains 8 parts by weight of available oxygen for oxidation.

11. Define equivalent weight of a reducing agent?

It is the number of parts by mass of the reducing agent which reacts with one equivalent weight of an oxidizing agent.

12. What is a standard solution?

A standard solution is a solution which contains a known mass of the substance dissolved in a known volume of the solvent.

13. What are the types of indicators used in volumetric analysis?

Internal indicators: Phenolphthalein, starch

External indicators: Potassium ferricyanide

Self indicators: Potassium permanganate.

EXPERIMENT NO. 7
TOTAL HARDNESS OF WATER

1. What is hard water?

Hard water is the one which does not give lather easily with soap. It also forms scales in boilers, heaters etc.

2. How is hardness of water caused?

Hardness of water is caused by the presence of certain salts of calcium, magnesium and other heavy metals dissolved in water.

3. How is temporary hardness of water caused?

Temporary hardness of water is caused by the presence of dissolved bicarbonates of calcium, magnesium and other heavy metals.

4. How can you remove temporary hardness?

Temporary hardness of water can be removed by boiling of water, during which bicarbonates decompose to give carbonates.

5. How is permanent hardness of water caused?

Permanent hardness of water is caused by the presence of chlorides and sulphates of calcium and magnesium in water.

6. Which is the unit used to express total hardness of water?

Parts per million (ppm), i.e., parts of $CaCO_3$ equivalent hardness per million parts (10^6) of water.

7. What is EDTA?

EDTA (Ethylene Diamine Tetra Acetic acid) is a complexing agent used in complexometric titrations. Its structure is
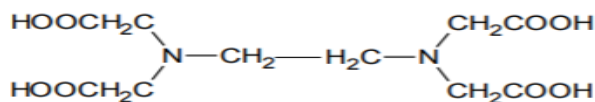
8. Why disodium salt of EDTA is preferred to EDTA?

EDTA is sparingly soluble in water. Hence soluble disodium salt of EDTA is preferred.

9. What is a buffer solution?

It is a solution which has a definite pH and does not change its pH on adding a small quantity of acid or base.

10. Why ammonia - ammonium chloride buffer is used in the determination of total hardness of water?

Ammonia - ammonium chloride buffer is used to maintain pH value of about 10.

11. Which indicator is used in determination of total hardness of water?
   Eriochrome black-T

12. What is the end point in the determination of total hardness of water?
   Wine red to clear blue.

13. Why the indicator shows a color change from wine red to blue?

When the indicator is added, a wine red coloured complex is formed because EBT combines with Ca^{2+} and Mg^{2+} ions to form an unstable complex at a pH 10. When EDTA is added, it reacts with the free Ca^{2+} and Mg^{2+} ions. When all the free ions are exhausted, EDTA reacts with the Ca^{2+} and Mg^{2+} ions that have formed a complex with EBT, releasing the indicator free. Hence a blue colour is obtained.

14. Why is ammonia added while preparing EDTA?

Ammonia is added to increase the rate of dissolution of EDTA.

15. Why are the titrations involving EDTA carried out slowly near the end point?

The titrations involving EDTA are carried out slowly near the end point because the rate of formation of EDTA complex with metal ions is very slow.

16. What is the application of hardness data in environmental engineering practice?
   1. In determining the suitability of water for domestic and industrial uses.
   2. In water softening process.

EXPERIMENT NO. 8
ESTIMATION OF CALCIUM OXIDE IN CEMENT

1. What are the constituents of cement?

Calcium oxide: 58-65%, Silica: 20-25%, Alumina: 4-11%, Ferric oxide: 0-4%, Magnesia: 0-4%, Sodium and potassium oxide: 0-3%.

2. What is Portland cement?
It is the finely ground mixture of calcium aluminates and silicates of varying composition, which hydrate when mixed with water to form a rigid solid structure with good compressive strength.

3. How cement solution is prepared?
A known weight of cement sample is treated with HCl acid. The insoluble residue containing silica is removed by filtration. Iron and aluminium in the filtrate are precipitated as their hydroxides by adding NH_4OH and filtered. The resulting solution containing calcium and magnesium is supplied for estimation.

4. Which indicator is used in the determination of CaO in the cement solution? What is the end point?
Patton and Reeder's indicator is used.
End point: wine red to clear blue.

5. Why Eriochrome Black - T can not be used in the determination of CaO in the cement solution?
Eriochrome Black - T can not be used in the determination of CaO, because it forms a very weak complex with calcium ions at a pH range of 12-14.

6. Why glycerol and diethyl amine are added to the titration flask?
Glycerol is added to get sharp end point.
Diethyl amine is added to maintain the pH of about 12-14.

7. Write the structural formula of Glycerol?
Glycerol is a trihydric alcohol and its structure is

$$\begin{array}{c} CH_2OH \\ | \\ CHOH \\ | \\ CH_2OH \end{array}$$
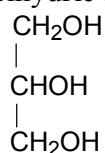
8. Why NaOH is added?
   To prevent the interference of Magnesium ions.

EXPERIMENT NO. 9
ESTIMATION OF COPPER IN BRASS

1. What are the constituents of brass?
Brass is an alloy of copper and zinc. The composition is Cu: 60-70%, Zn: 30-40%, and a small amount of tin, lead and iron.

2. How is brass solution prepared?
 Brass solution is prepared by dissolving brass foil in minimum amount of nitric acid.

$$Cu + 4HNO_3 \longrightarrow Cu(NO_3)_2 + 2H_2O + 2NO_2\uparrow$$

3. What is the purpose of adding urea to the brass solution?
Urea is added to destroy oxides of nitrogen otherwise oxides of nitrogen oxidize KI to I_2.

4. Why is the ammonium hydroxide added to brass solution in the determination copper?
Ammonium hydroxide is added to neutralize the mineral acid. Otherwise the mineral acid present in the brass solution will oxidize KI to iodine.

5. Why is the acetic acid added to brass solution in the determination copper?
Acetic acid is added to neutralize the excess of ammonium hydroxide and make the solution slightly acidic.

6. Why is KI added to the brass solution in the determination of copper?
KI is added to reduce cupric ion to cuprous iodide.
$$2Cu^{2+} + 4KI \longrightarrow Cu_2I_2 + 4K + I_2$$
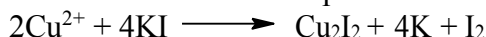
7. Why is the blue solution of brass turns brown after adding KI?
Brass solution turns brown after adding KI because iodine starts liberating from conical flask.

8. Why is starch indicator added towards the end point in iodometric titration?
Starch indicator is added near the end point when the concentration of I_2 is low. This is because,
- At high concentration of I_2, starch may form an insoluble starch – iodine complex.
- Also, the iodine gets adsorbed on colloidal starch particles in the solution.

9. What is the white precipitate left at the end point in the estimation of copper in brass?
White precipitate left at the end point is cuprous iodide (Cu_2I_2).

10. How is liberated iodine estimated?
The amount of liberated iodine is estimated by titrating it against standard sodium thiosulphate solution using starch indicator.

11. What is the reaction that occurs between iodine and sodium thiosulphate?
Iodine reacts with sodium thiosulphate to form sodium iodide and sodium tetrathionate, which are colorless products.
$$I_2 + 2Na_2S_2O_3 \longrightarrow 2NaI + Na_2S_4O_6$$
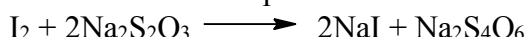
EXPERIMENT NO. 10
DETERMINATION OF COD OF WASTE WATER

1. What is chemical oxygen demand (COD)?
It is defined as the no of milligrams of oxygen required for oxidation of chemically oxidisable impurities present in 1 liter of waste water using a strong oxidizing agent like $K_2Cr_2O_7$.

2. What is biological oxygen demand (BOD)?
It is defined as the amount of oxygen required for oxidation of the organic matter present in 1 liter of waste water under aerobic conditions at 20°C for a period of 5 days.

3. What is the role of silver sulphate in the determination of COD?
Silver sulphate catalyses oxidation of straight chain organic compounds, aromatics and pyridine.

4. What is the role of mercuric sulphate in the determination of COD?
Mercuric sulphate avoids the interference of Cl^- ions by forming mercuric chloride and prevents the precipitation of silver chloride.

5. What is the indicator used in the determination of COD?
Ferroin. The chemical name is [1,10-phenanthroline Fe(II)] complex.

6. What is the color change at the endpoint in the determination of COD?
Color change is blue green to reddish brown.

7. Why is sulphuric acid added during the preparation of standard FAS solution?
Sulphuric acid is added to prevent the hydrolysis of ferrous sulphate to ferrous hydroxide.

8. What are the products formed after COD analysis?
During COD analysis, organic matter is completely oxidized to CO_2 and H_2O by acidified $K_2Cr_2O_7$.

9. What is meant by blank titration?
A blank titration is identical in all respects to the sample titration except the waste water. In place of waste water, 25ml of distilled water is taken.

10. What are the applications of COD test?
1. The COD test is extensively used in the analysis of industrial wastes.
2. The test is used to determine the level of polluted water.
3. Experimental results are obtained within a relatively short time.

EXPERIMENT No. 11
ESTIMATION OF IRON IN HAEMATITE

1. What is the main constituent of haematite ore?
The main constituent of haematite ore is Fe_2O_3 and silica (SiO_2).

2. What is oxidation state of iron in haematite ore?
The oxidation state of iron in haematite ore is Fe^{3+}.

3. What is the role of stannous chloride in the determination of iron in haematite?
Stannous chloride acts as a reducing agent to reduce Fe^{3+} to Fe^{2+}.
$$2FeCl_3 + SnCl_2 \longrightarrow 2FeCl_2 + SnCl_4$$
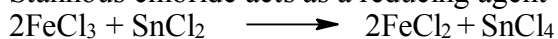
4. Why is mercuric chloride added in the determination of iron in haematite?
Mercuric chloride is added to remove excess of stannous chloride. It reacts with excess of stannous chloride to form a silky white precipitate of mercurous chloride.
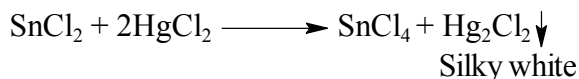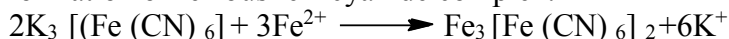
$$SnCl_2 + 2HgCl_2 \longrightarrow SnCl_4 + Hg_2Cl_2 \downarrow$$
<div align="center">Silky white</div>

5. What is the indicator used in the determination of iron in haematite ore solution?
Potassium ferricyanide (K_3 [(Fe (CN) $_6$]) is used as an external indicator.

6. What is the color of $K_3Fe(CN)_6$ with ferrous ion?
Potassium ferricyanide produces an intense deep blue color with ferrous ion due to the formation of ferrous ferricyanide complex.
$$2K_3 [(Fe (CN)_6] + 3Fe^{2+} \longrightarrow Fe_3 [Fe (CN)_6]_2 + 6K^+$$
7. Why the color of the drop remains same at the end point in the determination of iron in haematite ore solution?
Ferrous ion only forms intense deep blue color with Potassium ferricyanide. At the end point ferrous ions are completely oxidized to ferric ion by $K_2Cr_2O_7$. Ferric ion does not give any color; hence indicator drop remains same at the end point.

8. Why potassium ferricyanide cannot be used as an internal indicator in the determination of iron in haematite ore solution?
Potassium ferricyanide combines irreversibly with ferrous ion to form a deep blue ferrous ferricyanide complex. Theses ferrous ions involved in the complex formation are not available for reaction with potassium dichromate. Also, the end point cannot be detected as there is no color change.

9. What is the reaction that occurs during the titration?
Acidified $K_2Cr_2O_7$ oxidizes ferrous ion present in the haematite ore solution to ferric ion and itself gets reduced to chromic ion.
$$6Fe^{2+} + Cr_2O_7^{2-} + 14H \longrightarrow 6Fe^{3+} + 2Cr^{3+} + 7H_2O.$$

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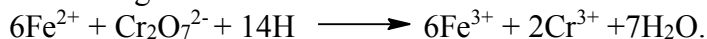
**EXPERIMENT NO. 12**
ESTIMATION OF CHLORINE IN BLEACHING POWDER

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1. What are the constituents of bleaching powder?
 Bleaching powder is a mixture of calcium hypochlorite $Ca(OCl)_2$, calcium chloride $CaCl_2$,and some amount of $Ca(OH)_2$.

2. Which is the active part of bleaching powder?
   Hypochlorite is the active part of bleaching powder from which chlorine is liberated.

3. Write the relation between hypochlorite and dilute acid?

   $$Ca (OCl)_2 + 2CH_3COOH \rightarrow Ca (CH_3COO)_2 + H_2O + Cl_2$$
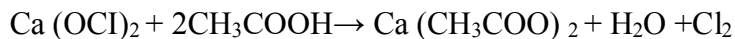
4. Why is the acetic acid added?
   To liberate chlorine from hypochlorite.

5. Why bleaching powder solution turns brown after adding KI?
Bleaching powder solution turns brown after adding KI because iodine starts liberating from the solution.

6. Why is starch indicator added towards the end point in iodometric titration?
Starch indicator is added near the end point when the concentration of I_2 is low. This is because,
- At high concentration of I_2, starch may form an insoluble starch – iodine complex.
- Also, the iodine gets adsorbed on colloidal starch particles in the solution.

7. How is liberated iodine estimated?
The amount of liberated iodine is estimated by titrating it against standard sodium thiosulphate solution using starch indicator.

8. What is the reaction that occurs between iodine and sodium thiosulphate?
Iodine reacts with sodium thiosulphate to form sodium iodide and sodium tetrathionate, which are colorless products.
$$I_2 + 2Na_2S_2O_3 \longrightarrow 2NaI + Na_2S_4O_6$$
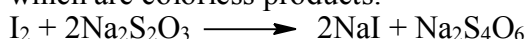
9. What is the percentage of chlorine in bleaching powder?
The chlorine content of bleaching powder varies from 35 – 40%.

10. What are applications of bleaching powder?
Along with bleaching action it has got strong germicidal and disinfectant properties also. It finds application as a disinfectant for drinking water or swimming pool water. Industrially, the bleaching powder finds major use in chemical, paper, textile and oil industries.

11. Bleaching, oxidizing or disinfecting potential of action of bleaching powder depends on what?
The bleaching, oxidizing or disinfecting potential of a sample of bleaching powder depends on the percentage of chlorine liberated on action of acid.

11. Why estimation of chlorine in bleaching powder is needed?
Due to its hygroscopic nature, bleaching powder absorbs moisture from atmosphere and evolves chlorine.

$$OCl^- + Cl^- + Ca^{+2} + H_2O \rightarrow Ca(OH)_2 + Cl_2$$
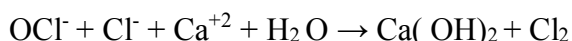
Due to this deterioration, a sample of bleaching powder may always contain lesser amount of chlorine than expected and therefore a sample of bleaching powder needs to be analysed for its effective or available chlorine

BRIEF PROCEDURES
1. ESTIMATION OF FAS BY POTENTIOMETRIC METHOD

Make up the Mohr's salt (FAS) solution kept in a 250ml standard flask to the mark with distilled water and shake well. Pipette out 25ml of this solution into a 100ml beaker. Add 1 test tube of dil. H_2SO_4. Immerse Calomel electrode and platinum electrodes and connect to a potentiometer. Add $K_2Cr_2O_7$ from the burette in increments of 0.5ml and measure the potential. Initially the emf increases slowly and at the equivalence point there will be a sudden jump in the emf. Record at least 4-6 readings after equivalence point.
Plot a graph of $\Delta E/\Delta V$ against volume of $K_2Cr_2O_7$ and measure the equivalence point.
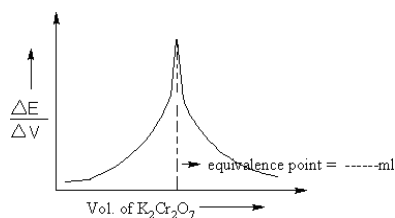
2.CONDUCTOMETRIC ESTIMATION OF ACID MIXTURE
Pipette out 50ml of the given acid mixture into a 100ml beaker. Immerse the conductivity cell in the acid mixture and connect it to the conductometer. Fill a burette with the given strong base, NaOH. Add 0.5ml of the base into the beaker at a time, stir well and note the conductance. The titration should be continued till the decreasing trend of conductance changes to a gradual increasing trend, and then to a sharp increasing trend, and at least 5-6 readings should be taken after that. Plot a graph of volume of base against conductance. The points of intersection of the curves give the equivalent points.
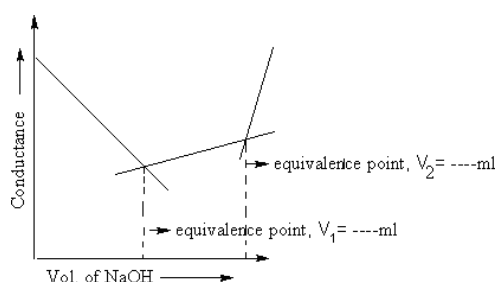
3.VISCOSITY COEFFICIENT OF A GIVEN LIQUID

Take a clean and dry viscometer, insert in water bath and fix vertically to a stand. Pipette out 10ml of the given organic liquid into the wider limb. Using a rubber tube, suck the liquid little above the upper mark of the narrow limb. Allow to flow the liquid freely through the capillary. When the level of the liquid just crosses the upper mark, start a stop clock and stop it when the liquid crosses the lower mark. Measure the time of flow of the liquid.

Repeat the same procedure with distilled water after drying it using acetone.

The coefficient of viscosity of liquid is given by Poiseuille's formula.

$$\eta = \frac{\pi\, P\, r^4\, t}{8\, V l}$$

Where,

V = volume of the liquid   r = radius of the tube

l = length of the tube   η = coefficient of viscosity of the liquid

t = time of flow   P = pressure difference between the two ends of the tube

4.COLORIMETRIC ESTIMATION OF COPPER

Transfer 5, 10, 15, 20 and 25ml of the given $CuSO_4$ solution into separate 50ml standard flasks. Add 5ml of 1:1 NH_3 to each of the flasks. Make up all these solutions to the mark with distilled water and shake well. Prepare a blank solution by diluting 5ml of 1:1 ammonia in another 50ml standard flask. Add 5ml of ammonia to the test solution and dilute up to the mark and mix well.

Set the colorimeter to zero absorbance at 620nm for blank solution. Measure the absorbance of each of the standard solutions and also of the test solution. Plot a graph of absorbance versus volume of copper sulphate and determine the amount of copper in the test solution.
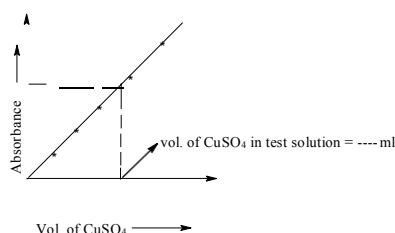
5. pKa VALUE OF A WEAK ACID

Make up the weak acid kept in a 250ml standard flask to the mark with distilled water and shake well. Pipette out 50ml of this solution into a 100ml beaker. Immerse the glass electrode + calomel electrode assembly into it and connect to a pH meter. Add NaOH from the burette in increments of 0.5ml and measure the pH. Initially pH increases slowly and at the equivalence point there will be a sudden jump. Record at least 4-6 readings after equivalence point.

Plot a graph of Δ pH / ΔV against volume of NaOH and determine the equivalence point. Plot another graph of pH against volume of NaOH and determine the half equivalence point.

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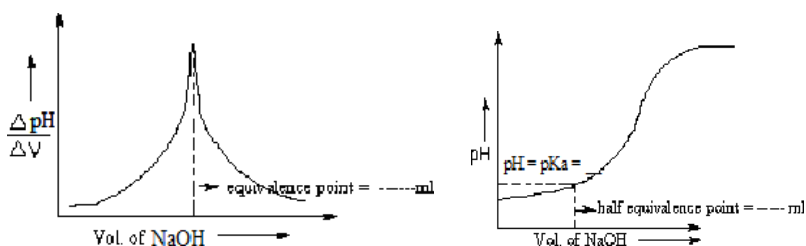
6. FLAME PHOTOMETRY

Transfer exactly 3, 6, 9, 12, 15, 18 and 21 ml of stock solution using a burette into seven separate 50ml volumetric standard flasks and dilute all the above solutions and the test solution with distilled water up to the mark and shake well for uniform concentration. Take the first standard solution in a small beaker and aspirate into the flame of the flame photometer. Measure the flame emission intensity of the standard by the flame photometer using sodium filter (598 nm). Repeat the same for all the standard solutions with distilled water being sprayed to the flame in between the trials. Plot the calibration curve by taking emission response along the Y-axis and volume of the solution along the X-axis. Then aspirate the given test sample into the flame and note down the value of emission response. From the calibration curve compute the volume of Na in the test sample.

Repeat the same procedure for potassium.
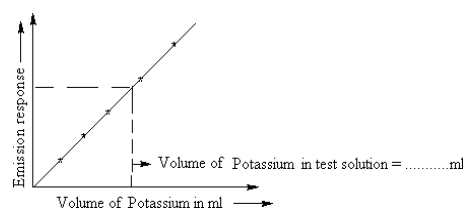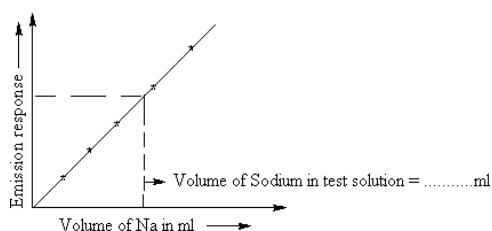
7. ESTIMATION OF TOTAL HARDNESS OF WATER

Weigh accurately about 0.8g of EDTA and transfer into a 250ml standard flask. Dissolve in distilled water. If necessary, add 2ml of NH_3. Make up the solution to the mark and shake well for uniform concentration. Take it in a 50ml burette.

Pipette out 25ml of given hard water into a clean conical flask. Add 2ml of NH_4OH – NH_4Cl buffer and 2 drops of EBT indicator. Titrate the resulting wine red colored solution against EDTA until clear blue solution is obtained. No tinge of reddish blue should remain at the end point. Repeat the titration to get agreeing values.

8. ESTIMATION OF CALCIUM OXIDE IN CEMENT

Weigh accurately about 0.8g of EDTA and transfer in to a 250ml standard flask. Dissolve in distilled water. If necessary, add 2ml of NH_3. Make up the solution to the mark and shake well for uniform concentration. Take it in a 50ml burette.

Pipette out 25ml of given cement solution into a clean conical flask. Add 5ml of 1:1 of glycerol followed by 5ml of diethyl amine buffer. Add 1 test tube of 4 N sodium hydroxide and 2 test tubes full of ion exchange water. Add 2 drops Patton and Reeder's indicator. Titrate the resulting solution in the conical flask against EDTA solution taken in the burette until wine red colour changes to clear blue. Repeat the titration to get agreeing values.

9. ESTIMATION OF COPPER IN BRASS

Weigh the given brass foil and transfer into a 250ml beaker, add ¼ test tube of conc. nitric acid. Dissolve the brass and add 1 test tube of distilled water. Add about 1g of urea and boil for 1-2 minutes. Cool and transfer the contents to a 250ml standard flask using funnel, make up to the mark and shake well for uniform concentration.

Pipette out 25ml of brass solution into a clean conical flask. Add NH_4OH solution in drops until bluish white precipitate persists. Dissolve the precipitate by adding acetic acid in drops followed by the 5ml of acetic acid in excess. Add 10ml of 10% KI to the conical flask and titrate against standard $Na_2S_2O_3$ solution taken in the burette until the solution changes to pale yellow. Now add 2ml of starch indicator. The solution turns dark blue. Continue the titration until a milky white solution remains. Repeat the titration to get agreeing values.

10. DETERMINATION OF COD OF WASTE WATER

Weigh accurately the given Mohr's salt and transfer into a 250ml standard flask. Add 1 test tube of dil. H_2SO_4, dissolve, make up to the mark with distilled water and shake well for uniform concentration. Take this solution in a burette.

Pipette out 25ml of waste water into a 250ml conical flask. Add 1g of $HgSO_4$ followed by 2 test tubes of silver sulphate - sulphuric acid mixture. Also pipette out 25ml of given potassium dichromate solution into it. Add a porcelain piece, attach the flask with a reflux condenser and reflux the mixture for half an hour. Cool, wash the inner side of the condenser with distilled water into the same flask. Now add 1 test tube of 1:1 sulphuric acid and 1-2 drops of ferroin indicator. Titrate the mixture with standard Mohr's salt solution till the colour changes from bluish green to dark red. Repeat the titration to get agreeing values.

Blank Titration: Repeat the above procedure by taking 25ml of distilled water in place of waste water.

11. ESTIMATION OF IRON IN HAEMATITE

Weigh the given $K_2Cr_2O_7$ crystals and transfer it into a 250ml standard flask. Dissolve the crystals in distilled water, make up to the mark and shake well for uniform concentration.

Pipette out 25ml of given haematite ore solution into a clean 250ml conical flask. Add 5ml of conc. HCl and heat to boiling. To the hot solution, add $SnCl_2$ solution drop by drop with swirling until the yellow colour disappears. Add 2 drops in excess. Cool the solution and add 5ml of $HgCl_2$ and shake well. A silky white precipitate should be obtained. Titrate the resulting solution against $K_2Cr_2O_7$ solution taken in a burette using $K_3[(Fe(CN)_6)]$ as the external indicator taken on a wax paper till the indicator drop doesn't turn blue. Repeat the titration for agreeing values.

12. ESTIMATION OF CHLORINE IN BLEACHING POWDER

Weigh given sample bleaching powder and transfer into a clean 250 ml standard flask. Dissolve using distilled water. Makeup and shake well for uniform concentration.

Pipette out 25ml of the turbid liquid into a conical flask. Add 1 test tube full of water, 10 ml of 10% KI solution and 5ml of glacial acetic acid. Titrate the liberated iodine against standard sodium thiosulfate solution taken in burette using starch as indicator near the end point till blue colour disappears. Repeat the titration to get concordant volumes.