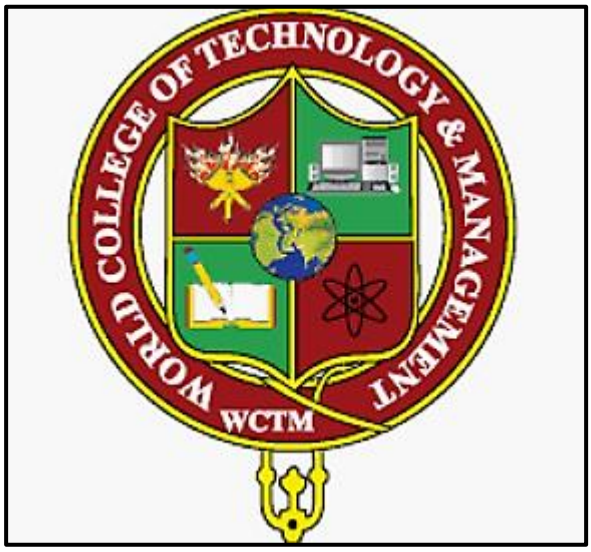
World College of Technology & Management

Chemistry Lab Manual



Faculty of Engineering and Technology

WCTM, Gurugram

List of Experiments

1. To determination the surface tension of a given liquid at room temperature using stalagmometer by drop number method.
2. To determine the viscosity of a given unknown liquid with respect to water at room temperature, by Ostwald's Viscometer.
3. To calculate the Rf value of given sample using Thin layer chromatography / Paper chromatography.
4. Removal of Ca^{2+} and Mg^{2+} hardness from given water sample using ion exchange column.
5. Determination of chloride content in given water sample.
6. Determination of strength of HCl solution by titrating it against NaOH solution conductometrically.
7. To prepare the of urea formaldehyde and phenol formaldehyde resin.
8. To Prepare iodoform.
9. Calculate the saponification value / acid value of given oil sample.
10. To determine the total hardness of given water sample by EDTA method.
11. Study the adsorption phenomena using acetic acid and charcoal.
12. To determine the EMF of a cell.
13. To determine the rate constant of a reaction.
14. Determination of the partition coefficient of a substance between two immiscible liquids.
15. Lattice Structure & Packing of Sphere.
16. Chemical analysis of two anions and two cations in given sample of salt.

DO'S

The Chemistry laboratory must be a safe place in which to work and learn about Chemistry.

1. Use protective clothing all the time (e.g. lab coat and safety glasses).

2. Be familiar with your lab assignment before you come to lab. Follow all written and verbal instructions carefully. Observe the safety alerts in the laboratory directions. If you do not understand a direction or part of a procedure, ask the teacher before proceeding.

3. Wash acid, base, or any chemical spill off of yourself immediately with large amounts of water. Notify your teacher of the spill.

4. Clean up spills immediately. If you spill a very reactive substance such as an acid or base, notify the people in the area and then obtain assistance from your teacher. Acid spills should be neutralized with baking soda, base spills with vinegar before cleaning them up.

5. If chemical substances get in your eye, wash the eye out for 15 minutes. Hold your eye open with your fingers while washing it out.

6. Place the reagents in a systemic manner.

7. If you burn yourself on a hot object, immediately hold the burned area under cold water for 15 minutes. Inform your teacher.

8. Observe good housekeeping practices. Work areas should be kept clean and tidy at all times. Only lab notebooks or lab handouts should be out on the table.

DON'T

1. Work in the laboratory without an instructor present. Work only with your lab partner(s). Do not venture to other lab stations for any reason.

2. Wear bulky or dangling clothing.

3. Eat or drink in the laboratory. Don't chew on the end of a pen which was lying on the lab bench.

4. Use Mobile Phones.

5. Directly touch any chemical with your hands. Never taste materials in the laboratory.

6. Waste the reagents.

7. When entering the lab/classroom, do not touch any equipment, chemicals, or other materials without being instructed to do so. Perform only those experiments authorized by the instructor.

8. When weighing never place chemicals directly on the balance pan. Never weigh a hot object.

9. Smell anything in the laboratory unless your teacher tells you it is safe. Do not smell a substance by putting your nose directly over the container and inhaling. Instead, waft the vapors toward your nose by gently fanning the vapors toward yourself.

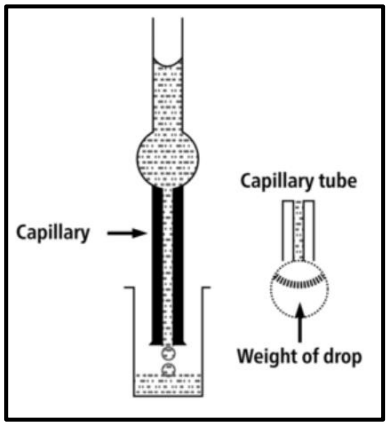
10. Absolutely no running, practical jokes, or horseplay is allowed in the laboratory.

11. Allow the reagent bottles to accumulate on the bench.

Experiment-I

Aim: To determine the surface tension of a given liquid at room temperature using stalagmometer by drop number method.

Requirements: Stalagmometer, specific gravity bottle, a small rubber tube with a screw pinch cork, distilled water, experimental liquid.



Theory: Surface tension is the tension of the surface film of a liquid caused by the attraction of the particles in the surface layer by the bulk of the liquid, which tends to minimize surface area. The surface tension of water is 72 dynes/cm at 25°C. It would take a force of 72 dynes to break a surface film of water 1 cm long. The surface tension of water decreases significantly with temperature. The surface tension arises from the polar nature of the water molecule.

At liquid–air interfaces, surface tension results from the greater attraction of liquid molecules to each other (due to cohesion) than to the molecules in the air (due to adhesion). Surface tension, represented by the symbol γ (alternatively σ or T), is measured in force per unit length. Its SI unit is Newton (N) per meter but the CGS unit of dyne per centimeter (cm). Water striders use the high surface tension of water and long, hydrophobic legs to help them stay above water.

In the drop number method, the number of drops formed by equal volumes of two liquid is counted. One of the liquids is water its surface tension and density are known. Then the surface tension of the given liquid can be calculated.

Procedure:

1. Clean the stalagmometer with water and dry it in an oven.

2. Attach a small piece of rubber tube having a screw pinch cock at the upper end of the stalagmometer.

3. Immerse the lower end of the stalagmometer in distilled water and suck the water 1-2 cm above mark A. Adjust the pinch cork, so that 10-15 drops fall per minute.

4. Clamp the stalagmometer allow the water drops to fall and start counting the number of drops when the meniscus crosses the upper mark A and stop counting when the meniscus passes mark B.

5. Repeat the exercise to take three to four readings. Rinse the stalagmometer with alcohol and dry it.

6. Suck the given liquid in the stalagmometer and count the drops, as in case of water.

7. Now, take a clean dry specific gravity bottle, weigh it empty as well as with water and given liquid.

Observation Table:

Liquid Samples	**No. of Drops**			**Average No. of Drops**	**Density (g/mL)**	**Surface Tension (γ) dynes/cm**
	1	**2**	**3**			
Water						
Experimental Liquid						

Calculations:

Determination of the Density of the given Experimental Liquid:

Weight of empty specific gravity bottle = W_1 g.

Weight of specific gravity bottle + water = W_2 g

Weight of empty specific gravity bottle + Sample Liquid = W_3 g

Weight of Water= (W_2-W_1) g

Weight of Sample Liquid= (W_3- W_1) g

Density of Water at room temperature (ρ_w) = 0.997 g/mL

Density of the given Sample Liquid (ρ_L) = Weight of Sample Liquid/ Weight of Equal Volume of water * Density of water

= (W_3- W_1)/ (W_2-W_1) * ρ_w

Determination of the Surface Tension of the given Sample Liquid:

Surface Tension of water at room temperature (γ_w) = 72.8 dyne/ cm

Surface Tension of Sample Liquid (γ_L) = $\rho_L n_1 / \rho_w n_2 * \gamma_w$

Where: n_1 : number of drops of water from point A to B

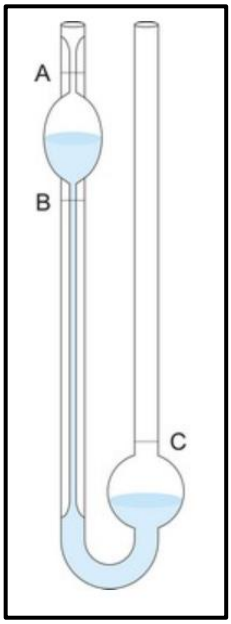
n_2 : number of drops of sample liquid from point A to B

Result: The Surface Tension of liquid is ……dynes/cm.

Experiment-II

Aim: To determine the viscosity of a given unknown liquid with respect to water at room temperature, by Ostwald's Viscometer.

Requirements: Ostwald's Viscometer, specific gravity bottle, a small rubber tube with a screw pinch cork, distilled water, experimental liquid, digital timer.



Theory: Viscosity, resistance of a fluid (liquid or gas) to a change in shape, or movement of neighboring portions relative to one another. Viscosity denotes opposition to flow. The reciprocal of the viscosity is called the fluidity. All liquids appear resistance to flow change from liquid to another, the water flow faster than glycerin, subsequently, the viscosity of water is less than glycerin at same temperature.

The factors effect on the viscosity:

1. **Effect of Temperature:** the temperature of the liquid fluid increases its viscosity decreases. In gases its opposite, the viscosity of the gases fluids increases as the temperature of the gas increases.

2. **Molecular weight:** the molecular weight of the liquid increases its viscosity increases.

3. **Pressure:** when increase the pressure on liquids, the viscosity increases because increase the attraction force between the molecules of liquid.

The SI Unit of Viscosity is pascal-second (Pa.s). The CGS Unit is expressed as Poise (P). It is more commonly expressed as Centipoise (cP) {1 cP = 0.001 Pa.s}.

Procedure:

1. Clean the Ostwald's viscometer with water and acetone, dry it in a preheated oven.

2. Attach a small piece of rubber tube having a screw pinch cock at the upper end of the Ostwald's viscometer.

3. Now fill the Ostwald's viscometer with water through pipette up to half of the bulb. Suck the water, 1-2 cm above mark A. Adjust the pinch cork, so that 1-2 drops fall per second.

4. Clamp the Ostwald's viscometer on a stand and allow the water drops to fall and start the timer when the meniscus crosses the upper mark A and stop it when the meniscus passes mark B. Note down the time taken from point A to B, in seconds.

5. Repeat the exercise to take three to four readings. Rinse the Ostwald's viscometer with alcohol and dry it.

6. Further, fill the given liquid in the Ostwald's viscometer with the help of pipette and note down the time, in seconds, as in case of water.

7. Now, take a clean dry specific gravity bottle, weigh it empty as well as with water and given liquid.

Observation Table:

Liquid Samples	**Time (sec.)**			**Mean Time (sec.)**	**Density (g/mL)**	**Viscosity (η_L) (cP)**
	1	**2**	**3**			
Water						
Experimental Sample						

Calculations:

Determination of the Density of the given Experimental Liquid:

Weight of empty specific gravity bottle = W_1 g.

Weight of specific gravity bottle + water = W_2 g

Weight of empty specific gravity bottle + Sample Liquid = W_3 g

Weight of Water= (W_2-W_1) g

Weight of Sample Liquid= $(W_3 - W_1)$ g

Density of Water at room temperature (ρ_w) = 0.997 g/mL

Density of the given Sample Liquid (ρ_L) = Weight of Sample Liquid/ Weight of Equal Volume of water * Density of water

$= (W_3 - W_1)/ (W_2 - W_1) * \rho_w$

Determination of the Viscosity of the given Sample Liquid:

Viscosity of water at room temperature (η_w) = 0.996 cP

Viscosity of Sample Liquid (η_L) = $\rho_L t_1/ \rho_w t_2 * \eta_w$

Where: t_1 : mean time of flow of sample liquid from point A to B.

t_2 : mean time of flow of water from point A to B.

Result: The Viscosity of liquid is ……cP.

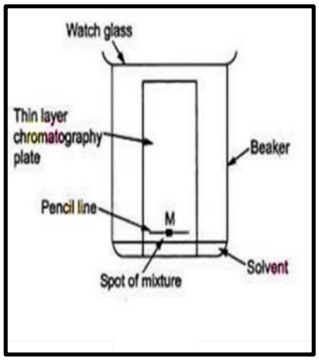
Experiment-III

Aim: To calculate the Rf value of given sample using Thin layer chromatography / Paper chromatography.

Requirement: TLC Plates, Beaker, Solvent (Ethanol/Ethyl Acetate), Capillary tube, Indigo dye, watch glass, scale, pencil.

Theory:

Thin Layer Chromatography (TLC) is a solid-liquid technique in which the two phases are a solid (stationary phase) and a liquid (moving phase). Solids most commonly used in chromatography are silica gel ($SiO_2.xH_2O$) and alumina ($Al_2O_3.xH_2O$). Both of these adsorbents are polar in nature. However, silica is acidic. Alumina is available in neutral, basic, or acidic forms. Thin Layer Chromatography (TLC) is a sensitive, fast, simple and inexpensive analytical technique. It is a micro technique; as little as 10^{-9} g of material can be detected, although the sample size is from 1 to $100x10^{-6}$ g.



TLC involves spotting the sample to be analyzed near one end of a sheet of glass or plastic that is coated with a thin layer of an adsorbent. The sheet, which can be the size of a microscope slide, is placed on end in a covered jar containing a shallow layer of solvent. As the solvent rises by capillary action up through the adsorbent, differential partitioning occurs between the components of the mixture dissolved in the solvent the stationary adsorbent phase.

The more strongly a given component of a mixture is adsorbed onto the stationary phase, the less time it will spend in the mobile phase and the more slowly it will migrate up the plate.

RF (retention factor) value is the distance travelled by a given component divided by the distance travelled by the solvent front. The larger the Rf of a compound, the larger the distance it travels on the TLC plate. When comparing two different compounds run under identical chromatography conditions, the compound with the larger Rf is less polar because it interacts less strongly with the polar adsorbent on the TLC plate.

Procedure:

1. Obtain one TLC plates. Draw a light pencil line about 1 cm from the end of each chromatographic plate.

2. First Spot will be known standard sample (reference) and the other spot with unknown commercial sample.

3. Use separate capillary tube for each standard and unknown solution.

4. Make each spot as small as possible, preferably no more than 2-3 mm in diameter.

5. Examine the plate under the ordinary light to see that enough of each compound has been applied; if not, add more.

6. The standards and commercial sample will be dissolved in 50/50 Ethanol/Ethyl Acetate solution.

7. Prepare a developing chamber as indicated in the picture using a large beaker as the chamber, a half-piece of filter paper inside, and foil or plastic wrap to cover.

8. Pour the eluting solvent, a 99/1 mixture of Ethyl Acetate/Glacial Acetic Acid, into the beaker to a depth of approximately 1 cm.

9. Place the prepared TLC plates in the developing chamber.

10. After the solvent has risen to near the top of the plate (about 1 cm from the top), remove the plate and mark the solvent front with a pencil.

11. Keep the plates in the hood until the majority of the eluting solvent has evaporated from the plates.

12. Examine the plate under ordinary light to see the components.

13. Outline the spots with a pencil and note anything distinctive about any of the compounds.

14. Remove the plates when a definite change in appearance takes place on your plates.

15. Measure the distance travelled by the two spots from base line

Observation Table:

	Distance Travelled from Baseline (cm)
Standard Liquid	
Sample Liquid	

Calculations:

RF = Distance Travelled by the sample spot/ Distance travelled by the solvent front

Result: RF value of the sample…….

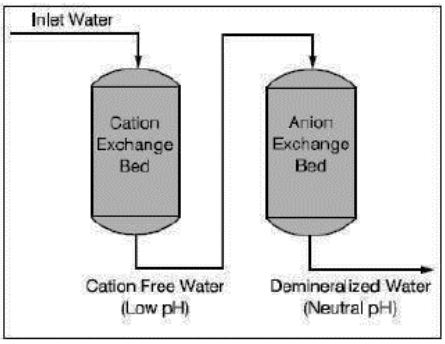
Experiment-IV

Aim: Removal of Ca^{2+} and Mg^{2+} hardness from given water sample using ion exchange column.

Requirement: Cation-Anion Exchange machine, Tap with inlet/ outlet pipe, pH meter, conductivity meter, beakers.

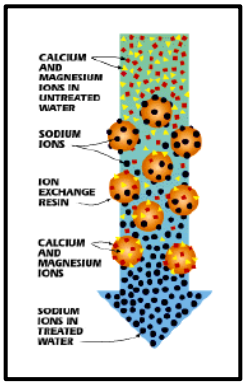
Theory:

Ion exchange softening, also known as zeolite softening, passes water through a filter containing resin granules. In the filter, known as a softener, calcium and magnesium in the water are exchanged for sodium from the resin granules. The resulting water has a hardness of 0 mg/L and must be mixed with hard water to prevent softness problems in the distributed water.



Ion exchange softening does not require the flash mixer, flocculation basin, and sedimentation basin required for lime-soda ash softening. In addition, the process does not require as much operator time. Ion exchange softening is effective at removing both carbonate and noncarbonate hardness and is often used for waters high in noncarbonate hardness and with a total hardness less than 350 mg/L.

Cation exchange materials are classified as either weak acid or strong acid depending on the type of exchange group. Strong acid cation exchanger contains $–SO_3$- functional, weak acid cation exchanger contain –COO- functional groups.



Anion exchange materials are classified as either weak base or strong base depending on the type of exchange group. Weak base resins act as acid adsorber, efficiently removing strong acids such as sulfuric and hydrochloric

Procedure:

1. Start the pump to feed the columns with filtered tap-water.
2. Measure continuously the effluent pH and specific conductivity after the cation exchange and after the anion exchange column separately.
3. Register the measured data every 5 minutes together with the volume of the demineralized water in a table.
4. Continue the feed until breakthrough. Draw the volume of the demineralized water-pH and volume of the demineralized water-Specific conductivity curves and estimate the breakthrough volume.

Observation Table:

Time(in min)	Volume (in ml)	Effluent of cation exchanger		Effluent of anion exchanger	
		spec. conductivity (in (µS/cm)	pH	spec. conductivity (in (µS/cm)	pH

Result: Breakthrough volume of the sample water……..mL

Experiment-V

Aim: Determination of chloride content in given water sample.

Materials Required:

- **Apparatus Required**

Burette with Burette stand, Pipettes with elongated tips, Conical flask (Erlenmeyer Flask), Standard flask, Beaker, Wash bottle.

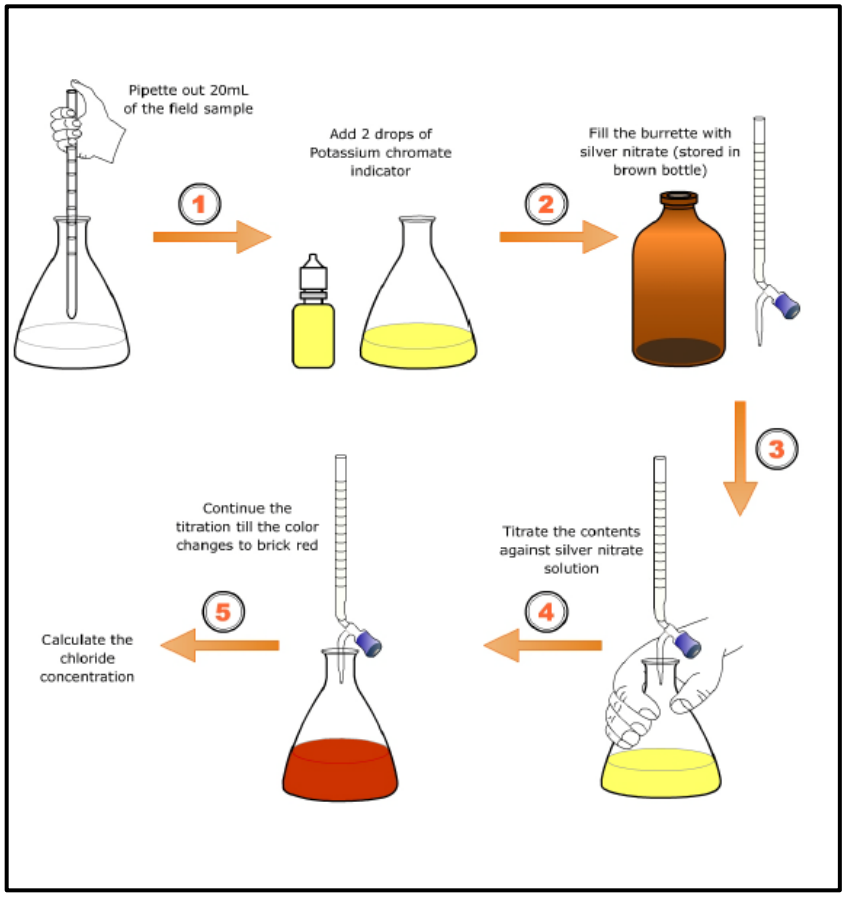
- **Chemicals Required**

Silver nitrate**,** Sodium chloride**,** Potassium chromate.

Theory:

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

- Chloride determinations in natural waters are useful in the selection of water supplies.
- Chloride determination is used to determine the type of desalting apparatus to be used.
- Chloride determination is used to control pumping of ground water from locations where intrusion of seawater is a problem.



Procedure:

Preparation of Reagents:

Standard Sodium Chloride Solution

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

Standard Silver Nitrate (0.0282 N)

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

Potassium Chromate Indicator

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

Testing of Water Sample

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

Blank Titration

1. Take 20 mL of the distilled water in a clean 250mL conical flask
2. Add 1 mL of Potassium Chromate indicator to get light yellow color
3. Titrate the sample against silver nitrate solution until the color changes from yellow to brick red. i.e., the end point.

4. Note the volume of silver nitrate added for distilled water (B).
5. The value of titration is 0.2 mL

Observation table:

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

Calculations:

Volume of Silver Nitrate for sample (Vs) = …… mL

Volume of Silver Nitrate for Blank (V_B) = ……. mL

Normality of Silver Nitrate = 0.0282 N

Volume of Sample = 20.0 mL

Equivalent weight of Chlorine = 35.45

Chlorides (mg/ L) = (Vs – V_B) * Normality * 35.45 * 1000/ Volume of sample taken

Result: The amount of chloride present in the given water sample is = ……..mg/L.

Precautions

• $AgNO_3$ should be stored in a brown amber bottle and should not be exposed to sunlight.

• While handling $AgNO_3$, care should be taken so that it is not spilled on your skin.

• If it spills on your skin, the scar will remain at least for ten to fifteen days.

Experiment-VI

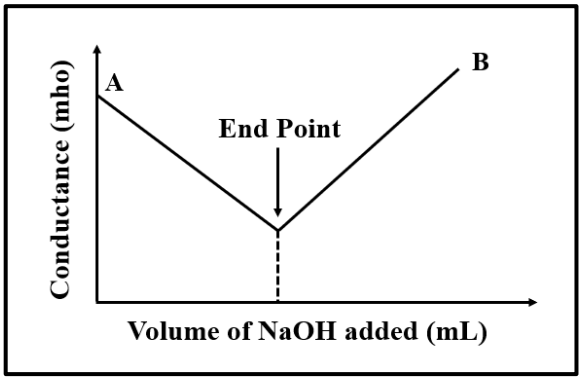
Aim: Determination of strength of HCl solution by titrating it against NaOH solution conductometrically.

Apparatus required: conductivity meter, conductivity cell

Chemical required: 0.01 N KCl, 0.1 N NaOH, 0.1 N HCl, distilled water.

Theory

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



End point of a volumetric analysis can also be found by conductometric titration which involves measurement of conductance of solution during titration. The principle of these titrations is that electrolytic conductance varies during the course of titration as it depends upon ions in solution and their mobility. The end point is found to from a plot of conductance of values against volume of titrant added which gives two lines intersecting each other. The point of intersection gives the end point of titration.

Titration of HCl vs NaOH (strong acid vs strong base) is studied by titrating it a known volume of acid against standard alkali and measuring conductance of solution at different times.

Initially the conductance is high as HCl is strong electrolyte and is highly ionized. On adding NaOH from burette, the conductance decreases, it is because fast moving H^+ ions of HCl are neutralized and replaced by slow moving Na^+ ions.

After complete neutralization, added NaOH increases the conductance of solution due to addition of highly mobile OH^- ions, conductivity is minimum at equivalent point. On plotting conductance vs vol. of NaOH added, a V-shaped graph is obtained (Fig 1). The point of intersection of two lines gives end point. From volume of NaOH used at end point, the strength of HCl solution can be determined.

Procedure

1. Calibrate the conductivity meter using N/10 KCl.
2. Pipette out 50 mL of given HCl solution in a 100 mL beaker.
3. Dip conductivity cell in a HCl solution.
4. Note down the conductance value of solution.
5. Add 1 mL of N/10 NaOH solution from burette. Stir solution with a glass rod and note down the conductance of solution when it becomes stable.
6. Add NaOH solution in 1 mL lots and note the corresponding conductance values till conductance becomes constant.
7. Plot a graph between conductance (Y-axis) and volume of added NaOH solution (along X-axis).
8. Find out the volume of NaOH used at end point of intersection of two lines in graph.

Observation

Temperature of HCl solution = …..^{o}C

Volume of HCl taken = 50 ml

Normality of NaOH taken = N/10

Observation Table: Titration of HCl vs NaOH solution

S. No.	**Vol. of NaOH added (mL)**	**Observed conductance (mho)**
1 2 3 . .	0 1 2 . .	

Calculations:

Let the volume of NaOH used at end point (from graph)

Applying normality equation:

$$N_1V_1 = N_2V_2$$

(HCl) (NaOH)

$$N_1 \times 50 = (1/10) \times V$$

$$N_1 = \ldots\ldots$$

Strength of HCl solution = normality × equivalent wt of HCl

= ……..g L^{-1}

Result: Strength of HCl solution is ……g L^{-1}

Experiment-VII

Section-I

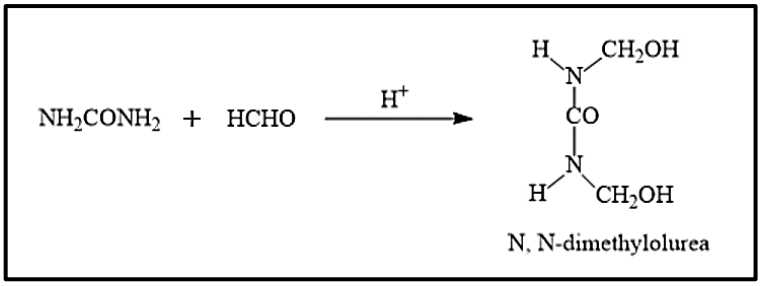
Aim: To prepare Urea formaldehyde resin.

Chemical required: Urea (2 g), 40% aq. formaldehyde solution or formalin (5 mL), conc. H_2SO_4 (3-4 drops).

Theory: Urea formaldehyde resins are formed by condensation of urea and formaldehyde in acidic medium in following steps:

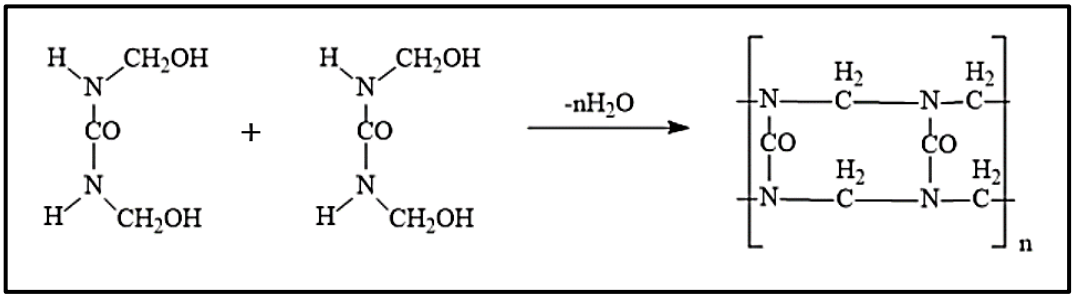
Step 1. Formation of methylol urea derivative

Initially urea and formaldehyde react to form methylol urea derivatives depending upon urea/formaldehyde ratio (U/F ratio).



Step 2: Polymerization of methylol urea

Several molecules of methylol urea derivatives condense with loss of water molecules to form a highly cross-linked urea formaldehyde resin.



Procedure

Take a 5 mL of 40% aqueous formaldehyde solution in a 100 mL beaker. To this add 2 g urea powder. Stir with a glass rod to make a saturated solution. Add a few drops of conc. H_2SO_4 and stir vigorously till a white solid mass is formed. Filter the residue and wash it several times with distilled water to remove any acid. Dry the residue in folds of filter paper or in an oven and weigh. Report the yield of urea formaldehyde polymer formed.

Observation

Weight of empty watch glass = W_1 g

Weight of watch glass + polymer formed = W_2 g

Weight of polymer formed = $W_2 - W_1$ g

Result: Weight of urea formaldehyde resin = …….g

Section-II

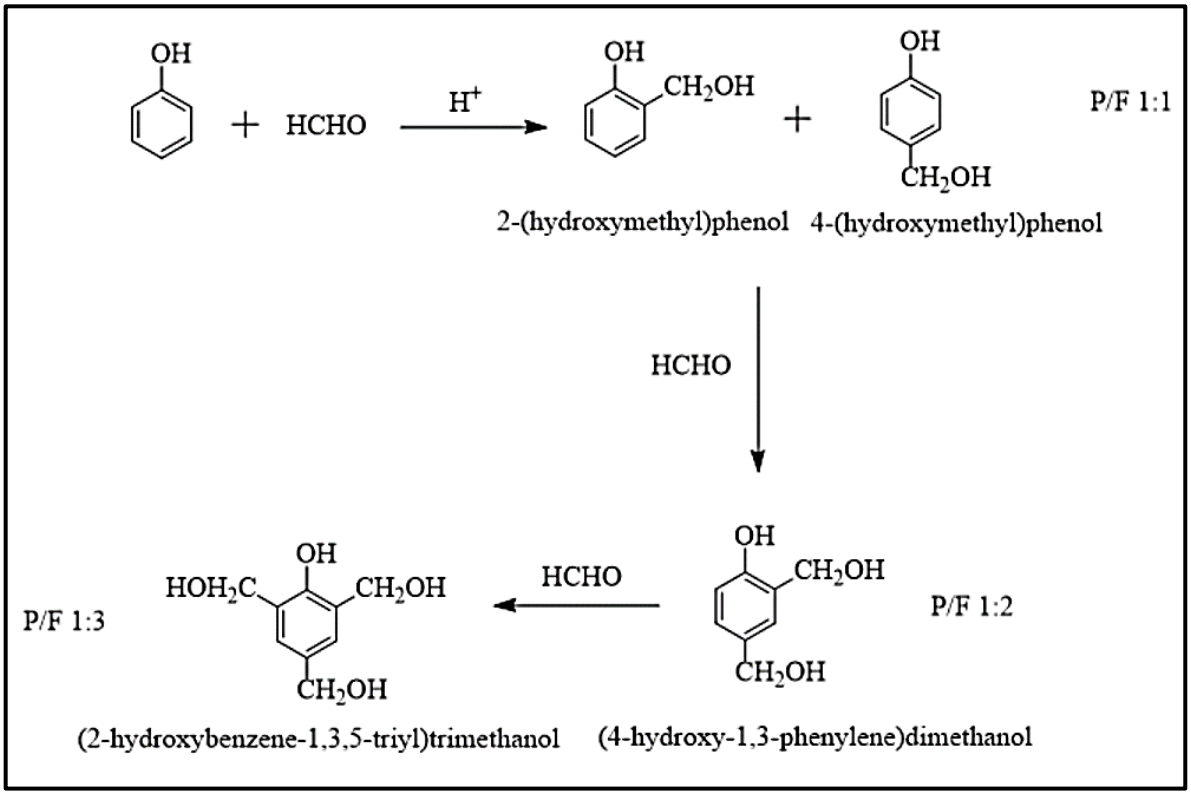
Aim: To prepare Phenol formaldehyde resin.

Chemicals required: Phenol (2 g), 40% aq. formaldehyde solution or formalin (2.5 mL), glacial acetic acid (5 mL) and conc. HCl (8mL).

Theory: Phenol formaldehyde resin or P-F resin or phenolic resins (also called phenoplasts) are important class of polymers which are formed by condensation polymerization of phenol and formaldehyde in acidic or alkaline medium. Following steps are involve.

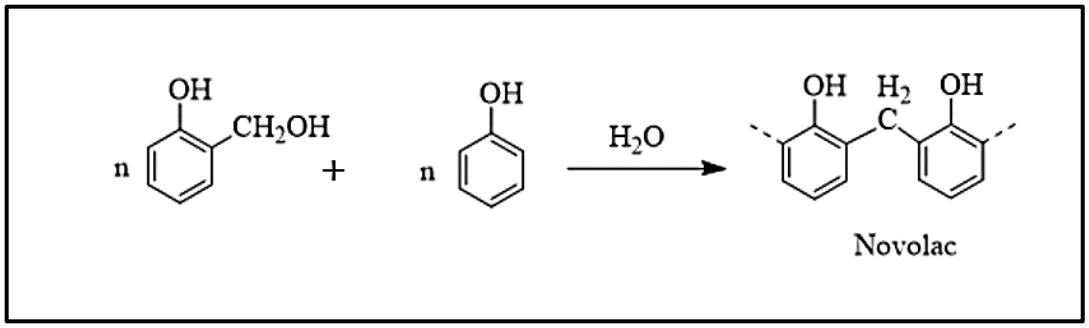
Step 1: Formation of methylol phenol derivative

Initially the monomers combine to form methylol phenol derivative depending upon phenol to formaldehyde ratio.:



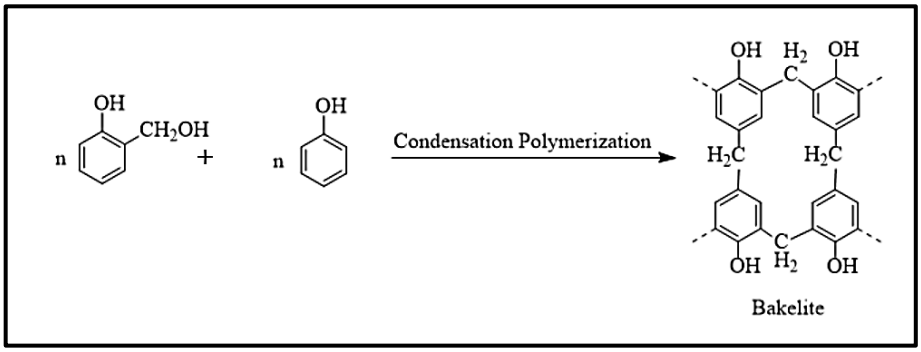
Step 2: The phenol formaldehyde derivatives react among themselves or with phenol to give a linear polymer or a higher cross-linked polymer.

(a) Linear polymer (Novolac)



(b) Cross linked polymer (Bakelite)

A highly cross-linked thermosetting polymer called Bakelite may be formed by further condensation of novolac or methylol derivative.



Procedure:

1. Place 5 mL of glacial acetic acid and 2.5 mL of 40 % aq. formaldehyde solution in a 100 mL beaker. Add 2 g phenol safely.
2. Wrap the beaker with a wet cloth or place it in a 250 mL beaker having small amount of water in it.
3. Add conc. HCl drop wise with vigorous stirring by a glass rod till a pink colored gummy mass appears.
4. Wash the pink residue several times with to make it free from acid.
5. Filter the product and weigh it after drying in folds of a filter or in an oven. Report the yield of polymer formed.

Observations

Weight of empty watch glass = W_1 g

Weight of watch glass + polymer formed = W_2 g

Weight of polymer formed = $W_2 - W_1$ g

Result

Weight of phenol formaldehyde resin = …….. g

Experiment-VIII

Aim: To prepare a pure sample of iodoform.

Chemicals: Acetone (5 mL)**,** Iodine (5 g)**,** NaOH (5 %)**,** Methylated spirit

Theory:

$CH_3COCH_3 + 4I_2 + 4NaOH \rightarrow CHI_3 + 3NaI + CH_3COONa + 2H_2O$

Procedure:

1. Dissolve 5 g of iodine in 5 ml acetone in a conical flask.
2. Add 5 % sodium hydroxide solution slowly with shaking until the colour of iodine is
3. discharged.
4. Allow contents of flask to stand for 10 – 15 minutes.
5. Filter the yellow precipitate of iodoform through Buchner funnel
6. Wash the precipitate with cold water.
7. Dry precipitate between filter paper and weigh it.

Result: Yield of crystals …… g

Colour of crystals …….

Precautions:

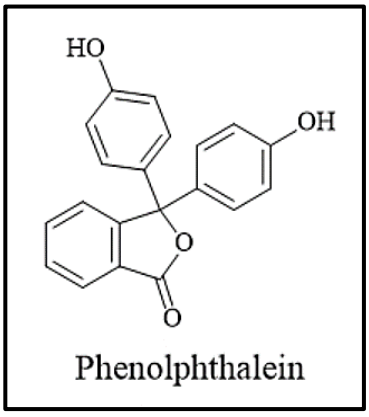
1. Use freshly prepared sodium hydroxide.
2. Add sodium hydroxide slowly and with constant stirring.

Experiment-IX

Aim: To find out saponification number of oil.

Chemicals required: Standard alc. $\frac{N}{2}$ KOH solution, standard alc. $\frac{N}{2}$ HCl solution, ethyl methyl ketone as solvent.

Indictor: Phenolphthalein



Phenolphthalein

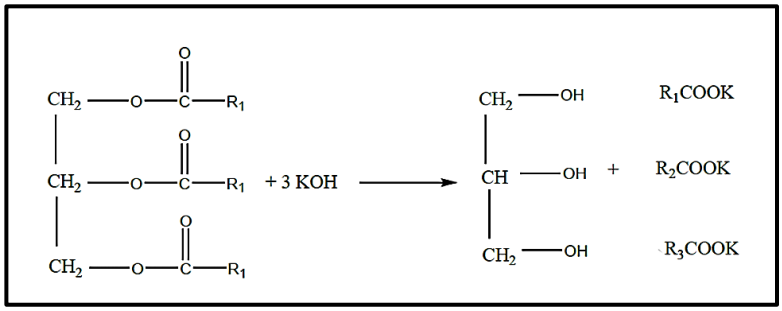
End point: Disappearance of pink colour

Theory

Saponification is process of alkaline hydrolysis of oils (vegetable or animal) and fats giving soap.

Saponification number is defined as number of milligrams of KOH required to saponify 1 mg of a fatty oil.

For determination of saponification number of an oil, a known weight of oil is refluxed with a known excess of standard alc. KOH in a suitable solvent. During refluxing saponification of oil takes place.



Amount of unreacted KOH left behind is determined by titration of mixture against standard HCl using phenolphthalein indicator. Disappearance of pink colour of solution marks the end point.

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

A blank experiment without oil is also performed. From the volume of HCl used in two titrations, saponification of oil can be obtained.

Procedure

1. Take two 250 mL conical flasks and label them as I and II. Weigh accurately about 1-2 gm of given oil in a weighing bottle and transfer it to flask I. Weigh the empty bottle also.

2. Add 25 mL of ethyl methyl ketone and 25 mL of alc. KOH solution to both flasks.

3. Put an air condenser in mouth of each bottle of each flask and keep them for refluxing on a water bath for 45 minutes.

4. Remove the flask from water bath. Wash the inner walls of each condenser with some distilled water into the respective flasks. Remove both condenser and cool both the flasks at room temperature.

5. Add 7-8 drops of phenolphthalein indicator to each flask. Titrate the solution of each flask against N/2 HCl taken in a burette till pink colour just disappears. Note burette reading in both titrations.

Observation table:

Exp. Flasks	Initial burette Reading	Final burette reading (mL)	Volume of HCl used (mL)
For flask I			
For flask II			

Calculations:

Weight of weighing bottle + oil sample = W_1 g

Weight of empty weighing bottle = W_2 g

Weight of oil taken for experiment = $W_1 - W_2$ g

Volume of ethyl methyl ketone added to each flask = 25 mL

Volume of N/2 alc. KOH added to each flask = 25 mL

Volume of N/2 HCl used by unreacted KOH in flask I = A mL

Volume of N/2 HCl used by unreacted KOH in flask II = B mL

Vol. of N/2 HCl is equivalent to volume of N/2 KOH used for saponification of $W_1 - W_2$ g oil
= (B-A) mL

Saponification number of oil = Volume of KOH used * Normality of KOH * Eq. of KOH / weight of Oil

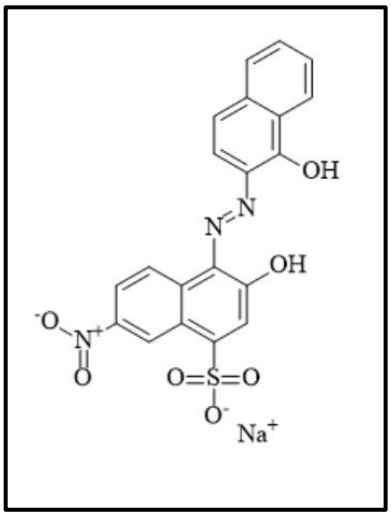
Result: Saponification number of given oil is ……...

Experiment-X

Aim: To determine the Ca^{++} and Mg^{++} hardness/ total hardness of given water sample using EDTA solution.

Apparatus/reagent required: Burette, beaker, conical flask, measuring cylinder, standard hard water, EDTA solution, ammonium buffer solution of $NH_4Cl + NH_4OH$ (pH-10), calcium precipitating buffer solution of $NH_4Cl + NH_4OH + (NH_4)_2C_2O_4$.

Indicator: Eriochrome black–T (EBT).



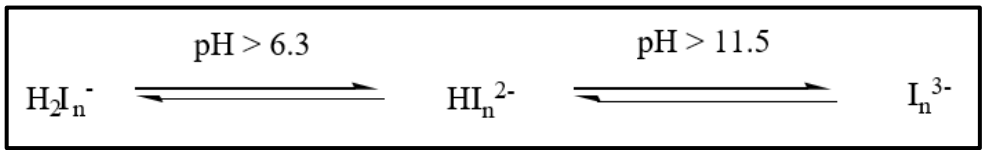
IUPAC: Sodium 1-(1-hydroxy-2-naphthylazo)-6-nitro-2-naphthol-4-sulphonate

End point: Colour changes from wine red to pure blue.

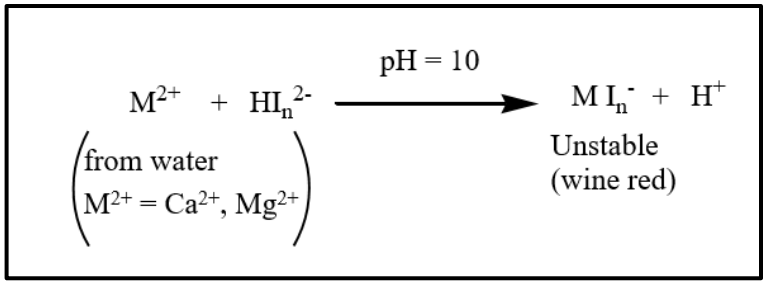
Theory

For finding the total hardness of water, a known volume of water sample, buffered to a pH around 10 with ammonical buffer solution of $NH_4Cl + NH_4OH$, is titrated with standard EDTA solution using EBT as indicator.

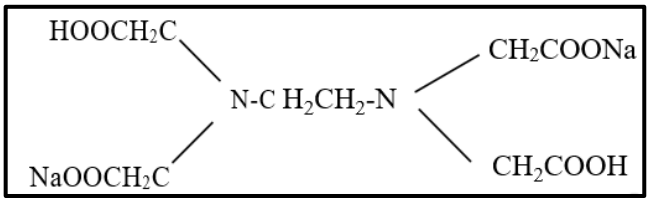
Erio-T indicator is an organic dye having two phenolic ionizable hydrogen atoms. It can have different forms of depending upon pH:



When indicator EBT is added to hard water at a pH around 10 maintained with ammonical buffer, it forms unstable wine-red colored complexes with bivalent metal ions of water as:



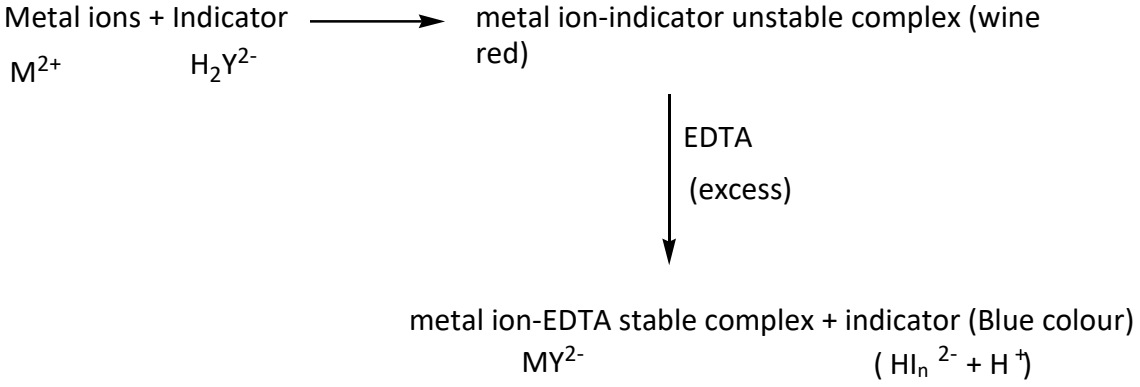
Ethylenediaminetetraacetic acid (EDTA) (Fig 2) is taken in the form of its disodium salt due to higher solubility.

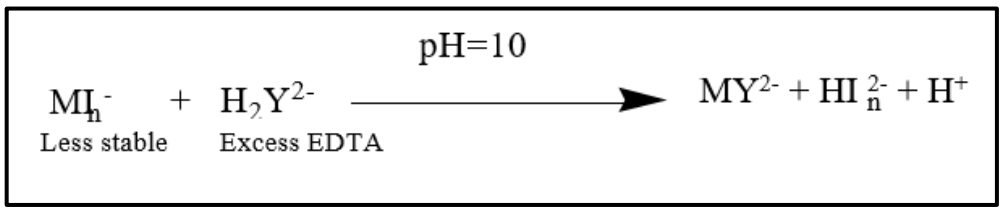


For the simplicity, it can be represented as H_2Y^{2-}. It ionizes in aqueous solution to give a strong chelating ion. It forms complexes with Ca^{++} and Mg^{++}. EDTA when added to wine red solution, it combines with free metal ions of hard water to form their respective soluble complexes. These are more stable than these are more stable than metal-indicator complexes.

When all free metal ions of hard water have complexed with EDTA, a slight excess of EDTA removes metal ions from weak metal indicator complexes to form stronger metal- complexes. This releases the indicator in free form which is blue in colour. This is end point of titration *i.e.* wine red to blue.

Scheme 1





After determination of total hardness in water sample, Ca^{2+} ions in hard water are precipitated as calcium oxalate by adding calcium precipitation solution *i.e.* $NH_4Cl + NH_4OH + (NH_4)_2C_2O_4$.

The solution is filtered to remove white ppt. Titration of filtrate against standard EDTA solution using EBT indicator gives Mg^{2+} hardness in water sample. Hardness due to Ca^{2+} is obtained by subtracting Mg^{2+} hardness from total hardness.

Procedure

Step 1. Standardization of EDTA solution

Fill burette with EDTA solution. Pipette out 20 mL of Standard Hard Water (SHW) in to a conical flask. To this add 5 mL ammonium buffer solution. On addition of 3-4 drops of Erio-T indicator to this wine red colour will appear. Titrate wine red colour solution against EDTA solution till the end point *i.e.* colour change from wine red to blue. Repeat for taking concordant readings. Let V_1 mL of EDTA solution be used.

Step 2. Total hardness in water samples

Titrate 20 mL of given hard water sample against EDTA as in step 1. Let volume of EDTA used be V_2 mL.

Step 3. Determination of Mg^{2+} hardness in sample

Pipette out 100 mL of sample in a 250 beaker. Add 25 mL calcium precipitating buffer solution to this with constant stirring of solution by glass rod. Keep as such for about 30 minutes for settling of white ppt formed. Filter the solution. Titrate 20 mL of given hard water sample against EDTA as in step 1. Let volume of EDTA used be V_3 mL.

General Calculations:

Step 1. Standardization of EDTA solution

1 mL of SHW = 1 mg of $CaCO_3$ (given)

Volume of SHW taken for titration = 20 mL

Concordant volume of EDTA solution used = V_1 mL

20 mL of SHW = V_1 mL of EDTA solution

1 mL of EDTA solution = 20/ V_1 mL of SHW

1 mL of EDTA solution = 20/ V_1 mL of $CaCO_3$

Step 2. Total hardness in water samples

Volume of hard water sample taken for titration = 20 mL

Concordant volume of EDTA solution used = V_2 mL

20 mL of hard water = V_2 mL of EDTA solution = $V_2 \times (20/ V_1)$ mg of $CaCO_3$

So, 1000 mL of hard water = $(V_2 \times (20/ V_1) \times 1000)/20$ mg of $CaCO_3$

Step 3. Determination of Mg^{2+} hardness in sample

Total hardness = $(V_2/ V_1) \times 1000$ ppm of $CaCO_3$

Amount of hard water sample taken for titration = 100 mL

Amount of calcium precipitating buffer added = 25 mL

Hardness of 125 mL prepared solution = Mg^{2+} hardness of 100 mL hard water sample

So, 20 mL filtrate = $(100 \times 20)/125$ = 16 mL of hard water

Concordant volume of EDTA solution used = V_3 mL

So 16 mL of hard water = V_3 mL of EDTA solution = $V_3 \times (20/ V_1)$ mg of $CaCO_3$

1000 mL of hard water = $(V_3 \times (20/ V_1) \times 1000)/16$ mg of $CaCO_3$

Mg^{2+} hardness in water sample = $(V_3 \times (20/ V_1) \times 1000)/16$ ppm in terms of $CaCO_3$

Ca^{2+} hardness in water sample = Total hardness - Mg^{2+} hardness in water sample.

Results:

Hardness due to Ca^{++} in water sample = ……ppm in terms of $CaCO_3$

Hardness due to Mg^{++} in water sample = ……ppm in terms of $CaCO_3$

Precautions

1. All solutions should be freshly prepared.
2. Distilled water should be checked with care before use.
3. The same amount of the indicator must be added to each time.
4. The reaction mixture must be shaken briskly during the reaction.
5. pH = 10 should be maintained during the titration.

Experiment-XI

Aim: Study the adsorption phenomena using acetic acid and charcoal.

Requirement: Beaker, Acetic Acid (5 mL), Funnel, Conical Flask, Measuring Cylinder, Filter Paper, Beaker, Tripod Stand, Burette, NaOH (0.1 N), Stoppered Bottle, pipette.

Indicator: Phenolphthalein

Theory:

Adsorption is a process that occurs when a gas or liquid solute accumulates on the surface of a solid or a liquid (adsorbent), forming a molecular or atomic film (adsorbate). It is different from absorption, in which a substance diffuses into a liquid or solid to form a solution. The term sorption encompasses both processes, while desorption is the reverse process.

Activated carbon, also called activated charcoal or activated coal, is a general term that includes carbon material mostly derived from charcoal. Also, one gram of activated carbon has a surface area of approximately 500 m^2 (for comparison, a tennis court is about 260 m^2).

Factors affecting adsorption:

The extent of adsorption depends upon the following factors:

- Nature of adsorbate and adsorbent.
- The surface area of adsorbent.
- Activation of adsorbent.
- Experimental conditions. E.g., temperature, pressure, etc.

Procedure:

1. Take five stoppered bottles and mark them, as 1, 2, 3, 4 and 5.

2. Add 0.5 N acetic acid to these bottles in the order of 50 mL, 40 mL, 30 mL, 20 mL and 10 mL and later add distil water 0 mL, 10 mL, 20 mL, 30 mL and 40 mL, respectively, to make up the total volume to 50 mL.

3. Now add 1 gm of activated charcoal to each stoppered bottle and shake well properly for 10 mins.

4. Then keep the bottles for 30 mins at room temperature. Filter out the solution from all bottles (1 to 5), with the help of normal filter paper.

5. Take 5 mL solution from each bottle and 1-2 drops of indicator to these solutions. Titrate each solution with 0.1 N NaOH solution, until a color change is observed. Note down the volume of NaOH used.

Observation Table

Serial No.	Volume of Acetic Acid (mL) (0.5 N)	Volume of Distil water (mL)	Normality of Acetic Acid+ Distil Water Solution	Burette Reading		Volume of NaOH Used (mL)
				Initial	Final	
1	50	0				
2	40	10				
3	30	20				
4	20	30				
5	10	40				

Serial No.	Initial Normality of Acetic Acid (C_o)	Final Normality of Acetic Acid+ Distil Water Solution (C_e)	Normality of Adsorbed Acetic Acid (C_o-C_e)	Log x/m (m= 1g)	Log C
1					
2					
3					
4					
5					

Calculations

Applying the normality equation:

$$N_1V_1 = N_2V_2$$

$$x/m = Kc^{1/n}$$

$$\text{Log } x/m = \log K + 1/n \log c$$

$$X = (C_o - C_e) * 60 /1000$$

Result: Adsorption of acetic acid on charcoal follow the isotherm pattern.

Experiment-XII

Aim: Determination of EMF of a Cell.

Requirement: Salt Bridge, copper strip, zinc strip, sand paper, connecting wires, Zinc sulphate solution Copper sulphate solution, voltmeter.

Theory: EMF of the cell increases with decrease in concentration of the electrolyte around the anode and increase in concentration of the electrolyte around cathode.

Procedure

1. Take two clean beakers.
2. In one beaker take 1 M copper sulphate solution and in the other take 1 M zinc sulphate solution.
3. Take a copper strip and clean it using a sand paper. Dip the copper strip into the beaker containing the 1 M copper sulphate solution.
4. Similarly, take a zinc strip and clean it using a sand paper. Then dip it into the beaker containing 1 M zinc sulphate solution.
5. Take a salt bridge and connect the two solutions using the salt bridge.
6. Take a voltmeter and connect the copper strip to the positive terminal and the zinc strip to the negative terminal using connecting wires.
7. Note the position of the pointer in the voltmeter and record the reading.
8. Repeat the experiment by taking different concentrations of zinc sulphate and copper sulphate solutions.

Observation Table

Concentration of $ZnSO_4$ solution (M)	Concentration of $CuSO_4$ solution (M)	EMF of the Cell (V)
1 M	1 M	
1 M	0.5 M	
1 M	0.025 M	
1 M	0.0125 M	
0.5 M	1 M	
0.025 M	1 M	
0.0125 M	1 M	

Result: EMF of Cell……..

Precautions

1. The concentration of copper sulphate and zinc sulphate should neither be too low nor too high.
2. Clean the zinc strip and copper strip with sand paper before use.
3. Connect the copper strip to the positive terminal and the zinc strip to the negative terminal of the voltmeter.
4. The two half cells should be connected using a salt bridge.
5. Note the reading only when the pointer becomes stable.

Experiment-XIII

Aim: To determine the rate constant of a reaction.

Theory: Kinetics and Thermodynamics

All chemical reactions are governed by two factors, kinetics and thermodynamics. The thermodynamic factor is the difference in free energy released during a chemical reaction. This free energy, termed spontaneity, is a complex value arising from the enthalpy (heat) and entropy (disorder) within a chemical reaction. Kinetics refers to the rate of a chemical reaction and how fast the system reaches equilibrium.

While kinetics can explain the speed of the reaction, thermodynamics yields information about its energetics. Simply stated, thermodynamics relates to stability and kinetics relates to reactivity.

Determining Rate

Rate is a time-based measurement, meaning it is constantly changing as a reaction proceeds. This can be represented using a differential rate law, expressing the change in concentration over a change in time. Experimentally, the differential rate law is difficult to use, so we can use calculus and represent the rate law as the integrated rate law by integrating the differential rate law. The integrated rate law represents the reaction concentrations at the start of the reaction and at a specified time interval. A table of order, rate law, and integrated rate law is shown below:

Order	Rate law	Integrated Rate Law	Linear plot	Slope	Units of k
0^{th}	rate = k	$[A]_0 - [A]_t = kt$	[A] vs t	$-k$	M/s
1^{st}	rate = k[A]	$\ln\frac{[A]_t}{[A]_0} = -kt$	ln[A] vs t	$-k$	s^{-1}
2^{nd}	rate = k[A]^2	$\frac{1}{[A]_t} - \frac{1}{[A]_0} = kt$	$\frac{1}{[A]}$ vs t	k	$M^{-1}s^{-1}$

Each order explains the dependence of reactant concentration on reaction rate. For example, a zeroth order rate law, rate = k, indicates that the rate is only dependent on the rate constant, not on reactant concentration. This is common in catalytic reactions where the catalyst is a solid and the surface area does not change during the reaction. A 1st order rate law shows that the rate is dependent on the concentration of one reactant, though other reactants may be present. A 2nd order rate law indicates that the rate is dependent on the concentration of two reactants in the reaction. Those reactants can be the same, i.e. rate = k$[A]^2$, or different, rate = k[A][B]. Since the two concentrations are constantly changing, second order rate constants can be difficult to measure in the lab. Regardless of the order, using the integrated rate law simplifies data analysis by allowing for data plotting and applying a linear equation to fit the data. Since only one integrated rate law will fit the data, a reaction's rate constant and reaction order can be immediately identified.

A Kinetics Experiment

Determining the rate law begins with setting up a kinetics experiment for the chemical reaction. A kinetics experiment is carefully controlled so that measurements are made in timed intervals in order to determine the change in concentration of a species over time. That species can either be a reactant (decreasing concentration with time) or a product (increasing concentration with time). If multiple reactants are involved, it is also very important that the concentration of only one reactant changes with time. Increasing the concentration of the other reactants much higher than the reactant being studied makes it appear that the concentration of only one reactant changes during the experiment.

In this experiment, the catalytic decomposition of hydrogen peroxide over a platinum catalyst is explored. Since the platinum is a catalyst, only one species is involved which decomposes into two products according to the reaction below:

$$2\ H_2O_2(aq) \rightarrow O_2(g) + 2\ H_2O(l)$$

Because one of the products, O_2, is a gas, the increase in pressure of the system over time can be measured and the Ideal Gas Law (PV = nRT) used to relate pressure to moles. Once that is done for several different concentrations of the reactant, the reaction order and rate law can be solved.

Procedure

Procedure

1. *Preparing H_2O_2 Dilutions*

Stock 3% hydrogen peroxide has a concentration of 0.882 M. Prepare 5 dilutions ranging from 0.882 M to 0.176 M (Table 1). Prepare these solutions volumetrically, but prepare them additively since the solute is very dilute and volumes of water are additive.

Place the solutions in a constant temperature water bath or leave them on the bench top to equilibrate at room temperature. A temperature range of 20–25 °C (293–298 K) is good for this reaction.

Table: H_2O_2 solution used

	Required volume of 3% H_2O_2 solution (mL)	Required volume of water (mL)	Concentration of H_2O_2 (M)
Trial 1 (3.0%)	50	0	0.882
Trial 2 (2.4%)	40	10	0.706
Trial 3 (1.8%)	30	20	0.530
Trial 4 (1.2%)	20	30	0.353
Trial 5 (0.6%)	10	40	0.176

2. *Preparing the Reaction Vessel*

To determine the volume of the reaction vessel, fill a large test tube to the top with water and insert a 1-hole rubber stopper into the test tube until tight and water pushes out the sides and through the top. Remove the stopper and pour the water into a graduated cylinder to determine the exact volume of the water. This is the total volume of the reaction vessel (test tube).

3. *Measuring Oxygen Evolution*

Replace the water with 50 mL of the first hydrogen peroxide solution and place it back into the water bath. Once equilibrated, add the platinum-coated reaction disc and seal the system with a stopper connected to a gas pressure sensor. These discs are commonly used in contact lens cleaning systems. Once the pressure sensor is setup to acquire data at 2 points/s, run the experiment for 120 s. The Vernier gas pressure sensor, GPS-BTA, is recommended for this experiment. Bubbles should be observed as the peroxide is decomposed to oxygen gas and water. Release the pressure, dispose of the solution, rinse, and replace the solution with the next hydrogen peroxide solution. Repeat the gas pressure measurement until all solutions are tested.

Calculations:

Determining the Rate Constant, k

For each trial, convert the rate, P_{O2}/s, into units of atm/s if the rate is in a different unit such as torr/s. Because bubbles were evolved in aqueous solution, subtract the vapor pressure of water from the system pressure for each trial. The new rate reflects only the pressure due to oxygen evolution. Apply the Ideal Gas law to convert the rate from atm/s into moles/s in each trial.

Rearrange PV = nRT to n = PV/RT. The s^{-1} unit remains unchanged. The volume is equivalent to the test tube volume minus the solution volume (50 mL). Use the balanced chemical reaction to convert from moles of oxygen produced to moles of hydrogen peroxide decomposed in each trial.

Divide the moles of H_2O_2 by the volume of the solution, 0.050 L, to yield the molarity of H_2O_2 decomposed per second, $[H_2O_2]/s$.

Because this experiment follows first-order kinetics, divide the rate, $[H_2O_2]/s$, by the original solution concentration for each trial, $[H_2O_2]_0$, to yield a rate constant, k. This solution for the rate constant would vary slightly based on the order of the reaction previously determined.

Average the rate constants for each trial together since the temperature is constant.

Result: Rate Constant of a reaction……..

Experiment-XIV

Aim: Determination of the partition coefficient of a substance between two immiscible liquids.

Requirement: 4 separation funnels (100 mL), 4 titrimetric flasks, burette, pipettes, 8 beakers, succinic acid (aqueous solution c=20 g/dm^3), NaOH (c=0.05 mol/dm^3), 1-butanol, phenolphthalein, distilled water.

Theory:

Consider a system consisting of two liquid layers (phases) of two immiscible or partially miscible liquids. If a third substance, which is soluble in both liquids, is added into the system it is found to distribute, or

divide, itself between the two layers in a definite manner. It has been shown experimentally that an equilibrium, at constant temperature, the ratio of the concentrations in the two layers has a definite value, independent of the actual amount of the dissolved substance. If c_1 and c_2 are the concentrations of this substance in the two layers, then:

$$k = \frac{c_1}{c_2} \qquad (1)$$

The dissolved substance irrespective of its total amount, distributes itself between the two layers in a constant concentration ratio, at constant temperature. The ratio, equal to the constant in Equation 1 is referred as the **partition constant or partition coefficient (k) (or sometime designated as P)**.

It will be shown below, that the distribution law in the form of Equation 1 can be applied only to dilute solutions. It is supposed also, that the partitioned substance undergoes no chemical changes, such as association or dissociation, in any of the two liquid phases. When two phases are in equilibrium the chemical potential (μ) of the partitioned substance will be the same in both phases. The chemical potential of the dissolved substance in any of the two phases may be represented by:

$$\mu_1 = \mu_1^0 + RT \ln a_1 \qquad \text{and} \qquad \mu_2 = \mu_2^0 + RT \ln a_2 \qquad (2)$$

where: a_1 and a_2 are the respective activities of the partitioned solute in two phases.

When the system is at equilibrium μ_1 and μ_2 must be equal, so that:

$$\mu_2^0 - \mu_1^0 = RT \ln \frac{a_1}{a_2} \qquad (3)$$

At constant temperature the standard chemical potentials are constant, and so are R and T; it follows, therefore, that:

$$\frac{a_1}{a_2} = const \qquad (4)$$

which is the exact form of the distribution law. For systems which are ideal or do not depart appreciably from ideal behaviour, the ratio of the activities may be replaced by the ratio of the mole fractions, i.e., x_1/x_2 should be constant. Further, for dilute solutions or for gases, the ratio of the concentrations may be used instead of the ratio of the mole fractions, and so in these circumstances c_1/c_2 should be constant, in agreement with the simple form of the distribution law given by the Equation 1.

Procedure:

1. Prepare 4 mixtures of butanol, water and succinic acid into separation funnels (Figure 1), following Table 1.

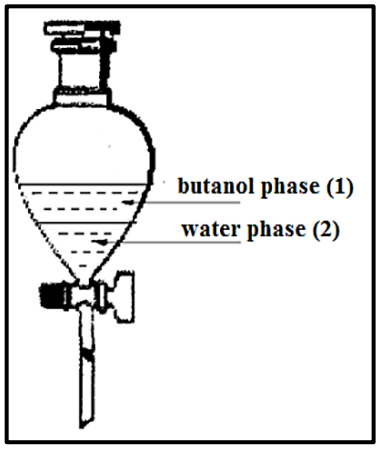


Figure 1. Separating funnel.

Table1. Composition of partition coefficients.

Funel No.	succinic acid [cm^3]	distilled water [cm^3]	1-butanol [cm^3]
1	20	-	15
2	10	15	15
3	15	-	15
4	10	10	15

2. Shake gently the separating funnels with prepared solutions for 20 min.
3. Put them to the holders and wait a few minutes until the mixture is apparently separated into two clear layers: lower layer is water (Wat), and upper layer is butanol (Org).
4. Pour carefully the water phase into a beaker using the funnel's valve.
5. Transfer the water solution from the beaker into two titrimetric flasks, 5 ml per flask, using a pipette with volume (V=5 ml).
6. Add 1 drop of phenolphthalein.
7. Fill a burette with a standard solution of NaOH (titrant) and perform titration.
8. When the endpoint of titration has been reached, read the used volume of the titrant, NaOH from the burette (VT). Write it down to the Table 2.
9. Repeat the procedure for funnels 2 – 4.
10. Proceed in the same way the upper layer (butanol) of the mixtures in the funnels, 1 – 4.

Observation Table:

Meas. No.	Volume of titrant (V_T) ml						c_{Org} mol dm^{-3}	c_{Wat} mol dm^{-3}	k_i
	1-butanol			water					
	$V_{1,1}$ [ml]	$V_{1,2}$ [ml]	$\overline{V_1}$ [ml]	$V_{2,1}$ [ml]	$V_{2,2}$ [ml]	$\overline{V_2}$ [ml]			
1									
2									
3									
4									

$\overline{k}$ =.....

Calculation:

$$k = \frac{c_{Org}}{c_{Wat}}$$

Result: The partition coefficient (k_i) of succinic acid (given substance) at each composition of the partition mixture……..

Experiment-XV

Aim: Lattice Structure and Packing of Sphere.

Requirement: Simple unit cell model, Face-centered unit cell model and Body-centered unit cell model.

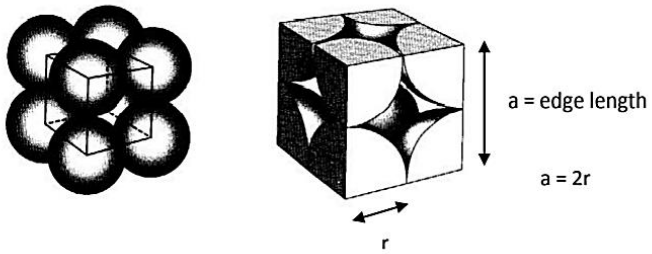
Theory:

Solid materials exist as a crystalline lattice. A crystalline lattice is an orderly arrangement of particles in a three-dimensional arrangement. The particles making up the crystal lattice may be atoms, molecules, or ions. The unit cell is the smallest part of the lattice that represents the entire array. Repeating the unit cell in three directions in space produces the crystal lattice. Shown below is a crystal lattice for sodium chloride formed from simple cubic unit cells.

The unit cell contains pockets of empty space which varies depending upon the type of unit cell. The arrangement of the atoms within a unit cell is called packing. Assuming the shape of an atom to be a sphere, any type of packing of the atoms within a unit cell would leave space in between the atoms. In order to find out how much space in a cube is occupied by the atoms, both the volume of the unit cell and the volume of the atomic sphere must be determined. The total volume of the sphere must account for all of the atoms (spheres) are contained within the unit cell. In this experiment, the percentage of space occupied by the atoms will be determined for the following three-unit cells: simple cubic, face centered

cubic, and body-centered cubic.

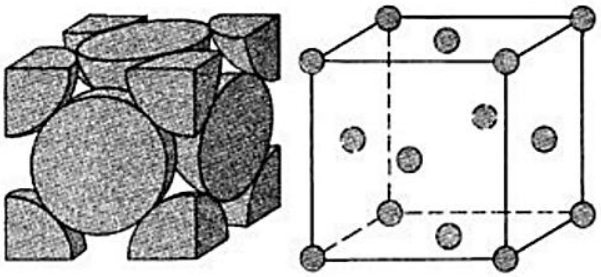
Simple Cubic Unit Cell



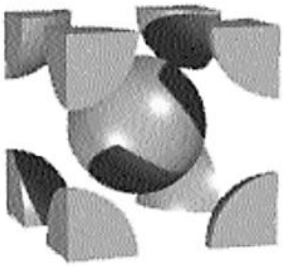
$V = 8r^3$

Face-Centered Unit Cell

$V = 16\sqrt{2}r^3$



Body-Centered Unit Cell



$V = \frac{64\sqrt{3}}{9}r^3$

Procedure:

The volume of a sphere is 4/3 πr^3. Assume that the radius of each sphere is 1.00 cm. (1 cm^3 = 1 mL)

Simple Cubic

1. Find the volume of the space occupied in the unit cell:
    a) Count the number of atoms(spheres) inside the unit cell.
    b) Calculate the total volume of the spheres inside the unit cell.
2. Find the volume of the unit cell using the above volume formula.
3. Calculate the percentage of space occupied in the unit cell.

$$\%\ space\ occupied\ by\ the\ spheres = \frac{V_{spheres}}{V_{Cell}} \times 100$$

Face-Centered Cubic

1. Find the volume of the space occupied in the unit cell:
    a) Count the number of atoms(spheres) inside the unit cell.
    b) Calculate the total volume of the spheres inside the unit cell.
2. Find the volume of the unit cell using the above volume formula.
3. Calculate the percentage of space occupied in the unit cell.

$$\%\ space\ occupied\ by\ the\ spheres = \frac{V_{spheres}}{V_{Cell}} \times 100$$

Body-Centered Cubic

1. Find the volume of the space occupied in the unit cell:
    a) Count the number of atoms(spheres) inside the unit cell.
    b) Calculate the total volume of the spheres inside the unit cell.
2. Find the volume of the unit cell using the above volume formula.
3. Calculate the percentage of space occupied in the unit cell.

$$\%\ space\ occupied\ by\ the\ spheres = \frac{V_{spheres}}{V_{Cell}} \times 100$$

Observation Table:

Cube	No. of Atoms	Vol of spheres	Vol of cell	% occupied	Density (g/mL)
Simple					
Face-centered					
Body-centered					

Calculations:

Assuming that the above solids are all aluminum, calculate the density for each unit cell.

$$d = \frac{mass}{volume} = \frac{g}{mL}$$

$$mass = No of\ atoms \times \frac{mol}{6.02x10^{23}} \times \frac{26.98g}{mol}$$

Result: Percentage space occupied by sphere…….

Experiment-XVI

Aim: Chemical analysis of two anions and two cations in given sample of salt.

Method:

Copper ion (Cu^{2+})

Copper ion is a microelement, and is necessary to activate the reserves of Fe^{2+} for hemoglobin biosynthesis. Copper is present in the blood, bound to the glycoprotein called ceruloplasmin.

Detection of Cu^{2+}

Ammonium hydroxide precipitates from $CuSO_4$ solution white-bluish, basic copper sulfate. After adding of ammonium hydroxide in excess, the solution clarifies and the color changes to dark blue, signaling formation of complex salt $[Cu(NH_3)_4]SO_4$ (tetraamminecopper (II) sulphate).

$$2CuSO_4 + 2NH_3 \cdot H_2O \rightarrow \downarrow[CuOH]_2SO_4 + (NH_4)_2SO_4$$
$$[CuOH]_2SO_4 + 6NH_3 \cdot H_2O + (NH_4)_2SO_4 \rightarrow 2[Cu(NH_3)_4]SO_4 + 8H_2O$$

Procedure: Add a few drops of 2M $NH_3 \cdot H_2O$ to 1 ml of $CuSO_4$ solution. The precipitate is white-bluish basic salt $[CuOH]_2SO_4$ that solubilizes in an excess of ammonia, giving the dark-blue color product – a tetraamminecopper sulphate – $[Cu(NH_3)_4]SO_4$.

Detection of Ag^{+}

Hydrochloric acid or soluble chlorides precipitate white silver chloride sediment from solutions containing soluble silver salts (e.g.silver nitrate). Silver chloride can be solubilized in ammonium hydroxide ($NH_3 \cdot H_2O$), yielding complex compound - $[Ag(NH_3)_2]Cl$, diamminesilver chloride.

$$AgNO_3 + HCl \rightarrow \downarrow AgCl + HNO_3$$
$$AgCl + 2\ NH_3 \cdot H_2O \rightarrow [Ag(NH_3)_2]Cl + 2H_2O$$
Diamminesilver chloride

.

Procedure: Add 0.5 - 1.0 ml of silver salt solution to a test tube and then add 2 drops of 2M HCl. Observe formation of AgCl white precipitate. Take a portion of the precipitate and transfer to another test tube. Add the $NH_3 \cdot H_2O$ solution in excess. The precipitate will dissolve. AgCl precipitate can be obtained again on addition of nitric acid to the solution:

$$[Ag(NH_3)_2]Cl + 2\ HNO_3 \rightarrow \downarrow AgCl + 2\ NH_4NO_3$$

Chloride ion (Cl^-)

Chloride is very important anion, present in blood serum and in other extracellular media. Chloride ions are very common in nature and are present in tap water.

Silver nitrate ($AgNO_3$) reacts with chloride solution to precipitate white silver chloride. Silver chloride is soluble in excess of $NH_3 \cdot H_2O$, in solutions of $Na_2S_2O_3$, KCN and concentrated HCl.

Detection of Cl^-

$$NaCl + AgNO_3 \rightarrow \downarrow AgCl + NaNO_3$$

$$AgCl + 2\ NH_3 \cdot H_2O \rightarrow [Ag(NH_3)_2]Cl + 2\ H_2O$$

Diamminesilver chloride

Procedure: Add a few drops of silver nitrate solution to a test tube containing 0.5 - 1.0 ml of chloride anions solution. As a result AgCl precipitate is formed. Transfer a portion of the precipitate to another test tube and add drop by drop 2M $NH_3 \cdot H_2O$ solution, until the precipitate is dissolved. AgCl can be reprecipitated again upon addition of nitric acid.

Detection of CO_3^{2-}

1. Calcium nitrate or barium hydroxide precipitates from carbonic ions solutions a sediment of calcium or barium carbonate:

$$Ca(NO_3)_2 + Na_2CO_3 \rightarrow \downarrow CaCO_3 + 2\ NaNO_3$$

Calcium carbonate

Procedure: Add a few drops of calcium nitrate ($Ca(NO_3)_2$) solution to 0.5 – 1.0 ml of carbonate ions solution to obtain a white sediment of calcium carbonate.

2. Strong acids decompose carbonate salts, releasing carbon dioxide gas, which can be detected as gas bubbles on the test tube walls, or in a form of white sediment of barium carbonate, after adding a few drops of baryta water - $Ba(OH)_2$.

$$Na_2CO_3 + 2\ HCl \rightarrow 2\ NaCl + H_2CO_3$$

$$H_2CO_3 \rightarrow \uparrow CO_2 + H_2O$$

$$CO_2 + Ba(OH)_2 \rightarrow \downarrow BaCO_3 + H_2O$$

Barium carbonate

Procedure: Add a few drops of 1M H_2SO_4 (or HCl) solution to the carbonate salt solution (0.5 - 1.0 ml) and seal the test tube using cork stopper with U-shaped glass tubing. Carefully mix the tube content and observe gas bubbles, settling on the tube walls. Quickly immerse free end of the tubing in baryta water and observe formation of barium carbonate white sediment.