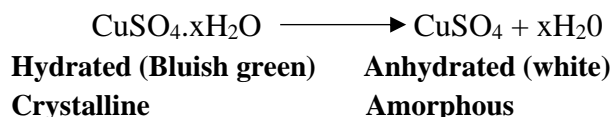




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Experiment 1: Determination of the water of crystallization of copper (II) sulfate (CuSO_4).

Water of crystallization is the number of water molecules, chemically combined in a definite molecular proportion, with the salt in its crystalline state. This water is responsible for the geometric shape and color of the crystals. Copper sulfate exists in hydrated form, as $\text{CuSO}_4 \cdot x\text{H}_2\text{O}$, which is bluish green in color. On heating, water molecules are expelled from hydrated copper (II) sulfate and anhydrous copper (II) sulfate is formed.



It is possible to calculate the 'x' value from the weight of CuSO_4 (before and after strong heating).

Chemical: Hydrated copper (II) sulfate salt

Apparatus: Crucible, Electric balance, Magnetic hotplate, etc.

Procedure

1. Weigh out a dry crucible and record it as first reading.
2. Take 1.0 g hydrated copper sulfate in the crucible and record the weight as a second reading.
3. Place the crucible on the hot plate and heat it for 5-10 min until the bluish green color changes to white salt.
4. Allow it to cool down and record the weight as third reading.

Experimental data

- Weight of crucible =
- Weight of crucible + hydrated CuSO_4 =
- Weight of **hydrated CuSO_4** = [(weight of crucible + hydrated CuSO_4) - weight of crucible]
- Weight of the crucible (with CuSO_4) after heating =
- Weight of **anhydrous CuSO_4** = [weight of the crucible after heating - weight of crucible]
- Weight of expelled H_2O = [weight of hydrated CuSO_4 - weight of anhydrous CuSO_4]

Calculation

n mole of CuSO_4 is chemically combined with n moles of H_2O

$$\begin{aligned} 1 \text{ mole of } \text{CuSO}_4 & \dots\dots\dots = \frac{n \text{ moles of } \text{H}_2\text{O}}{n \text{ moles of } \text{CuSO}_4} \\ & = \frac{\text{Weight of expelled } \text{H}_2\text{O} \div \text{Molecular mass of water}}{\text{Weight of anhydrated } \text{CuSO}_4 \div \text{Molecular mass of } \text{CuSO}_4} \end{aligned}$$

$$\begin{aligned} \text{Percentage (\%) of error} &= \left(\frac{\text{Theo. value} - \text{exp. value}}{\text{Theo. value}} \right) \times 100 \\ &= \dots\dots\dots \% \end{aligned}$$

Result:

Practices

1. What do you mean by crystallization of water?
2. Calculate the molecular mass of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$.
3. Define Lattice energy.
4. Explain crystalline and amorphous state.
5. What do you mean by hydrated and anhydrated CuSO_4 ?
6. What is blue vitriol and its applications in daily use?
7. Discuss the precautions of the above experiment.



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Experiment 2: Standardization of a strong acid (HCl) with a weak base (Na₂CO₃).

Sodium carbonate (Na₂CO₃) is the salt of a weak acid, carbonic acid (H₂CO₃). Its reaction with HCl is a two-step process and may be represented as,



The overall reaction may be represented as



Here, 1 mol of Na₂CO₃ reacts with 2 moles of HCl, so the following expression can be written as,

$$(M_{\text{Na}_2\text{CO}_3} \times V_{\text{Na}_2\text{CO}_3}) / (M_{\text{HCl}} \times V_{\text{HCl}}) = 1/2$$

$$\text{or, } (M_{\text{HCl}} \times V_{\text{HCl}}) = 2 \times (M_{\text{Na}_2\text{CO}_3} \times V_{\text{Na}_2\text{CO}_3}) \dots\dots\dots (4)$$

Chemicals: Na₂CO₃, HCl, Methyl orange.

Apparatus: Volumetric flask, conical flask, burette, pipette, funnel, wash bottle, etc.

Procedure

a) Preparation of standard 0.05 M Na₂CO₃ solution

Weigh out of 1.325 gm of Na₂CO₃ accurately on an electric balance by using this formula, $w = SMV/1000$ ($= 0.05 \text{ M} \times 106 \times 250 \text{ mL}/1000$) and place it in a 250 mL volumetric flask. Add a small amount of distilled water and shake mildly until all the solute dissolves in water. Fill up to the 250 mL mark with water.

b) Standardization of a strong acid (HCl)

1. After cleaning a burette with distilled water, take the supplied HCl solution in the burette. Set up the burette with a stand and record the initial reading.
2. Pipette out 10 mL of standard Na₂CO₃ solution into a 250 mL conical flask; add 2 drops of methyl orange (**indicator**), turns into yellowish solution.
3. Titrate with the HCl solution until the first drop changes its color from yellow to red and at the end point, record the final reading from the burette.
4. Calculate the difference between two burette readings, which is the amount of HCl solution required to neutralize the Na₂CO₃ solution.
5. Repeat the titration two more times.
6. Record the data for pH and volume changes during titration and draw a graph of pH vs volume showing end point.

Experimental data: Standardization of supplied HCl solution with the standard Na₂CO₃ solution.

No. of observation	Volume of HCl solution (mL)			Average volume (mL)
	IBR	FBR	Difference	
1.				
2.				
3.				

Calculation

From equation (4), calculate the concentration of HCl, $M_{\text{HCl}} = \frac{2 \times M \text{ Na}_2\text{CO}_3 \times V \text{ Na}_2\text{CO}_3}{V \text{ HCl}}$

$$\text{Percentage (\%) of error} = \left(\frac{\text{Theo. value} - \text{exp. value}}{\text{Theo. value}} \right) \times 100$$
$$= \dots\dots\dots \%$$

Result:

Practices

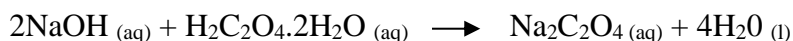
1. What do you mean by standard solution and standardization?
2. Define primary/secondary standard substances and give examples.
3. What is molarity?
4. Why is methyl orange used as an indicator in this experiment?
5. What is the name of the technique used in this experiment?
6. What do you mean by end point and equivalent point?
7. Discuss the precautions of the above experiment.



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Experiment 3: Standardization of a strong base (NaOH) with a standard weak acid (H₂C₂O₄)

Standardization is the process of determining the exact concentration of a solution. Titration is one type of analytical procedure often used in standardization. In a titration, an exact volume of one substance is reacted with a known amount of another substance. Strong bases (NaOH/KOH etc.) are defined as the chemical substances that can be dissociated into their respective ions completely. On the other hand, weak acids (H₂C₂O₄, H₂CO₃ etc.) are substances that can be dissociated into their respective ions partially. The reaction between NaOH and H₂C₂O₄ is follows,



Chemicals: NaOH, H₂C₂O₄·2H₂O, Phenolphthalein, etc.

Apparatus: Volumetric flask, Conical flask, Burette, Pipette, Funnel, Wash bottle, etc.

Procedure

a) Preparation of 0.05 M, 250 mL oxalic acid solution

Weigh out of 1.575 gm of oxalic acid dihydrate by following this formula, $w = \text{SMV}/1000$ (where, molar mass of oxalic acid dihydrate is 126.03 g/mol) in 250 mL volumetric flask and add small amount of distilled water. Shake the flask to dissolve the solute. Finally add more water up to 250 mL mark.

b) Standardization of NaOH

1. After cleaning the burette with distilled water, fill up the burette with supplied NaOH and carefully set up the burette with a stand.
2. Record the initial burette reading.
3. Take 10 mL of 0.05 M oxalic acid solution in a conical flask with the help of pipette.
4. Add two drops of phenolphthalein indicator.
5. Titrate until the last drop of NaOH solution leaves a permanent pink color in the solution and record the final burette reading.
6. Repeat the titration two more times.
7. Record the data for pH and volume changes during titration and draw a graph of pH vs volume showing end point.

Experimental data: Standardization of supplied NaOH solution with H₂C₂O₄ solution.

No. of observation	Volume of NaOH solution (mL)			Average volume (mL)
	IBR	FBR	Difference	
1.				
2.				
3.				

Calculation

According to the reaction it can be written as, $2 \times M_{\text{acid}} \times V_{\text{acid}} = M_{\text{base}} \times V_{\text{base}}$

Therefore, strength of NaOH, $M_{\text{base}} = \frac{2 \times M_{\text{acid}} \times V_{\text{acid}}}{V_{\text{base}}}$

$$\begin{aligned} \text{Percentage (\%) of error} &= \left(\frac{\text{Theo. value} - \text{exp. value}}{\text{Theo. value}} \right) \times 100 \\ &= \dots\dots\dots \% \end{aligned}$$

Result:

Practices

1. What do you mean by strong base and weak acid?
2. Write down the characteristics of primary and secondary standard substances.
3. Why is phenolphthalein considered a suitable indicator for this type of titration?
4. Explain using titration curve.
5. Define molarity and normality.
6. What do you mean by pH and pOH?
7. Discuss the precautions of the above experiment.



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Experiment 4: Determination of the molar mass of an unknown diprotic acid using NaOH.

A monoprotic acid is an acid that donates only one proton or hydrogen atom per molecule to an aqueous solution, such as: Hydrochloric acid (HCl), Nitric acid (HNO₃), Acetic acid (CH₃COOH). The molar mass of a chemical compound is the mass of a sample of that compound divided by the amount of substance in that sample, measured in moles. Molar masses are usually expressed in g/mol. Chemical reactions between acids and bases are used to analyze the quantity of a pure substance in a mixture. In this experiment, the standardized base, NaOH, is used to determine the molar mass of an unknown weak acid. Based on the molar mass, one can determine the identity of the unknown weak acid. The reaction as follows,



Since 1 mol of NaOH reacts with 1 mol of HA, the following expression can be written as

$$(M_{\text{HA}} \times V_{\text{HA}}) = (M_{\text{NaOH}} \times V_{\text{NaOH}}) \dots\dots\dots (1)$$

where, M_{NaOH} = Molarity of NaOH

V_{NaOH} = Volume of NaOH (**average burette reading**)

V_{HA} = Volume of HA

M_{HA} = Molarity of HA

Chemicals: Diprotic acid, NaOH, Phenolphthalein, etc.

Apparatus: Volumetric flask, Conical flask, Burette, Pipette, Funnel, Wash bottle, etc.

Procedure

a) Preparation of 0.1 M, 250 mL NaOH solution

Weigh out of 1.000 gm of NaOH by following this formula, $w = \frac{SMV}{1000}$ (where, molar mass of NaOH = 40 g/mol) in 250 mL volumetric flask and add small amount of distilled water. Shake the flask to dissolve the solute. Finally add more water up to 250 mL mark.

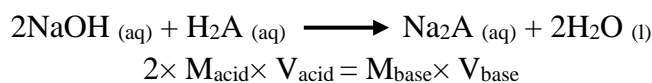
b) Determination of the concentration of H₂A

1. Fill the burette with prepared NaOH and carefully set up the burette with a stand.
2. Record the initial burette reading.
3. Take 10 mL of H₂A (as 3.15 g in 500 mL) in a conical flask with the help of pipette.
4. Add two drops of phenolphthalein indicator.
5. Titrate until the last drop of NaOH solution leaves a **permanent pink color** in the solution and record the final burette reading.
6. Repeat the titration two more times.

Experimental data: Determination of the concentration of NaOH solution with H₂A solution.

No of observation	Volume of NaOH solution (mL)			Average volume (mL)
	IBR	FBR	Difference	
1.				
2.				
3.				

Reaction



Calculation

- Concentration of H₂A, $M_{\text{acid}} = Y \text{ mol/L} = Y \text{ M}$
- Moles of H₂A, $n = Y \times (500 \text{ mL}/1000 \text{ mL}) \text{ mol}$
- Mol, $n = \text{Weight}/\text{Molar Mass} = 3.15/\text{Molar Mass}$

Therefore, Molar mass = g/n

$$\text{Percentage (\%) of error} = \left(\frac{\text{Theo. value} - \text{exp. value}}{\text{Theo. value}} \right) \times 100$$

$$= \dots\dots\dots \%$$

Result:

Practices

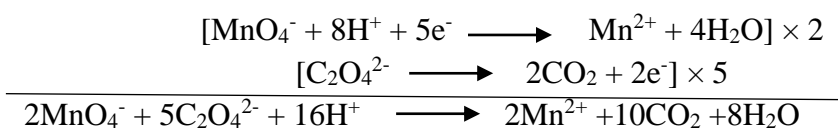
1. What are monoprotic, diprotic and triprotic acid?
2. Why is phenolphthalein used as a suitable indicator for this titration?
3. Discuss the precautions of the above experiment.



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Experiment 5: Standardization of KMnO_4 solution with standard $\text{Na}_2\text{C}_2\text{O}_4$ / $\text{H}_2\text{C}_2\text{O}_4$ solution.

An oxidation-reduction (redox) reaction is a type of chemical reaction that involves a transfer of electrons between two species in which the oxidation number of a molecule, atom, or ion is changed by gaining or losing an electron (s). Redox reactions are common and vital to some of the basic functions of life, including photosynthesis, respiration, combustion, and corrosion or rusting. Titration is a technique where a solution of known concentration is used to determine the concentration of an unknown solution.



Chemicals: KMnO_4 , $\text{H}_2\text{C}_2\text{O}_4$, H_2SO_4 .

Apparatus: Conical flask, Volumetric flask, Burette, Dropper, Funnel, Pipette filler, etc.

Procedure

a) Preparation of 250 mL 0.05 M $\text{H}_2\text{C}_2\text{O}_4$ solution

Weigh out of 1.575 gm of $\text{H}_2\text{C}_2\text{O}_4$ by following this formula, $w = \text{SMV}/1000$ (where, equivalent weight of $\text{H}_2\text{C}_2\text{O}_4$ is 126.000 g/mol) in 250 mL volumetric flask and add small amount of distilled water. Shake the flask to dissolve the solute. Finally add more water up to 250 mL mark.

b) Standardization of KMnO_4 solution

1. Fill out the burette with KMnO_4 solution.
2. Take 10 mL of 0.05 M standard $\text{H}_2\text{C}_2\text{O}_4$ solution in a conical flask.
3. Add 2 N H_2SO_4 to the $\text{H}_2\text{C}_2\text{O}_4$ sample.
4. Heat the acidified $\text{H}_2\text{C}_2\text{O}_4$ solution to about 60-75 °C.
5. Record the initial burette reading. Titrate the hot solution with the KMnO_4 solution until the appearance of a **faint pink color**. Record the final burette reading and calculate the volume of KMnO_4 used in the titration.
6. Repeat the titration two more times.

Experimental data: Standardization of KMnO_4 solution with standard $\text{H}_2\text{C}_2\text{O}_4$ solution

No. of observation	Volume of KMnO_4 solution (mL)			Average volume (mL)
	IBR	FBR	Difference	
1.				
2.				
3.				

Calculation

According to above reaction, 2 moles of KMnO_4 reacts with 5 moles of $\text{H}_2\text{C}_2\text{O}_4$,

Therefore,

$$\frac{M_{\text{KMnO}_4} \times V_{\text{KMnO}_4}}{M_{\text{H}_2\text{C}_2\text{O}_4} \times V_{\text{H}_2\text{C}_2\text{O}_4}} = \frac{2}{5}$$
$$\therefore M_{\text{KMnO}_4} = \frac{2}{5} \times \frac{10 \times 0.05}{V_{\text{KMnO}_4}} = \text{mol/L}$$

Percentage (%) of error = $\left(\frac{\text{Theo. value} - \text{exp. value}}{\text{Theo. value}} \right) \times 100$
= %

Result:

Practices

1. What is redox titration?

Ans: A **redox titration** is a titration of a reducing agent by an oxidizing agent or titration of an oxidizing agent by a reducing agent. Typically, this type of titration involves a redox indicator or a potentiometer.

2. Why is no indicator used in this titration?

Ans: As we do the titration we would reach the end point. The end point indicates that the standard solution was completely oxidized. And in this case, there will be permanent color change in the solution, white turns into faint pink. This faint pink color is due to the presence of a very little amount of extra KMnO_4 as there is no more standard solution left to oxidize it. Thus, KMnO_4 indicates that the standard solution was completely oxidized.

Thus, KMnO_4 acts as a self-indicator.

3. What happens if the titration is conducted at room temperature?

Ans: We heat the titration flask containing oxalic acid to about $60-70^\circ\text{C}$ and then titrate it against KMnO_4 . If the temperature is too low (below 55°C), the interaction between the oxalate and the potassium permanganate will move too slow. Above 60°C , oxalate acid begins to decompose, so it's important to stay in this range.

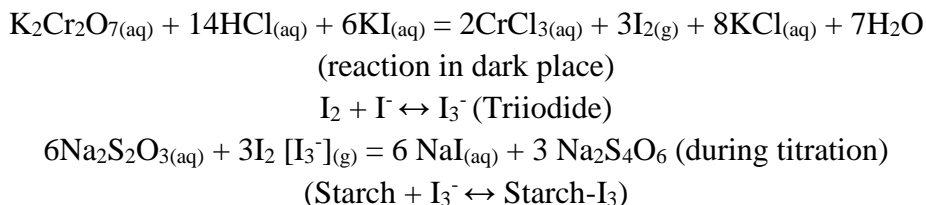
4. Discuss the precautions of the above experiment.



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Experiment 6: Standardization of $\text{Na}_2\text{S}_2\text{O}_3$ solution with a standard $\text{K}_2\text{Cr}_2\text{O}_7$ solution by iodometric titration.

The objective of this experiment is to determine the strength of $\text{Na}_2\text{S}_2\text{O}_3$, which is a very important industrial raw material. $\text{Na}_2\text{S}_2\text{O}_3$ solution is standardized against $\text{K}_2\text{Cr}_2\text{O}_7$ in presence of HCl . First, $\text{K}_2\text{Cr}_2\text{O}_7$ oxidizes the iodide ion in acidic medium to equivalent amount of iodine [Reaction (1)]. Then, the iodine formed in the reaction oxidizes $\text{Na}_2\text{S}_2\text{O}_3$ giving sodium tetrathionate ion [Reaction (2)] and the end point is detected by starch solution. Here the quantity of the iodine is equivalent in quantity to the oxidant being determined.



Chemicals: $\text{K}_2\text{Cr}_2\text{O}_7$, $\text{Na}_2\text{S}_2\text{O}_3$, NaHCO_3 , Conc. HCl , 10% KI , Starch solution, etc.

Apparatus: Conical flask, Volumetric flask, Burette, Dropper, Funnel, Pipette filler, etc.

Procedure

a) Preparation of 0.01 M 250 mL $\text{K}_2\text{Cr}_2\text{O}_7$ solution

Weigh out of 0.735 gm of $\text{K}_2\text{Cr}_2\text{O}_7$ by following this formula, $w = \text{SMV}/1000$ (where, molar mass of $\text{K}_2\text{Cr}_2\text{O}_7$ is 294 g/mol) in 250 mL volumetric flask and add small amount of distilled water. Shake the flask to dissolve the solute. Finally add more water up to 250 mL mark.

b) Standardization of $\text{Na}_2\text{S}_2\text{O}_3$ solution with a standard $\text{K}_2\text{Cr}_2\text{O}_7$ solution

1. Take the supplied $\text{Na}_2\text{S}_2\text{O}_3$ solution in the burette and note the initial reading.
2. Take 50 mL distilled water in a conical flask.
3. Add 1 g of NaHCO_3 and shake to dissolve the salt.
4. Add 3 mL of Conc. HCl slowly while gently rotating the flask to mix the liquid.
5. Add 10 mL of $\text{K}_2\text{Cr}_2\text{O}_7$ solution into the conical flask and mix the solution well.
6. Add 4 mL of 10% KI solution into conical flask and mix the solution well. Cover the flask immediately with a watch glass and allow the solution to stand for about 5 min in the dark (inside the desk, and color of the solution will be dark brown).
7. Take out the solution from the dark.

8. Titrate the liberated iodine with the $\text{Na}_2\text{S}_2\text{O}_3$ from the burette until the color fades to turn pale yellow. Now add 10 drops of starch solution to it and the color of the solution changes to deep violet.
9. Continue the addition of $\text{Na}_2\text{S}_2\text{O}_3$ from the burette until the color turns to light green or light blue. Note the burette reading and this is the end point.
10. Repeat the steps two to more times and take the average of the volume of the $\text{Na}_2\text{S}_2\text{O}_3$ solution.

Experimental data: Standardization of $\text{Na}_2\text{S}_2\text{O}_3$ solution with $\text{K}_2\text{Cr}_2\text{O}_7$ solution

No. of observation	Volume of $\text{Na}_2\text{S}_2\text{O}_3$ solution (mL)			Average volume (mL)
	IBR	FBR	Difference	
1.				
2.				
3.				

Calculation: From the above reaction,

1 mole $\text{K}_2\text{Cr}_2\text{O}_7 \equiv 3 \text{ mole I}_2 \equiv 6 \text{ mole Na}_2\text{S}_2\text{O}_3$

1 mole $\text{K}_2\text{Cr}_2\text{O}_7 \equiv 6 \text{ mole Na}_2\text{S}_2\text{O}_3$

Calculate the molarity of $\text{Na}_2\text{S}_2\text{O}_3$ solution from the following formula:

$$\frac{M \text{ K}_2\text{Cr}_2\text{O}_7 \times V \text{ K}_2\text{Cr}_2\text{O}_7}{M \text{ Na}_2\text{S}_2\text{O}_3 \times V \text{ Na}_2\text{S}_2\text{O}_3} = \frac{1}{6} \quad \text{or,} \quad M \text{ Na}_2\text{S}_2\text{O}_3 = \frac{6 \times M \text{ K}_2\text{Cr}_2\text{O}_7 \times V \text{ K}_2\text{Cr}_2\text{O}_7}{V \text{ Na}_2\text{S}_2\text{O}_3}$$

$$\text{Percentage (\%) of error} = \left(\frac{\text{Theo. value} - \text{exp. value}}{\text{Theo. value}} \right) \times 100$$

$$= \dots\dots\dots \%$$

Result: The strength of the supplied $\text{Na}_2\text{S}_2\text{O}_3$ solution was found to be.....

Practices

1) Why NaHCO₃ is added?

In aqueous solution, sodium thiosulfate dissociates into ions: $\text{NaS}_2\text{O}_3 \Rightarrow \text{Na}^+_{(\text{aq})} + (\text{S}_2\text{O}_3)^{2-}_{(\text{aq})}$. As the acid is titrated into the sodium thiosulfate solution, the acidity of solution increases. Thiosulfate is unstable under acidic pH conditions (abundance of H^+ ion), causing the thiosulfate to decompose into sulfur dioxide, elemental sulfur, and water: $(\text{S}_2\text{O}_3)^{2-}_{(\text{aq})} + \text{H}^+_{(\text{aq})} \Rightarrow \text{S (s)} + \text{SO}_2_{(\text{g})} + \text{H}_2\text{O}_{(\text{l})}$. This decomposition of thiosulfate will cause the whole experiment become pointless because we want the thiosulfate ions to react with the iodine in the acid, not with the H^+ ions. So, it must be stopped. Now, sodium bicarbonate in solution is alkaline. it will react with the H^+ ions. The abundance of hydroxide anions prevents the thiosulfate from encountering the positive charges that would trigger its decomposition. So, the acidity of the solution is reduced. So, sodium bicarbonate can be used either to stabilize thiosulfate, or to lower its reaction rate with anything else that it's been combined with.

2) Why do we put the flask in a dark area in iodine titration?

Iodine is light sensitive — you keep it in the dark so it's concentration will not decrease due to decomposition induced by light. And reaction 1 is slow, so it needs time 5 min.

3) Why do we use potassium iodide instead of iodine solution?

As a diatomic molecule with two identical atoms, I_2 is totally non-polar; it therefore dissolves well in organic solvents but not in water. Usually potassium salt (KI), are very soluble in water and are still oxidizing agents that behave similarly to I_2 . Adding KI to iodine in water rapidly converts the molecular iodine (I_2) to the much more soluble triiodide ion (I_3^-), pentaiodide ion (I_5^-), and higher polyiodides, and exist in equilibrium with free I_2 and I^- . It thus becomes possible to dissolve a relatively large amount of I_2 in a solution of some iodide salt (most commonly KI).

4) What do you mean by iodometry and iodimetry?

Thiosulfate titration can be an iodometric procedure. Iodometry involves the use of iodide (the iodine anion) to indicate the presence of an oxidizing or reducing agent. Because elemental iodine is highly electronegative, it will tend to draw one of the two extra electrons in the thiosulfate to itself. Iodometry describes the type of titration to determine the concentration of oxidizing agents through an indirect process involving iodine as the intermediary. In the presence of iodine, the thiosulphate ions oxidize quantitatively to the tetrathionate ions. An iodometric titration is used to determine an oxidant which is difficult to observe, evaporates rapidly. [When an analyte that is a reducing agent is titrated directly with a standard iodine solution, the method is called 'iodimetry'.]

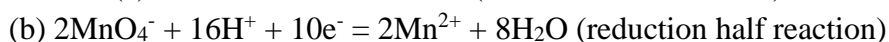
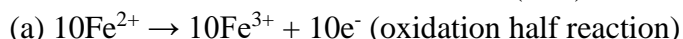
5) Discuss the precautions of the above experiment.



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Experiment 7: Estimation of ferrous ion (Fe^{2+}) by a standard KMnO_4 solution.

Determination of the concentration of ferrous and ferric ions is essential for understanding the composition of a sample. This is particularly important in analytical chemistry and industries where precise knowledge of elemental concentrations is crucial. The reaction as follows,



Chemicals: $\text{H}_2\text{C}_2\text{O}_4$, KMnO_4 , $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$, H_2SO_4 , etc.

Apparatus: Conical flask, Volumetric flask, Burette, Dropper, Funnel, Pipette filler, etc.

Procedure:

Solution Preparation:

0.02 M KMnO_4 solution:

Mol. Mass of KMnO_4 158.03 g/mol

Dissolve 0.316 gm solid in 100 mL to make approx. 0.02 M KMnO_4 solution.

Dissolve 1.58 gm solid in 500 mL to make approx. 0.02 M KMnO_4 solution.

0.1M Ferrous sulphate solution:

Sulfuric acid, H_2SO_4 [2 N], (conc.98%, sp.gr. 1.84)

1 mL conc. H_2SO_4 contain $(0.98 \times 1.84) = 1.8032$ gm H_2SO_4

1000 mL, ,, ,, = $1.8032 \times 1000 = 1803.2$ gm

49 gm of H_2SO_4 in 1000 mL make solution = 1 N

1766.4 gm ,, ,, in 1000 mL ,, ,, = 36 N

$$V_{\text{conc. H}_2\text{SO}_4} \times N_{\text{conc. H}_2\text{SO}_4} = V_{\text{dil. H}_2\text{SO}_4} \times N_{\text{dil. H}_2\text{SO}_4}$$

$$V_{\text{conc. H}_2\text{SO}_4} \times 36 \text{ N} = 1000 \text{ mL} \times 2 \text{ N}$$

$$V_{\text{conc. H}_2\text{SO}_4} = 55.56 \text{ mL} \approx 55 \text{ mL}$$

Working procedure:

a) **Standardization of KMnO_4 solution by standard $\text{H}_2\text{C}_2\text{O}_4$ solution as expt. 5.**

b) **Determination of Fe^{2+} in a supplied solution by the standard KMnO_4 solution.**

1. Pipette out 10 mL of the Ferrous sulfate heptahydrate solution in a conical flask.
2. Titrate the solution with the standard KMnO_4 solution.
3. The end point is indicated by the first appearance of permanent light pink color as expt. 5.
4. Repeat the titration at least 3 times.

Experimental data: Titration of Ferrous sulfate solution with KMnO_4 solution

No. of observation	Volume of KMnO_4 solution (mL)			Average volume (mL)
	IBR	FBR	Difference	
1.				
2.				
3.				

Calculation

According to reaction, 2 moles $\text{KMnO}_4 \equiv 10$ moles FeSO_4 , i.e. 1 mol $\text{KMnO}_4 \equiv 5$ moles FeSO_4

- 1 mL 1 N $\text{KMnO}_4 \equiv 0.05584$ gm of Fe^{2+} [From reaction, 1000 mL 1 M $\text{KMnO}_4 \equiv 5$ M FeSO_4]
- 1000 mL 158.030 gm $\text{KMnO}_4 \equiv 5 \times 55.84$ gm of Fe^{2+}
- 1000 mL 31.610 gm $\text{KMnO}_4 \equiv 55.84$ gm of Fe^{2+}
- 1000 mL 0.02 M $\text{KMnO}_4 \equiv 55.84$ gm of Fe^{2+}
- Amount of Fe^{2+} in 10 mL = $(0.05584 \times V \times S)$ gm
- Amount of Fe^{2+} in 500 mL = $(0.05584 \times V \times S \times 50)$ gm (**experimental value**)

[V = volume of KMnO_4 solution required to titrate 10 mL FeSO_4 solution and S= strength of KMnO_4 solution = 0.02 M]

Theoretical value calculation: Known value* of Iron (Fe^{2+} ions) in 500 mL of iron solution = $55.84 \text{ g/mol} \times (0.1 \text{ M Ferrous sulfate} \times 0.5 \text{ L}) = 2.79 \text{ gm}$

*Known value calculation is not volume dependent.

$$\text{Percentage (\%) of Error:} = \left(\frac{\text{Known value} - \text{Experimental value}}{\text{Known value}} \right) \times 100$$

$$= \dots\dots\dots \%$$

Result: Amount of Fe^{2+} ions in 500 mL of supplied iron solution is gm.

Practices

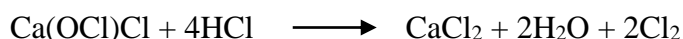
1. What do you mean by redox titration?
2. How will you prepare 0.003 M 350 mL KMnO_4 solution?
3. Write down the formula of green vitriol?
4. What do you mean by oxidant and reductant?
5. Calculate the equivalent weight of KMnO_4 ?
6. Discuss the precautions of the above experiment.



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Experiment 8: Determination of the available chlorine present in bleaching powder.

Bleaching powder is used as a bleaching agent and as a disinfectant. The main constituent of bleaching powder is calcium hypochlorite $[\text{Ca}(\text{OCl})\text{Cl}]$ which supplies chlorine $[\text{Cl}_2]$ with dilute acids.



So, the available chlorine is defined as the percentage of chlorine made available by bleaching powder when treated with dilute acids. The available chlorine present in bleaching powder sample is determined iodometrically by treating its solution with an excess of potassium iodide solution in the acidic medium.



The liberated iodine (I_2) is treated with sodium thiosulphate ($\text{Na}_2\text{S}_2\text{O}_3$) solution using freshly prepared starch solution as indicator to be added near the end point.



Chemicals: $\text{Ca}(\text{OCl})\text{Cl}$, $\text{Na}_2\text{S}_2\text{O}_3$, 10% KI, CH_3COOH , freshly prepared starch solution.

Apparatus: Digital Balance, Burette, Conical flask, Measuring flask, Funnel, Glass rod, Beakers.

Procedure:

1. Fill up the burette with standard $\text{Na}_2\text{S}_2\text{O}_3$ solution.
2. 1.0 g of bleaching powder is accurately weighed and transferred to a 100 mL volumetric flask. Then the solution is made up to the volume. The flask is shaken well for uniform concentration.
3. Pipette out 10 mL of the solution is taken into a conical flask.
4. Add 10 mL of CH_3COOH into it.
5. Then add 5 mL of 10% potassium iodide (KI) solution.
6. Cover the flask immediately with a watch glass and allow the solution to stand for about 5 min in the dark (inside the desk, and color of the solution will be dark brown).
7. This solution [which contains liberated iodine (I_2)] is titrated with $\text{Na}_2\text{S}_2\text{O}_3$ solution until the dark brown color changes to pale yellow.
8. To this add 2 mL of freshly prepared starch solution as indicator, so the color of the solution turns into blue. Continue the titration till the disappearance of blue color of the solution and note down the volume of the titrant used. The titration is repeated until a concordant volume is obtained.

Experimental data: Titration of bleaching powder solution with $\text{Na}_2\text{S}_2\text{O}_3$ solution.

No. of observation	Volume of KMnO_4 solution (mL)			Average volume (mL)
	IBR	FBR	Difference	
1.				
2.				
3.				

Calculation

- Normality of standard $\text{Na}_2\text{S}_2\text{O}_3$ solution, $N_1 = 1/10 = 0.1 \text{ N}$
- Volume of bleaching powder solution, $V_2 = 10 \text{ mL}$
- Normality of given bleaching powder solution, $N_2 = \frac{N_1 \times V_1}{V_2} = \dots\dots\dots \text{ N}$
- Amount of available chlorine, $m_{\text{Cl}} = N_2 \times \text{Eq. wt. of chlorine} = N_2 \times 35.45 \text{ g/L} = \dots\dots\dots \text{ g/L}$
- Amount of available chlorine present in 100 mL of the solution $= \frac{m_{\text{Cl}} \times 100}{1000} = \dots\dots\dots \text{ g}$
- The percentage of available chlorine present in the given sample of bleaching powder (BP)

$$= \frac{\text{Amount of Cl}}{\text{Weight of BP}} \times 100 = \frac{\dots\dots\dots}{\dots\dots\dots} \times 100 = \dots\dots\dots \%$$

Result: The percentage of available chlorine in the bleaching powder is..... %
 (a good quality bleaching powder contains 25-30 % available chlorine to act as a disinfectant).

Practices

1. Write the molecular formula and molar mass of bleaching powder.
2. Why is bleaching powder used?
3. Discuss the precautions of the above experiment.



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Experiment 9: Preparation of a buffer solution of different pH.

The objective of this experiment is to make the students learn the process and calculation of preparing buffer solutions (of a definite pH). A buffer solution is one that is resistant to change in pH when small amounts of strong acid or base are added. For example, when 0.01 mole of strong acid or base are added to distilled water, the pH drops to 2 with the acid and rises to 12 with the base. If the same amount of acid or base is added to an acetic acid–sodium acetate buffer, the pH may only change a fraction of a unit. Two species are required in a buffer solution. One can react with OH^- and the other will react with H_3O^+ . The two species must not react with each other. Many buffers are prepared by combining a weak acid and its conjugate (acetic acid and sodium acetate) or a weak base and its conjugate (ammonia and ammonium chloride). In general, the pH range in which a buffer solution is effective is \pm one pH unit on either side of the pK_a . The Henderson–Hasselbalch provides the information needed to prepare a buffer.

$$\text{pH} = \text{pK}_a + \frac{[\text{Conjugate base}]}{[\text{Conjugate acid}]}$$

Chemicals: Acetic acid, sodium acetate, distilled water.

Apparatus: Analytical balance, weighing paper, spatula, distilled water, pH meter, beaker, filter paper & funnel, measuring cylinder, glass rod.

Procedure

1. Clean all glassware using normal water & then distilled water.
2. Take two 250 mL beakers and add 100 mL of distilled water to both beakers.
3. Calculate the amount of acetic acid and sodium acetate required to prepare a buffer of pH 5 using The Henderson–Hasselbalch equation. Use 0.1 M acetic acid and 0.3 M acetic acid to prepare 100 mL buffer of pH 5. Calculate the amount of sodium acetate required for both solutions (pK_a of acetic acid is 4.75).
4. Now measure the calculated amount of acetic acid using analytical balance and add it in the two beakers and stir using glass rod.
5. When all the acetic acid is dissolved, add the calculated amount of sodium acetate (required to prepare buffer of pH 5) in the same solution with continuous stirring after weighing.
6. Measure the pH of the resulting solutions using a calibrated pH meter and record all data in the observation table.

Observation table

No.	Amount of acetic acid in 100 mL	Amount of sodium acetate in 100 mL	pH of the resulting buffer (using pH meter)
1.			
2.			
3.			

Result: pH of the resulting buffer solutions is:

- 1.
- 2.
- 3.

Practices

1. What do you mean by pH and pOH?
2. Explain buffer solution with example?
3. What do you mean by conjugate acid and conjugate base?
4. Blood is a buffer system-explain.
5. Discuss the precautions of the above experiment.



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Experiment 10: Solubility test for unknown inorganic compounds.

The solubility of a solute is the maximum quantity of solute that can dissolve in a certain quantity of solvent or quantity of solution at a specified temperature. There are a few patterns in the data obtained from measuring the solubility of different salts. These patterns form the basis for the rules outlined in the table below, which can guide predictions of whether a given salt will dissolve in water. These rules are based on the following definitions of the terms soluble, insoluble, and slightly soluble. A salt is soluble if it dissolves in water to give a solution with a concentration of at least 0.1 moles per liter at room temperature. A salt is insoluble if the concentration of an aqueous solution is less than 0.001 M at room-temperature. Slightly soluble salts give solutions that fall between these extremes.

Solubility rules for Ionic compounds in water:

Soluble salts

1. The Na^+ , K^+ , and NH_4^+ ions form soluble salts. Thus, NaCl , KNO_3 , $(\text{NH}_4)_2\text{SO}_4$, Na_2S , and $(\text{NH}_4)_2\text{CO}_3$ are soluble.
2. The nitrate (NO_3^-) ion forms soluble salts. Thus, $\text{Cu}(\text{NO}_3)_2$ and $\text{Fe}(\text{NO}_3)_3$ are soluble.
3. The chloride (Cl^-), bromide (Br^-), and iodide (I^-) ions generally form soluble salts. Exceptions to this rule include salts of the Pb^{2+} , Hg_2^{2+} , Ag^+ , and Cu^+ ions. ZnCl_2 is soluble, but CuBr is not.
4. The sulfate (SO_4^{2-}) ion generally forms soluble salts. Exceptions include BaSO_4 , SrSO_4 , and PbSO_4 , which are insoluble, and Ag_2SO_4 , CaSO_4 , and Hg_2SO_4 , which are slightly soluble.

Insoluble salts

1. Sulfides (S^{2-}) are usually insoluble. Exceptions include Na_2S , K_2S , $(\text{NH}_4)_2\text{S}$, MgS , CaS , SrS , and BaS .
2. Oxides (O^{2-}) are usually insoluble. Exceptions include Na_2O , K_2O , SrO , and BaO , which are soluble, and CaO , which is slightly soluble.
3. Hydroxides (OH^-) are usually insoluble. Exceptions include NaOH , KOH , $\text{Sr}(\text{OH})_2$, and $\text{Ba}(\text{OH})_2$, which are soluble, and $\text{Ca}(\text{OH})_2$, which is slightly soluble.
4. Chromates (CrO_4^{2-}) are usually insoluble. Exceptions include Na_2CrO_4 , K_2CrO_4 , $(\text{NH}_4)_2\text{CrO}_4$, and MgCrO_4 .
5. Phosphates (PO_4^{3-}) and carbonates (CO_3^{2-}) are usually insoluble. Exceptions include salts of the Na^+ , K^+ , and NH_4^+ ions.

Procedure

Amount of material required: It is convenient to employ an arbitrary ratio of 0.1 g of solid or 0.2 mL of liquid for 3.0 mL of solvent.

Test for soluble/insoluble salts in distilled water.

Sample	Room Temperature	Hot water (50-60) °C

Practices

1. What do you mean by solubility, solution?
2. What is salt?
3. What do you mean by neutralization reaction?
4. Discuss the precautions of the above experiment.



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Experiment 11: Qualitative analysis for organic functional groups (-OH, -COOH, -CHO, >CO, -NH₂, -NO₂).

Since carbon and hydrogen are invariably present in organic compounds, it is not necessary to test for these elements. In fact, if the tests for other elements e.g. nitrogen, sulfur, and halogens are negative, it is assumed that the compound belongs to this category. Nevertheless, the presence of carbon and hydrogen may be ascertained by appropriate tests described under detection of elements. For detection of oxygen there is no direct test but if the substance is readily soluble in water, then the presence of oxygen in the compound is almost certain. The tests for various functional groups should be performed in the following order only if the compound responds positively to these tests, then the presence of that group is certain.

Chemicals: 2, 4-dinitrophenyl hydrazine, AgNO₃, NH₄OH, NaOH, Tollen's reagent, Fehling solution A, Fehling Solution B, Sodium bicarbonate, Litmus paper, Ferric chloride Solⁿ. Acetone, Formaldehyde, Acetaldehyde, Benzaldehyde, Glucose, Metallic Sodium, Methyl alcohol, Benzoic acid, Nitro aniline, Picric acid, Phenol, etc., HCl, NaNO₂, etc.

Apparatus: Test-tube, test-tube holder, wash bottle, pipette, spatula etc.

(A) Tests for Carboxylic acid group (-COOH)

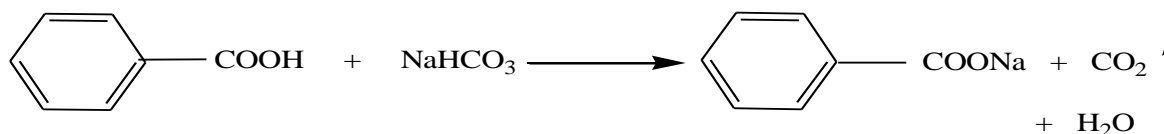
(i) Bicarbonate Test

Procedure

1. Take 1ml saturated solution of sodium bicarbonate in a test tube.
2. Warm the solution.
3. Then add a pinch of the organic compound (Acetic acid/Formic acid).

An **effervescence** indicates the presence of a **-COOH** group.

For example,



(ii) Litmus paper test

Procedure

1. Take a dilute aqueous solution of the organic compound.
2. Add Litmus paper.

If the litmus paper turns **red**, the organic compound may be **carboxylic acid**.

(B) Test for phenolic (-OH) group

Ferric chloride test

Procedure

1. Dissolve 0.05 gm of the compound.
2. Add 1 ml of water.
3. Add just 1 drop of ferric chloride solution.

The appearance of a blue, green, red, or violet coloration indicates the presence of phenolic group.



Observation: Violet coloration indicates the-----of **phenolic** group.

Result: Phenolic group (-OH) is present in that compound.

(C) Test for carbonyl group (aldehydes and ketones)

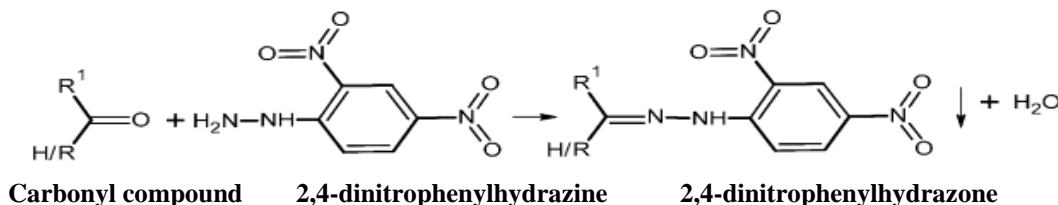
2,4-dinitrophenyl hydrazine test (2,4-DNPH)

Aldehydes and Ketones contain the carbonyl group, hence a general test for carbonyl compounds will immediately identify both classes of compounds. This is the most common and preferred test for aldehyde and ketone detection. 2,4-dinitrophenyl hydrazine gives sparingly soluble dinitrophenylhydrazone with carbonyl compound.

Procedure

1. Dissolve 0.1 gm of carbonyl compound in a test tube.
2. Add 3.0 mL of 2,4-dinitrophenyl hydrazine reagent and shake.
3. The mixture is allowed to stand at room temperature.

The appearance of **orange/yellow** color indicates the **carbonyl** group.



Observation: Crystalline precipitate indicates the presence of a carbonyl compound (-CHO/>CO).

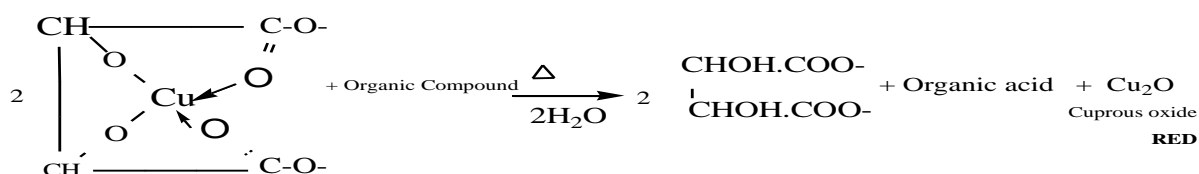
a) Tests for aldehydes (-CHO)

(1) Fehling solution test

Procedure

1. In a test tube, mix 1 mL each of Fehling A and Fehling B solutions when a deep blue color appears.
2. add 0.1 gm of substance into this.
3. Boil for 5 minutes.
4. The blue color of Fehling's solution slowly fades away and a **reddish-brown** precipitate of Cu_2O is obtained indicates the presence of **aldehydic** group.

Reaction



Observation: A **reddish-brown** precipitate indicates that the aldehydic (**-CHO**) group is present.

Result: -CHO group is present in that compound.

(2) Tollen's test

Procedure

1. Take 1 mL Tollen's reagent in a test tube (AgNO_3).
2. Then add 1 mL of NH_4OH Solution.
3. Add 0.1 gm of substance.
4. Keep the test tube in boiling water without disturbing it for 5 minutes.

A shining **silver mirror** deposits on the walls of test tube indicating the presence of **-CHO** group.

Reaction



Observation: A shining silver mirror indicates the presence of **-CHO** group.

Result: **-CHO** group is present in that compound.

b) Tests for ketones (>CO)

Method 1: If the tests for aldehydes are negative, perform the following tests for ketones.

Procedure

1. To take a solution of 0.5 gm of substance.
2. Add 5 mL dilute HCl acid.
3. Add 2 mL of solution of 2,4-dinitrophenyl hydrazine.
4. Cool and allow to stand for 2 minutes.

The **yellow, orange, or red** colored crystalline precipitate indicates the presence of **ketonic** group.

Observation: Colored crystalline precipitate indicates the presence of >CO group.

Result: >CO group is present in that compound.

Method 2: Sodium test

Procedure

1. Take 1 ml of the supplied alcohol in a test tube.
2. Add a small piece of dry sodium metal.

A **vigorous effervescence** indicates the presence of **hydroxyl group** in the organic compound.

Observation: Vigorous effervescence indicates the presence of hydroxyl (-OH) group.

Result: -OH group is present in that compound.

(D) Tests for amino (–NH₂) group

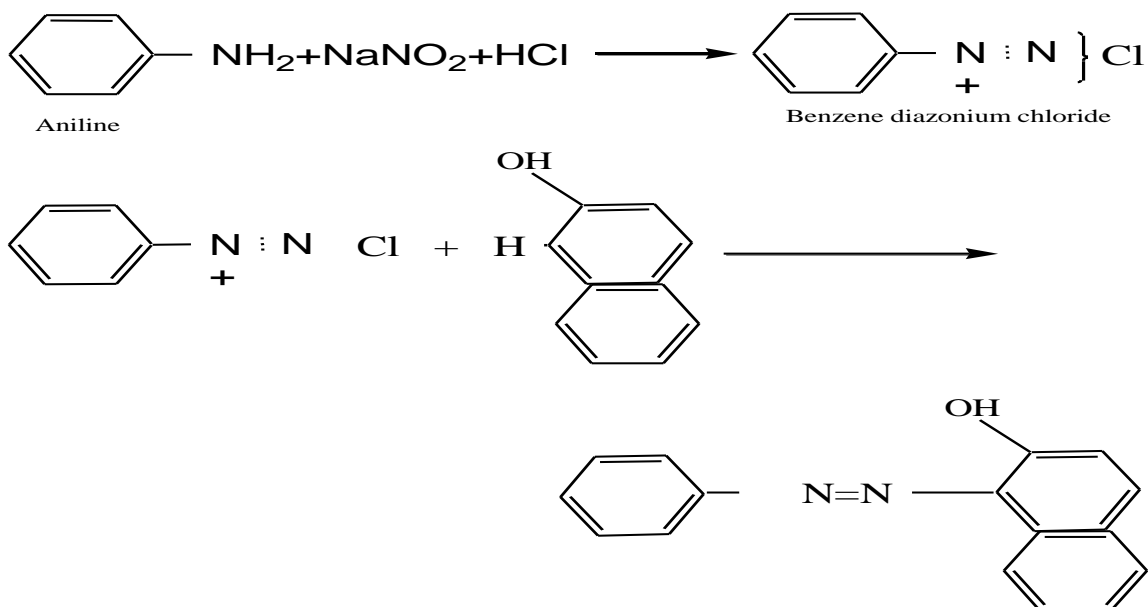
(i) Azo-dye test

Procedure

1. Dissolve 0.25 gm of the supplied organic compound in 1 mL distilled water.
2. Add a Slight excess of dil. HCl (~1.5mL)
3. Cool in ice water
4. Add sodium nitrite dropwise till there is a slight excess of the nitrite.
5. Add 1 mL of a strong solution of sodium acetate.
6. Pour slowly a solution of β-naphthol in NaOH.

If a **red or orange** precipitate of azo dye is formed, the compound is a **primary aromatic amine**.

Reaction



Observation: A red or orange precipitate is formed; the compound is primary aromatic amine.

Result: Primary aromatic amine is present in that compound.

(E) Test for Nitro ($-\text{NO}_2$) group

Mulliken and Barker's reaction:

Procedure

1. Dissolve a little amount of the organic compound in 1mL of distilled H_2O .
2. Add 3 mL of diluted alcohol.
3. Add six drops of CaCl_2 solution.
4. Add a pinch of Zinc dust.
5. Then heat to a boil and set aside for 5 minutes.
6. Add Tollen's reagent (few drops).

A **black** deposit of **silver** is observed.

Observation: A black deposit of silver indicates the presence of $-\text{NO}_2$ group.

Result: $-\text{NO}_2$ group is present in that compound.



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Experiment 12: Test for Carbohydrate/Reducing sugar detection.

Molisch's test (general test for carbohydrates)

A color test for sugar, which condenses with alpha-naphthol or thymol in the presence of strong sulfuric acid, which converts the sugar to furfural derivatives.

Procedure

1. Place 0.5 mg of substance in a test tube
2. Add 1mL of water.
3. Mix 2 drops of 10% solution of 2-naphthol (**Molisch's reagent**)
4. Allow 1.0 mL of concentrated H_2SO_4 to flow down the side of the inclined tube.
5. Forming a layer of acid beneath the sugar.

A **reddish violet** zone appears at the junction between the two liquids.

Observation: If carbohydrate is present, a red ring appears at the common surface of the liquids. Acid forms a layer beneath the aqueous solution without mixing with it.

Conformation test: The color quickly changes on standing or shaking of the experimented test-tube and a dark purple solution being formed. Shake and allow the mixture to stand for two minutes. Dilute with 5.0 mL of water. A dull-violet precipitate will appear immediately.

Result: Carbohydrates are -----.

Barfoed's test (test for monosaccharide)

Procedure

1. Pour 1.0 mL of dilute solution of carbohydrate in a test tube.
2. Add 1.0 mL of Barfoed's reagent.
3. Heat the test tube in a beaker of boiling water/over burner.

Remarks: If **red** ppt is formed within 2 minutes, a monosaccharide is present. The ppt is due to the formation of copper (I) oxide.

Result: Supplied compound-----.

Benedict's test

Modification of Fehling's test (positive test for reducing sugar)

Copper sulfate of Benedict's qualitative solution is reduced by reducing substances on boiling to form the colored precipitate of cuprous oxide. The light green, green, yellow and brick red precipitates of cuprous oxide depend on the concentration of reducing sugar.

Procedure

1. Place 1 mL of a 2% solution of carbohydrate in a test tube
2. Add 5.0 mL of Benedict's solution.
3. Boil for 2 minutes.
4. Allow cooling spontaneously.

Remarks

Light green precipitate	0.1 to 0.5 of reducing sugars
Green precipitate	0.5 to 1.0% of reducing sugars
Yellow precipitate	1 to 2% of reducing sugars
Brick Red precipitate	Above 2% of reducing sugars
Clear solutions	No reducing sugar

Result: The supplied sample contains.....



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Experiment 13: Qualitative analysis of inorganic compounds.

Qualitative analysis deals with the identification of elements or grouping of elements present in a sample. It is the determination of non-numerical information about a chemical species, a reaction, etc. An inorganic compound is typically a chemical compound that lacks carbon-hydrogen bonds. The qualitative tests are used for the identification of chemicals which are important in inorganic chemistry.

Apparatus

1. Test tube	4. Litmus paper
2. Beaker	5. Watch glass
3. Bunsen burner	6. Stirring Rod

Chemicals

1. Dil. CH_3COOH	8. 2 N CH_3COOH
2. $(\text{NH}_4)_2\text{C}_2\text{O}_4$ solution	9. 0.1 N $\text{Ba}(\text{OH})_2$
3. AgNO_3 solution	10. HNO_3
4. KI solution	11. H_2SO_4
5. Dil. NH_3 solution	12. FeSO_4 solution
6. NH_4Cl solution	13. Dil. HCl

Supplied compound

- | | |
|---------|---------|
| 1. | 3. |
| 2. | 4. |

Procedure:

- Physical examination: Crystalline/Amorphous
- Color (if any):
- Odor (if any):
- Test for cation.

1) Flame test: The flame test is used to visually determine the identity of an unknown metal of an ionic salt based on the characteristic color the salt turns the flame of a Bunsen burner. First, you need a clean wire loop! Platinum or nickel-chromium loops are most common. They may be cleaned by dipping in hydrochloric or nitric acid, followed by rinsing with distilled or deionized water. Test the cleanliness of the loop by inserting it into a Bunsen burner flame. If a burst of color is produced, the loop is not sufficiently clean. Ideally, a separate loop is used for each sample to be tested, but a loop may be carefully cleaned between tests. The clean loop is dipped in either a powder or solution of an ionic (metal) salt. The loop with sample is placed in the clear or blue part of the flame and the resulting color is observed.

Color	Metal
Red	Carmine: Lithium compounds. Masked by barium or sodium. Scarlet or Crimson: Strontium compounds. Masked by barium. Yellow-red: Calcium compounds. Masked by barium.
Yellow	Sodium compounds, even in trace amounts. A yellow flame is not indicative of sodium unless it persists and is not intensified by addition of 1% NaCl to the dry compound.
White	White-green: Zinc
Green	Emerald: Copper compounds, other than halides. Thallium. Blue-green: Phosphates, when moistened with H_2SO_4 or B_2O_3 . Faint-green: Antimony and NH_4 compounds. Yellow green: Barium, molybdenum.
Blue	Azure: Lead, selenium, bismuth, CuCl_2 and other copper compounds moistened with hydrochloric acid. Light blue: Arsenic and some of its compounds. Greenish Blue: CuBr_2 , antimony
Violet	Potassium compounds other than borates, phosphates, and silicates. Masked by sodium or lithium. Purple red: Potassium, rubidium, and/or cesium in the presence of sodium when viewed through a blue glass.

Na^+ ion: The most common method of identification of Na^+ is the flame test. Sodium imparts a brilliant, long lasting, yellow flame that masks colors from other ions. The test may be performed on a small sample of the unknown treated with concentrated HCl or a few drops of solution unknown treated with concentrated HCl. The flame should be bright, and it should last as long as that of 0.1 M NaCl. Sodium is a common impurity, and traces will be found in almost any unknown. You must learn to distinguish between an unknown that has sodium ion as the cation and an unknown that has sodium ion as an impurity.

K^+ ion: The most common method of identification of K^+ is the flame test. The test may be performed on a small sample of the unknown treated with concentrated HCl or a few drops of solution unknown treated with concentrated HCl. The violet flame is not intense, but it is clearly visible in the absence of sodium ions. Cobalt glass filters yellow light from sodium impurities and allows the violet flame to be seen. Do not confuse the glowing wire with the potassium flame.

2) Confirmation test for cations

Chemical test	Observation
Ca²⁺: To a solution of the substance being examined add a few drops of a (NH ₄) ₂ C ₂ O ₄ . $(NH_4)_2C_2O_4 + CaCl_2 = CaC_2O_4 + 2NH_4Cl$	A white precipitate is obtained. That is only sparingly soluble in dilute CH ₃ COOH but soluble in HCl.
Cu²⁺: The solution of substance, add dil. ammonia. $Cu(OH)_{2(s)} + 4NH_{3(aq)} + 2H_2O_{(l)} \rightarrow [Cu(NH_3)_4(H_2O)_2]^{2+}_{(aq)} + 2OH^-$	A greenish blue precipitate is formed which dissolved in excess amount of reagent forming deep bluish solution.
Pb²⁺: The solution of substance, add 2 mL of water and 0.2 mL of potassium iodide. $2KI + PbSO_4 = PbI_2 + K_2SO_4$	A yellow precipitate forms. Heat to boil for one or two minutes and allow to cool; the precipitate is formed as glistening, yellow plates.
Mg²⁺: Dissolved about 0.015 g of the substance being examined in 2 mL water or use 2 mL of the prescribed solution. Add 1 mL of dil. NH ₄ OH; a white precipitate form that is redissolved by adding 1 mL of 2 M NH ₄ Cl. Add 1 mL of 0.25 M Na ₂ HPO ₄ .	A white crystalline precipitate is formed.
Al³⁺: To a solution of the substance, add NaOH. Then add a few drops of NH ₄ OH. To sol ⁿ . add 1 or 2 drops of litmus soln. Then dil. HCl until acid (if not already); then add NH ₄ OH until just alkaline.	Colorless precipitate of Al(OH) ₃ is formed, which is forming white gelatinous precipitate. Blue lake obtained (hydroxide ppt. adsorbing dye to leave soln. almost colorless).
Zn²⁺: 1. Zinc forms one of the few insoluble white sulfides. It is precipitated from a solution of the ammonia complex. Small traces of cations that form dark colored sulfides will obviously interfere. Add an excess of 3 M NH ₃ to the test solution, so that any zinc present is in the form of [Zn(NH ₃) ₄] ²⁺ . Then add a few drops of 0.1 M Na ₂ S solution. 2. To sol ⁿ , add 1 or 2 drops of NH ₄ OH. Evaporate the suspension in an evaporating basin and ignite (play flam on to residue direct) 3. To sol ⁿ , add NH ₄ Cl then NH ₄ OH pass H ₂ S.	A white precipitate indicates the presence of Zn ²⁺ . White precipitate of Zn(OH) ₂ gives ZnO which is yellow when hot turning white on cooling. White (often dirty white) ppt. of ZnS.
Ba²⁺: To sol ⁿ , add K ₂ CrO ₄ sol ⁿ .	Yellow ppt. of BaCrO ₄ confirms Ba ²⁺ .

NH₄⁺ ion test

Chemical test	Observation	Inference
NH₄⁺ ion: 1. Take a small amount of the material to be tested and place it in a 50 mL beaker. Add 6 M NaOH and smell cautiously. The odor of ammonia indicates the presence of ammonium ions. If you do not smell ammonia, warm the beaker, and again smell the emitted vapors. The liberated ammonia will also change the color of a moistened strip of red litmus paper held at the entrance of the test tube. $\text{NH}_4^+_{(\text{aq})} + \text{OH}^-_{(\text{aq})} \rightarrow \text{NH}_{3(\text{g})} + \text{H}_2\text{O}$	1.Red litmus paper will form blue color	NH ₄ ⁺ present
2. Used to detect ammonia to solution add 1 or 2 drops of Nessler's reagent.	2) Then solution gives a yellow coloration, it indicates the presence of ammonia: at higher concentrations, a brown precipitate may form. $\text{NH}_4^+ + 2[\text{HgI}_4]^{2-} + 4\text{OH}^- \rightarrow \text{HgO} \cdot \text{Hg}(\text{NH}_2)\text{I} + 7\text{I}^- + 3\text{H}_2\text{O}$	NH ₄ ⁺ present

Difference between Fe²⁺ and Fe³⁺ ion:

Reagent	Fe ²⁺ ion	Fe ³⁺ ion
1) NaOH Solution	First white or whitish green color form then it will form permanently radish-brown color	It will be formed permanently radish-brown color
2) K ₄ [Fe(CN) ₆] solution	White or pale blue ppt form	Blue color ppt. form.

Conformation tests for anions

Chemical tests	Observation
<p>CO₃²⁻ ion: Take 1 mL dilute HCl in test tube. Then add substance. Dilute HCl gives vigorous effervescence with carbonates, evolving CO₂:</p> $\text{CO}_3^{2-}(\text{aq or s}) + 2\text{H}^+(\text{aq}) \rightarrow \text{H}_2\text{O}(\text{l}) + \text{CO}_2(\text{g})$	<p>The colorless, odorless CO₂ can be identified by bubbling it through a saturated solution of Ba(OH)₂, with which it forms a white precipitate of BaCO₃.</p> $\text{CO}_2(\text{g}) + \text{Ba}^{2+}(\text{aq}) + 2\text{OH}^-(\text{aq}) \rightarrow \text{BaCO}_3(\text{s}) + \text{H}_2\text{O}(\text{l})$
<p>X⁻ ion: Dissolved in 2 mL of water a quantity of the substance being examined equivalent to about 2 mg of halide ion or use 2 mL of the prescribed solution. Acidify with dil. HNO₃ and add 0.5 mL of silver nitrate solution. Shake and allow to stand.</p>	<p>A curdy white ppt. soluble in NH₄OH indicates Cl⁻ in the inorganic compound. A light-yellow ppt. sparingly soluble in NH₄OH indicates Br⁻ in the inorganic compound. A pale-yellow ppt. insoluble in NH₄OH indicates I⁻ in the inorganic compound.</p> $\text{Ag}^+(\text{aq}) + \text{Cl}^-(\text{aq}) \rightarrow \text{AgCl}(\text{s})$ $\text{AgCl}(\text{s}) + 2\text{NH}_3(\text{aq}) \rightarrow \text{Ag}(\text{NH}_3)_2^+(\text{aq}) + \text{Cl}^-(\text{aq})$ $\text{Ag}(\text{NH}_3)_2^+(\text{aq}) + \text{Cl}^-(\text{aq}) + 2\text{H}^+(\text{aq}) \rightarrow \text{AgCl}(\text{s}) + 2\text{NH}_4^+(\text{aq})$
<p>NO₃⁻ ion: Dissolved 15 mg of the substance being examined in 0.5 mL of water, add cautiously 1 ml of H₂SO₄, mix, and cool. Incline the tube and carefully add, without mixing, 0.5 mL of ferrous sulphate solution. The most notable feature of the chemistry of the nitrate ion is its oxidizing ability as illustrated by the following reactions:</p> $3\text{Fe}^{2+}(\text{aq}) + 4\text{H}^+(\text{aq}) + \text{NO}_3^-(\text{aq}) \rightarrow \text{NO}(\text{g}) + 2\text{H}_2\text{O} + 3\text{Fe}^{3+}(\text{aq})$ <p>In the last reaction the nitrogen oxide reacts with excess Fe²⁺ to give the brown complex ion Fe(NO)²⁺. It is the formation of this brown complex that is used to identify NO₃⁻ (called the brown ring test).</p>	<p>A brown color ring of NO₃⁻ is formed at the interface of the two liquids.</p>
<p>SO₄²⁻ ion: Use 5 mL of the prescribed solution. Add 1 mL of dil. HCl and 1 mL of BaCl₂ solution.</p> $\text{Ba}^{2+}(\text{aq}) + \text{SO}_4^{2-}(\text{aq}) \rightarrow \text{BaSO}_4(\text{s})$	<p>A white precipitate of SO₄²⁻ is formed.</p>
<p>S²⁻ ion: To solution add (CH₃COO)₂Pb aq.</p> $\text{Na}_2\text{S} + \text{Pb}(\text{CH}_3\text{COO})_2 = \text{PbS} \downarrow + 2\text{CH}_3\text{COONa}$	<p>Black ppt. of PbS indicates S²⁻ ion.</p>
<p>PO₄³⁻ ion: Acidify the sample with conc. HNO₃ and add several drops in excess. Then treat the solution with ammonium molybdate reagent and warm.</p>	<p>Yellow ppt. of ammonium phosphomolybdate.</p> $12\text{MoO}_4^{2-} + 3\text{NH}_4^+ + \text{PO}_4^{3-} + 24\text{H}^+ \rightarrow (\text{NH}_4)_3[\text{P}(\text{Mo}_{12}\text{O}_{40})] + 12\text{H}_2\text{O}$

Observation:**Practices**

1. Explain qualitative and quantitative analysis.
2. What do you mean by flame test?
3. How one can identify CO_3^{2-} , SO_4^{2-} , X^- , NO_3^- , PO_4^{3-} ?
4. How can you differentiate Fe^{2+} and Fe^{3+} ?
5. Write down the confirmation test of NH_4^+ , Ca^{2+} , Al^{3+} .
6. Discuss the precautions of the above experiment.