

Experiment	Aim	Chemicals	Glassware & Apparatus	Graph	Procedure
1. <u>Cell constant</u>	<p>1. To determine cell constant of a conductivity cell</p> <p>2. To determine the concentration of a weak acid and strong base in a mixture by conductometric titration with a strong base</p>	<ul style="list-style-type: none"> KCl salt (to make standard solution) NaOH solution Acid mixture (HCl + CH₃COOH solution) Distilled water 	<ul style="list-style-type: none"> 2x Beaker Burette 10 mL pipette 2x 100mL Volumetric flask Spatula Conductometer Small funnel Magnetic Stirrer 		PROCEDURE: A) DETERMINATION OF CELL CONSTANT Connect conductivity cell to conductivity bridge and keep the electrodes of the cell dipped in distilled water. Prepare 0.1N and 0.01N KCl solutions. To measure conductance of a particular solution, soak the electrodes of conductivity cell twice with that solution. Take 25ml of that solution in 100ml beaker and dip the electrodes in that solution. Care should be taken to see that both platinum electrodes of the cell are completely immersed in the solution. Select appropriate conductance range. Note down the conductance of 0.1N and 0.01N KCl solutions and determine the cell constant using equation $K = \frac{G_1}{C_1} \text{ or } C = \frac{G}{K}$ B) PROCEDURE Dip the conductivity cell in the given solution of mixture of acids taken in a beaker, such that the two platinum electrodes are immersed completely. The cell is connected to the conductivity bridge. Stir the solution with the help of magnetic stirrer and measure the conductance. Take the given NaOH solution in a burette mixed with the same solution. Discharge 0.5ml of NaOH solution each time to the beaker. Conductance decreases in beginning. Continue titration until conductance starts to increase slightly and continue till the conductance increases by a large value. Record few more readings. Plot the graph of conductance against volume of NaOH added. From the graph, find the neutralization points. Calculate the normality and amount of HCl and CH ₃ COOH present in the given solution.
2. <u>Potentiometric Titration</u>	<p>AIM: To determine the amount of iron present in FAS solution by potentiometric titration method.</p>	<ul style="list-style-type: none"> 0.1 N FAS Solution 0.05 N K₂Cr₂O₇ 2N H₂SO₄ Distilled Water 	<ul style="list-style-type: none"> Burette Beaker Test Tube + Holder Magnetic Stirrer [includes magnetic bid] 		PROCEDURE: Pipette out 25ml of the homogeneous ore solution in to a 100ml beaker. Hold 1ml of conc. HCl and heat the solution just to boil. To the hot solution add stannous chloride (SnCl ₂) until the color changes from yellow to colourless. Cool the solution to room temperature and addition of HgCl ₂ at a stretch, to get silver white precipitate. Dip the electrode assembly into the solution and connect to a potentiometer. Stir the solution by using magnetic stirrer and measure the potential. Add 0.5ml of potassium dichromate solution from a burette. Stir the solution well and measure the potential. Continue the process till the potential shows a rapid increase. Plot a graph of E Vs V (volume of dichromate added), ΔE/ΔV and ΔE/Δ ² V Vs V and find out the end point using these plots. Calculate the normality of the former solution and determine the amount of iron in the given volume of the solution.
3. <u>pK_a value of weak acid by pH-titration</u>	To determine pKa value of a weak acid by pH titration method	<ul style="list-style-type: none"> Acetic/Formic Acid NaOH solution 	<ul style="list-style-type: none"> Beaker (2?) Burette Digital pH-meter Magnetic Stirrer Combined electrode (calomel electrode + glass electrode) 		PROCEDURE: Pipette out 25ml of the given weak acid into a clean 100ml beaker. Insert glass electrode and calomel electrode assembly into it and connect it to pH meter. Stir the solution by using the magnetic stirrer and measure pH of the weak acid. Fill the burette with the NaOH solution. In the beginning add 0.5ml of NaOH and stir the solution. Note down the pH of the solution. Continue adding 0.5ml of NaOH each time and measure the pH regularly after each addition until the increase in pH is large. Plot the graph and determine the pKa of the given weak acid from the two graphs. Find equivalent volume of NaOH added i) ΔpH/ΔV against volume of NaOH added From the graph: Equivalence Point: 5.5 ml Half Equivalence Point: 2.75 ml pH corresponding to half equivalence point: 3.9
4. <u>Colorimetry</u>	To estimate the amount of copper present in a given solution by colorimetric method	<ul style="list-style-type: none"> Standard CuSO₄ solution NH₃ solution 	<ul style="list-style-type: none"> 2 Burettes 7 50mL Flasks Cuvette Colorimeter 		PROCEDURE: Transfer 2.4g of 10ml of given copper sulphate solution into different volumetric flasks from the burette. Add 2.5ml of ammonia to all the volumetric flasks and make up to the mark by adding distilled water. Close the volumetric flasks with stopper and shake well to get uniform concentration. Prepare a blank solution by taking 2.5ml of ammonia solution in another 50mL volumetric flask. Make up to the mark by adding distilled water and shake well. After the mixture runs, the absorbance of all solution against blank at 620nm using a photoelectric colorimeter. Note down the optical density of all solutions and calculate the readings. Draw a calibration curve by plotting absorbance against concentration of copper or volume of copper sulphate taken. Using the calibration curve find out the unknown volume of copper sulphate solution and calculate the amount of copper present in the unknown solution.

<p>5. PVA [polymer viscosity average]</p>	<p>To determine the molecular weight of a polymer by viscosity measurements using Mark-Houwink equation</p>	<ul style="list-style-type: none"> • Polyvinyl alcohol (stock solution with concentration of 1gm/dL) • Acetone • Viscometer • Volumetric flask • Water Bath • Pipette • Pipette Head • Stop Watch • Stand • Burette 		<p>PROCEDURE:</p> <ol style="list-style-type: none"> i) From the given stock solution (1g/dL) prepare polymer solutions of concentrations 0.2g/dL & 0.5g/dL. ii) For 0.2g/dL solution take 10ml of stock solution into 50ml standard flask and make up to mark with deionized water. iii) Take a clean and dry viscometer. Fix it vertically to a stand in a constant temperature bath (water containing water). Using a clean and dry pipette transfer a known volume (1ml) of the prepared polymer solution (0.2g/dL) into the wider bulb of viscometer. Allow 5 minutes for viscometer to thermally equilibrate. By applying suction, bring the sample above the upper mark of the small bulb of the viscometer. Allow the solution to flow freely through the capillary. When the solution crosses the upper mark of the bulb, start a stop watch and when the solution crosses the lower mark of the small bulb stop the stop watch. Note down the time flow of the polymer solution in seconds. Repeat time flow measurements atleast 3 times and calculate the avg time. iv) Remove the solution from viscometer and clean well with acetone. Dry the viscometer in hot air oven for 15 minutes and cool it to room temperature. v) For 0.5g/dL solution take 25ml of stock solution into 50ml standard flask and make up to mark with deionized water. Measure the time flow of the solution. vi) Remove the solution from viscometer and clean well with acetone. Dry the viscometer in hot air oven for 15 minutes and cool it to room temperature. vii) Now transfer the same volume of distilled water (1ml) to the viscometer and measure the time flow of water (in sec). viii) Draw the graph and find the intercept value [η].
<p>6. Eutectic system</p>	<p>To construct a phase diagram for a simple eutectic system using cooling curves</p>	<ul style="list-style-type: none"> • Biphenyl • Naphthalene 	<ul style="list-style-type: none"> • Electronic wight balance • Spatula • Temperature Probe • Stop Watch • Water Bath • Boiling Tube • Stand with a Clamp to hold the boiling tube 	
<p>7. Complexometric Titration</p>	<p>To determine the water quality parameters</p> <ol style="list-style-type: none"> 1. pH 2. Conductivity 3. Total Dissolved Salts 4. Total Hardness associated with the given water sample 	<ul style="list-style-type: none"> • Hard Water • NH₃ • Buffer solution • Eriochrome Black-T [EBT] • Na₂EDTA (Sodium ethylenediamine tetra acetic acid) • Burette • 25ml Pipette • 250ml volumetric flask • Small and Big funnel • Crucible • Wash Bottle • Conical Flask • Beaker • Weighing Balance • Bunsen Burner • pH-meter • Conductivity Meter 	N/A	<p>PROCEDURE:</p> <p>A) pH:</p> <p>Wash the combined electrode (glass electrode and saturated calomel electrode) of a calibrated pH-meter with deionized water. Take the sample water in a 100 ml. clean beaker and insert the combined electrode of the pH-meter into it. Note the pH of the water sample.</p> <p>B) Conductivity:</p> <p>Wash the conductivity cell of the conductometer with deionized water. Take 10ml of water sample in a 100ml beaker and insert the conductivity cell. Measure the conductance of the water sample after 10 minutes.</p> <p>C) Total Dissolved salts:</p> <p>The initial weight of a dry crucible is noted. 10 ml. of a sample of water is poured into a crucible. The water is evaporated and the crucible is allowed to cool. The weight of the crucible is recorded again. The difference between the two weights gives an estimate of the TDS of the water sample.</p> <p>D) Hardness:</p> <p>a) Preparation of standard Na₂EDTA solution:</p> <p>Weigh the weighing bottle containing disodium salt of EDTA accurately and transfer the salt onto the funnel placed in a 250 ml. volumetric flask. Weigh the bottle again. The difference in weights will give the amount of Na₂EDTA transferred. Add 3 ml. of ammonia solution onto the funnel. Add deionized water through the funnel to ensure that all crystals slide down the flask. Wash the funnel with deionized water 4-5 times. Add deionized water up to half the flask. Swirl the flask continuously till the salt dissolves completely. Make the solution up to the mark in such a way that lower meniscus of the solution coincides with the mark. Shake the flask well for uniform concentration.</p> <p>b) Estimation of hardness of water:</p> <p>Pipette out 25 ml. of the given sample of hard water into a clean conical flask. Add 3 ml. of NH₄OH-NH₄Cl buffer followed by pinch of Eriochrome black-T indicator. Titrate this against standard Na₂EDTA solution till the color of the solution changes from wine red to clear blue. Repeat the experiment to get concordant values.</p>

8. <u>COD</u> [Redox Titration]	TO ESTIMATE CHEMICAL OXYGEN DEMAND OF A GIVEN INDUSTRIAL WASTE WATER SAMPLE	<ul style="list-style-type: none"> • Industrial waste water solution • $K_2Cr_2O_7$ solution • Ferroin indicator • H_2SO_4 solution (1:1) 	<ul style="list-style-type: none"> • 250ml Volumetric Flask • 25ml Pipette • 10ml Pipette • 250ml Conical Flask • Big Small Funnel • Burette • Test Tube with Holder 	N/A	PROCEDURE: A) Preparation of Standard Ferrous Ammonium Sulphate (FAS) Solution: Weigh accurately the given FAS crystals and transfer it into a 250mL volumetric flask using a funnel. Add 1/2 test tube of 1:1 sulfuric acid followed by small amount of water. Dissolve the salt and make up to the mark and shake well for uniform concentration. B) Blank Titration: Pipette out 10mL of potassium dichromate solution into a clean conical flask. Add 3/4 th test tube of 1:1 sulfuric acid. Add 3-4 drops of Ferroin indicator. Titrate this against standard FAS solution taken in the burette until the colour changes from bluish green to reddish brown. Note the burette reading and repeat the titration to get concordant values. C) Back Titration: Pipette out 25mL of industrial waste water in to a clean conical flask. Add 10mL of potassium dichromate solution. Add 3/4 th test tube of 1:1 sulfuric acid. Add 3-4 drops of Ferroin indicator. Titrate this against standard FAS solution taken in the burette until the colour changes from bluish green to reddish brown. Note the burette reading and repeat the titration to get concordant values.
9. α -Al ₂ O ₃	AIM: To synthesise α -Al ₂ O ₃ Nano Metal Oxide by combustion method.	<ul style="list-style-type: none"> • 20.0 g of Aluminium nitrate [(Al(NO₃)₃).9H₂O; Mol. Wt = 375.134 g/mole] • 5.0 g of urea [NH₂CONH₂; Mol. Wt = 60] • Distilled Water 	<ul style="list-style-type: none"> • 250 mL beaker • Weighing Balance • Magnetic Stirrer • Furnace 	N/A	PROCEDURE: Weigh exactly 20.0 g of Aluminium nitrate [(Al(NO ₃) ₃).9H ₂ O; Mol. Wt = 375.134 g/mole] and 5.0 g of urea [NH ₂ CONH ₂ ; Mol. Wt = 60] in 250 mL beaker and dissolve the salts by adding minimum quantity of distilled water and stirring on a magnetic stirrer for 10 min. The uniformly dissolved redox mixture is kept in a pre-heated furnace maintained at 500 °C. The redox solution undergoes evaporation and finally results in viscous-gel containing uniformly mixed Aluminium nitrate and urea. After some time it catches fire with the liberation of gases and heat energy, finally results in highly porous, white powder of nano α -Al ₂ O ₃ . $2Al(NO_3)_3(s) + 5CH_2N_2O(s) \xrightarrow{500^\circ C} \alpha-Al_2O_3 + 5CO(g) + 8N_2(g) + 8H_2O(g)$