Model procedure writing for I / II semester
Chemistry Practical Examination

Expt. No. 1 :Estimation of Total hardness of water by EDTA complexometric method

- **Part A**

  * Weigh out the given Na_2EDTA crystals accurately into a 250 ml volumetric flask and add 3ml of Ammonia solution. Dissolve in distilled water and make up to the mark. Mix well and find the Molarity.

  $$\text{Molarity of } Na_2EDTA = \frac{\text{Weight of } Na_2EDTA \times 4}{\text{Molecular weight of EDTA (372.24)}}.$$

- **Part B**

Burette : Standard Na_2EDTA solution

Conical flask: 25ml of water sample + 4ml of (NH_3NH_4Cl) buffer solution to maintain pH =10
Indicator : Eriochrome Black-T (a pinch)

End point : Wine red to clear blue

Conclusion : From the volume of Na_2EDTA consumed calculate the total hardness of given water sample

Expt. No. 2 :Estimation of CaO in cement solution by rapid EDTA method

- **Part A**

Weigh out the given Na $_2$EDTA crystals accurately into a 250ml volumetric flask, add 3ml of Ammonia solution Dissolve it in distilled water and made up to the mark. Mix well and find the Molarity.

$$\text{Molarity of } Na_2EDTA = \frac{\text{Weight of } Na_2EDTA \times 4}{\text{Molecular weight of EDTA (372.24)}}.$$

- **Part B**

Burette: Standard Na_2EDTA solution

Conical flask: 25ml of Cement solution + 4 ml of 4N NaOH + 3ml of Diethylamine + 3ml 1:1Glycerol

Indicator: Patton & Reeder's Indicator
End point: Wine red to clear blue

Conclusion : From the volume of Na_2EDTA consumed calculate the percentage of Calcium Oxide in the given cement Solution.

Expt. No. 3 :Determination of percentage of Copper in brass using standard sodium thiosulphate solution

- **Part A**

Weigh out the given brass sample into a dry conical flask. Add few drops of conc. HNO_3 to dissolve, and add 1/2 T.T of distilled water, boil till all brown fumes are expelled. Add 1gm of urea, boil and cool to room temperature.

- **Part B**

Burette: Standard $Na_2S_2O_3$solution.

Conical Flask: Brass solution prepared above + NH_4OH till bluish white ppt + 5ml dil acetic acid+ 10ml. of 10% KI.

Indicator: Starch (added towards the end point)

End Point: Blue to white

Conclusion : From the Volume of Na $_2S$ $_2O_3$ consumed, calculate the percentage of Copper in the given brass sample.

Expt. No. 4: Estimation of Iron in haematite ore solution using standard $K_2Cr_2O_7$ solution by External Indicator method

- **Part A**

Weigh out the given Potassium Dichromate crystals accurately into 250 ml volumetric flask. Dissolve in distilled water. Dilute up to the mark. Mix well and find the normality of $K_2Cr_2O_7$ solution by using the relation.

$$\text{Normality of } K_2Cr_2O_7 = \frac{\text{Weight of } K_2Cr_2O_7 \times 4}{\text{Equivalent weight of } K_2Cr_2O_7(49)}$$

- **Part B**

Burette: Standard $K_2Cr_2O_7$solution.

Conical Flask: 25ml of Haematite ore solution + 5ml Conc. HCl, boil + $SnCl_2$to hot solution till colorless + Cool + 5ml of $HgCl_2$

Indicator: $K_3[Fe(CN)_6]$ (external)

End Point: No change in the color of indicator with the test solution drop

Conclusion : From the Volume of K $_2Cr_2O_7$ consumed, calculate the percentage of Iron in the given haematite sample.

Expt. No. 5: Determination of COD of waste water

- **Part A**

Weigh out the given ferrous ammonium sulphate (FAS) crystals accurately into a 250ml volumetric flask. Add ½ T.T of dil H_2SO_4 dissolve the crystals, dilute up to the mark and mix well and find the normality by using the relation.

$$\text{Normality of FAS} = \frac{\text{Weight of FAS} \times 4}{\text{Equivalent weight of FAS (392)}} .$$

- **Part B: Back titration**

Burette: Standard FAS solution

Conical Flask: 25ml waste water sample + 10ml $K_2Cr_2O_7$ (pipette out) + 1 t.t. of 1:1 H_2SO_4

Indicator : Ferroin (ferrous 1, 10 phenonthrolinesulphate)

End Point: Blue green to reddish brown

Blank titration

Perform the blank titration in the same way as above but without wastewater

Conclusion : From the difference in the titre value, calculate the COD of Waste water sample.

Expt. 6: Estimation of percentage of available chlorine in the given sample of bleaching powder (Iodometric method)

Part-A

Preparation of std. sod. thiosulfate solution

Weigh out the given sodium thiosulfate crystals accurately into 250ml volumetric flask. Dissolve in distilled water. Dilute up to the mark. Mix well and find the normality of $Na_2S_2O_3$ solution by using the relation.

$$\text{Normality of } Na_2S_2O_3 = \frac{\text{Weight of } Na_2S_2O_3 \times 4}{\text{Equivalent weight of } Na_2S_2O_3}$$

Part-B

Burette – Standard solution

Conical flask-10 mL of given sample + 5 mL of acetic acid + 10 mL of KI solution

Indicator –starch (Towards the end point)

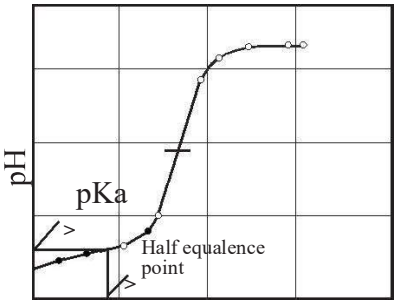
End point-Blue to white

Conclusion : From the volume of $Na_2S_2O_3$, Find out the available chlorine of given sample.

Expt. No. 7 : Determination of pKa of weak acid using pH meter

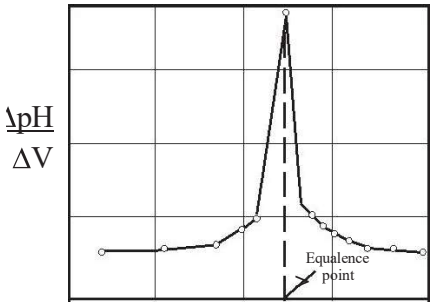
Pipette out 25ml of given weak acid into a beaker. Immerse the glass electrode + Calomel electrode assembly into it. Connect the electrodes to pH meter and measure the pH. Now add NaOH from the burette in increments of 0.5 ml and measure the pH after each addition.

Plot graph $\Delta p^H / \Delta V$ against the volume of NaOH. Determine the equivalence point plot another graph of pH against volume of NaOH and determine pKa of the given weak acid as shown below.



(a) **Volume of NaOH (cm^3)**

First derivative curve



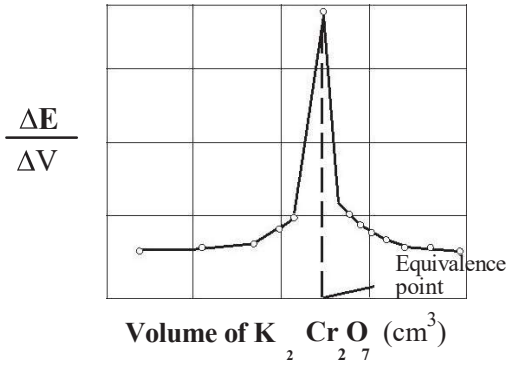
(b) **Volume of NaOH (cm^3)**

Expt. No. 8: Potentiometric estimation of FAS using standard $K_2Cr_2O_7$ solution

Pipette out 25ml of FAS into a beaker. Add 1 t.t dil H_2SO_4, immerse calomel electrode + platinum electrode into it. Connect the assembly to a potentiometer and measure the potential by adding $K_2Cr_2O_7$ in the increments of 0.5ml.

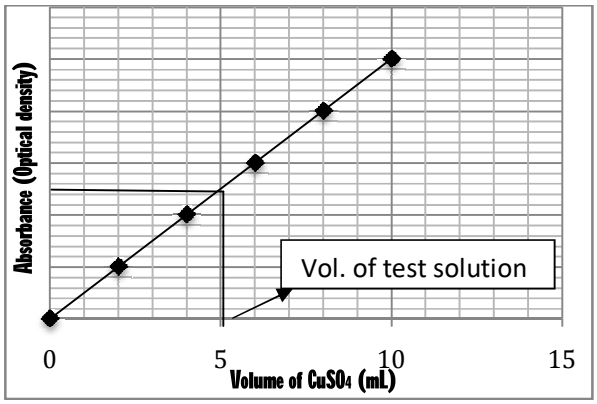
Plot graph $\Delta E/ \Delta V$ against volume of $K_2Cr_2O_7$, and determine the equivalence point. From the normality and volume $K_2Cr_2O_7$, solutions calculate the normality and the weight of FAS in the given solution.

First derivative curve



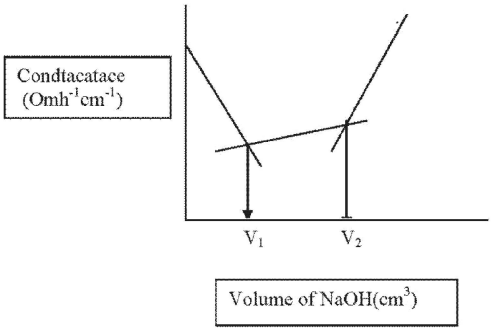
Expt. No. 9 : Colorimetric Estimation of Copper

Draw out 2, 4, 6, 8, and 10 ml cm^3 of the Copper sulphate solution into $50cm^3$ volumetric flask. Add $5cm^3$ of ammonia solution to each of them and dilute upto the mark with distilled water and mix well. Prepare a blank solution by diluting $5cm^3$ of ammonia solution in $50cm^3$ volumetric flasks. For test solution add 5ml of NH_3 and make up to the mark. Measure the absorbance of each of these against blank solution at 620 nm. Plot a graph of absorbance (OD) against volume of copper sulphate solution and determine the volume of copper sulphate solution in the test sample as shown in the figure and find the amount of copper present in it.



Expt.No.10 Estimation of Acids in acid mixture conductometrically

Pipette out 50ml acid mixture into a beaker. Immerse the conductivity cell into it. Connect the conductivity cell to a conductivity meter and measure the conductance by adding NaOH from the burette by increment of 1 ml.Plot a graph of conductance against volume of NaOH. Determine the two neutralization points from the graph as shown below and find the weight of HCl and CH_3 COOH.



Expt. No. 11 : Determination of Viscosity co-efficient of the given liquid using Ostwald's viscometer

Pipette out 10 ml of the given liquid in to the wide limb of the dried viscometer and suck the liquid through the other limb. Determine the time of flow between two fixed points. One above and one below the bulb in the narrow limb of the viscometer. Repeat and calculate the average time of flow. Pour out the liquid, rinse the viscometer with Acetone and dry it. Now pipette out 10 ml of water into the wider limb and determine the average time of flow for water as before. From the density of the liquid and of water and the viscosity coefficient of water, determine the viscosity coefficient of the given liquid.

$\eta_l = \eta_w x \frac{t1\ d1}{twdw}$

Where,

η_1= coefficient of viscosity of the test liquid.

η_w= coefficient of viscosity of the water.

t_w= time taken (in seconds) by the water to flow from point A to B.

t_l= time taken (in seconds) by the liquid to flow from point A to B.

d_1= density of the given liquid.

d_w= density of water.