Bhakta Kavi Narsinh Mehta University

BSc Semester VI
CBCS
Chemistry Practicals

Question Papers
With effect from June - 2018

Bhakta Kavi Narsinh Mehta University
BSc Semester VI [CBCS]
Chemistry Practical Examination

INORGANIC QUALITATIVE ANALYSIS

Time: 3.00 Hrs **Marks: [35+5]**

Exercise 1: Inorganic Qualitative Analysis

In the container bearing the **No ……** you are given a mixture of inorganic salts containing not more than six radicals. Perform the qualitative analysis of the inorganic salt mixture and detect all the six radicals present in the given mixture.

NOTE:

- First perform the dry tests and detect the radicals. Show the tests for each radical to the Examiner.
- **Get the results of the dry test initialed by the Examiner before proceeding to analyze the mixture by the wet tests.**
- The results of the wet test for the detected group as well as the confirmatory tests for each radical should be shown to the Examiner
- Systematically write all the tests:
  - Preliminary Tests
  - Dry Tests:
    - ✓ For Positive Radical
    - ✓ For Negative Radical
  - **Results of Dry Test**

Radicals	**I**	**II**	**III**
Positive Radicals			
Negative Radicals			

  - Preparation of the Original Solution
  - Wet Tests:
    - ✓ Detection of Positive Radical with confirmatory test
    - ✓ Detection of Negative Radical with confirmatory test
  - Result (In the form of a tabulation as indicated below)

Results of Wet Tests

Radicals	**I**	**II**	**III**
Positive Radicals			
Negative Radicals			

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ORGANIC SYNTHESIS

Time: 3.00 Hrs **Marks: [30]**

Exercise No. 2 Acetylation of Salicylic acid

Aim: Synthesize Acetyl Salicylic acid (Aspirin) from Salicylic acid and determine the percentage yield of the product. Re-crystallize the product and note its melting point

Requirements

..... gms Salicylic acid

..... gms (..... ml) Acetic Anhydride

..... ml Conc H_2SO_4

Procedure

Take ..... gms Salicyclic acid in a 250 ml conical flask. Add ..... gms (.....ml) Acetic Anhydride and ..... ml conc H_2SO_4 and warm the mixture in a water bath at about 50-60 ^{0}C for half an hour (shake the flask well during the reaction). Add about 150 ml distilled water and shake it well, white precipitates of Aspirin separates out. Filter, wash, dry and weigh the product.

Re-crystallize the product and note its melting point.

Show the product to the Examiner and get the signature of Examiner on the practical yield of the synthesized product.

Ask the Examiner for the theoretical yield of the product and then calculate the percentage yield of the synthesized product

Result:

Structure of the Product	MP ^{0}C	Theoretical Yield in gms	Practical Yield in gms	Percentage Yield

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ORGANIC SYNTHESIS

Time: 3.00 Hrs **Marks: [30]**

Exercise No. 3 Acelytation of Aniline

Aim: Synthesize Acetanilide from Aniline and determine the percentage yield of the product. Re-crystallize the product and note its melting point

Requirements

..... gms (..... ml) Aniline

..... gms (..... ml) acetic anhydride

Aqueous solution of HCl (10ml HCl in 250 ml water)

Procedure

Take ..... gms (..... ml) Aniline in a 500 ml conical flask containing 250 ml aqueous solution of HCl (10ml HCl in 250 ml water). Shake the flask well, Aniline dissolves in the solution.

Then add ..... gms (..... ml) acetic anhydride drop by drop.

Keep the flask in an ice bath for about half an hour, white precipitates of acetanilide separates out. Filter, wash, dry and weigh the product.

Re-crystallize from rectified spirit and note its melting point

Show the product to the Examiner and get the signature of Examiner on the practical yield of the synthesized product.

Ask the Examiner for the theoretical yield of the product and then calculate the percentage yield of the synthesized product

Result:

Structure of the Product	MP ^{0}C	Theoretical Yield in gms	Practical Yield in gms	Percentage Yield

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ORGANIC SYNTHESIS

Time: 3.00 Hrs **Marks: [30]**

Exercise No. 4 Acetylation of Phenol

Aim: Synthesize Phenyl acetate from Phenol and determine the percentage yield of the product and note its boiling point

Requirements:

..... gms Phenol

.....gms (..... ml) acetic anhydride

80 ml 10 % NaOH solution

10 ml CCl_4

Na_2CO_3

Procedure:

Take 80 ml 10 % NaOH solution in a 250 ml conical flask. Add .....gms Phenol and shake it till the phenol dissolves in the NaOH solution.

Now add a few pieces of rushed ice and ..... gms (.....ml) acetic anhydride; shake it well for about 10 minutes. An emulsion of phenyl acetate is obtained. Take the emulsion in a separating flask and add 10 ml CCl_4. Shake it well phenyl acetate distributes in the CCl_4 layer.

Take the CCl_4 layer in another separating flask add Na_2CO_3 till the evolution of CO_2 ceases. Shake it well and discard the lower layer.

Take the upper layer in a round bottom flask and distill to remove CCl_4 (CCl_4 distills at 170 ^{0}C). After CCl_4 is distilled out warm further to 194 ^{0}C the residue is Phenyl acetate

Measure the quantity of phenyl acetate obtained and Note is boiling point

Show the product to the Examiner and get the signature of Examiner on the practical yield of the synthesized product.

Ask the Examiner for the theoretical yield of the product and then calculate the percentage yield of the synthesized product

Result:

Structure of the Product	BP ^{0}C	Theoretical Yield in gms	Practical Yield in gms	Percentage Yield

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ORGANIC SYNTHESIS

Time: 3.00 Hrs **Marks: [30]**

Exercise No. 5 Benzoylation of Aniline

Aim: Synthesize Benzanilide from anilne and determine the percentage yield of the product. Re-crystallize the product and note its melting point

Requirements

..... gms (..... ml) Aniline

..... gms (..... ml) Benzoylchloride

50 ml 10 % NaOH solution

Procedure

Take ..... gms (..... ml) Aniline in a 250 ml conical flask and add 50 ml 10 % NaOH solution.

Now add ..... gms (..... ml) benzoylchloride slowly and gradually. Shake the flask well; a white precipitate of Benzanilide separates out and simultaneously the unpleasant odour of benzoylchloride disappears.

Add 25 ml of distilled water to this alkaline mixture and shake the flask well. Filter, wash, dry and weigh the product;

Re-crystallize from rectified spirit and note its melting point

Show the product to the Examiner and get the signature of Examiner on the practical yield of the synthesized product.

Ask the Examiner for the theoretical yield of the product and then calculate the percentage yield of the synthesized product

Result:

Structure of the Product	MP ^{0}C	Theoretical Yield in gms	Practical Yield in gms	Percentage Yield

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ORGANIC SYNTHESIS

Time: 3.00 Hrs **Marks: [30]**

Exercise No. 6 Benzoylation of Phenol

Aim: Synthesize Phenyl benzoate from Phenol and determine the percentage yield of the product. Re-crystallize the product and note its melting point

Requirements

..... gms Phenol

.....ml Benzoyl chloride

30 ml 10% Aqueous NaOH solution

Procedure

Take .....gms Phenol in a 250 ml conical flask containing 30 ml 10 % NaOH solution. Now add .....ml benzoyl chloride slowly and gradually. Close the flask with a cork and shake it well for about 15 mins, white precipitates of phenyl benzoate separates out and simultaneously the unpleasant odour of benzoyl chloride disappears.

Add 25 ml of distilled water to this alkaline mixture and shake it well. Filter, wash well with distilled water, dry and weigh the product.

Re-crystallize from hot rectified spirit and note its melting point

Show the product to the Examiner and get the signature of Examiner on the practical yield of the synthesized product.

Ask the Examiner for the theoretical yield of the product and then calculate the percentage yield of the synthesized product

Result:

Structure of the Product	MP ^{0}C	Theoretical Yield in gms	Practical Yield in gms	Percentage Yield

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Time: 3.00 Hrs **Marks: [30]**

Exercise No. 7 Preparation of iodoform from ethanol

Aim: Synthesize iodoform from ethanol and determine the percentage yield of the product. Re-crystallize the product and note its melting point

Requirements

.....ml (.....g) ethanol

25 ml 33 % aqueous K_2CO_3 solution

..... gms solid Iodine

Procedure

Take .....ml (..... g) ethanol and 25 ml 33 % aqueous K_2CO_3 solution in a round bottom flask. Fit a water condenser and heat in a water bath at about 60-65 ^{0}C.

Then slowly add the powdered solid I_2 with constant stirring. Continue heating the flask in a water bath for another 15-20 minutes. [If iodine is in excess the solution will be dark yellow or orange. In such a case add a few drops of 10 % NaOH to decolourize the solution]

Cool, lemon yellow crystals of iodoform are obtained. Filter, wash dry and weigh.

Re-crystallize from rectified spirit and note its melting point

Show the product to the Examiner and get the signature of Examiner on the practical yield of the synthesized product.

Ask the Examiner for the theoretical yield of the product and then calculate the percentage yield of the synthesized product

Result:

Structure of the Product	MP ^{0}C	Theoretical Yield in gms	Practical Yield in gms	Percentage Yield

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Time: 3.00 Hrs **Marks: [30]**

Exercise No. 8 Preparation of iodoform from acetone

Aim: Synthesize iodoform from acetone and determine the percentage yield of the product. Re-crystallize the product and note its melting point

Requirements

.....gm (..... ml Acetone)

15 ml 10% NaOH

..... ml Iodine solution

Procedure

Take 15 ml 10% NaOH in 250 ml conical flask, now add drop by drop .....ml Acetone to the alkali solution and shake it well.

Add the saturated I_2 solution slowly with constant stirring till a yellow colour persists.

Warm the flask in a water bath at 60 ^{0}C for ten minutes. Then cool, lemon yellow crystals of iodoform are obtained. Filter, wash, dry and weigh.

Re-crystallize from rectified spirit and note its melting point

Show the product to the Examiner and get the signature of Examiner on the practical yield of the synthesized product.

Ask the Examiner for the theoretical yield of the product and then calculate the percentage yield of the synthesized product

Result:

Structure of the Product	MP ^{0}C	Theoretical Yield in gms	Practical Yield in gms	Percentage Yield

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Time: 3.00 Hrs **Marks: [30]**

Exercise No. 9 Preparation of m-dinitrobenzene from benzene

Aim: **Synthesize m-dinitrobenzene from benzene and determine the percentage yield of the product. Re-crystallize the product and note its melting point**

Requirements

..... gm (..... ml) Benzene

..... ml Fuming HNO_3

..... ml Conc H_2SO_4

Procedure

Take ..... ml fuming HNO_3 in a 250 ml round bottom flask. Slowly drop by drop add ..... ml conc H_2SO_4, shake it well and cool the mixture in ice cold water.

Now drop by drop add ..... gm (..... ml) Benzene to the mixture in the round bottom flask. Fit a water condenser over the round bottom flask and reflux the mixture for one and a half hour in a water bath.

Cool the mixture and then pour it in a beaker containing crushed ice.

Stir well yellow coloured solid m-dinitrobenzene separates out. Filter, wash with water, dry and weigh.

Re-crystallize from rectified spirit and note its melting point

Show the product to the Examiner and get the signature of Examiner on the practical yield of the synthesized product.

Ask the Examiner for the theoretical yield of the product and then calculate the percentage yield of the synthesized product

Result:

Structure of the Product	MP ^0C	Theoretical Yield in gms	Practical Yield in gms	Percentage Yield

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Time: 3.00 Hrs **Marks: [30]**

Exercise No. 10 Preparation of p-nitroacetanilide from acetanilide

Aim: **Synthesize p-nitroacetanilide from acetanilide and determine the percentage yield of the product. Re-crystallize the product and note its melting point**

Requirements

..... gms Acetanilide

..... ml Glacial Acetic Acid

..... ml conc H_2SO_4

Nitrating mixture: .....gms (.....ml) conc HNO_3 + ... ml conc H_2SO_4

Procedure

Take ..... gms Acetanilide in a 250 ml conical flask. Add ..... ml glacial Acetic Acid and ..... ml conc H_2SO_4. Shake it well till a clear solution is obtained.

Keep the conical flask in a freezing mixture (ice + salt mixture) so that the mixture attains 0-2 ^{0}C.

Then add the Nitrating mixture i.e. .....gms (.....ml) conc HNO_3 + ... ml conc H_2SO_4 and stir the flask well (the temperature should not exceed 10 ^{0}C while the nitrating mixture is being added).

Keep the mixture at room temperature for 30 minutes.

Then pour the mixture in a beaker containing ice cold water, white crystals of p-nitroacetanilide separates. Filter, wash with water, dry and weigh.

Re-crystallize from rectified spirit and note its melting point

Show the product to the Examiner and get the signature of Examiner on the practical yield of the synthesized product.

Ask the Examiner for the theoretical yield of the product and then calculate the percentage yield of the synthesized product

Result:

Structure of the Product	MP ^{0}C	Theoretical Yield in gms	Practical Yield in gms	Percentage Yield

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Time: 3.00 Hrs **Marks: [30]**

Exercise No. 11 Preparation of p-bromoacetanilide from acetanilide

Aim: **Synthesize p-bromoacetanilide from acetanilide and determine the percentage yield of the product. Re-crystallize the product and note its melting point**

Requirements

..... gms Acetanilide

..... ml Glacial Acetic Acid

.....gms (.....ml) bromine in ..... ml glacial acetic acid

Procedure

Take ..... gm acetanilide in a 250 ml conical flask and dissolve it in ..... ml glacial acetic acid. Keep the flask in an ice bath.

Now drop by drop add the Bromine in glacial acetic acid to the solution and shake it well. The mixture turns orange colour due to the bromination reaction.

Keep the reaction mixture at room temperature for one and a half hour and then pour it in a beaker containing crushed ice. Stir well p-bromo acetanilide separates out.

Filter, wash with water, dry an weigh.

Re-crystallize from rectified spirit and note its melting point.

Show the product to the Examiner and get the signature of Examiner on the practical yield of the synthesized product.

Ask the Examiner for the theoretical yield of the product and then calculate the percentage yield of the synthesized product

Result:

Structure of the Product	MP ^{0}C	Theoretical Yield in gms	Practical Yield in gms	Percentage Yield

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Time: 3.00 Hrs **Marks: [30]**

Exercise No. 12 Preparation 2:4:6 -tribromo phenol from phenol

Aim: **Synthesize 2, 4, 6 -tribromophenol from phenol and determine the percentage yield of the product. Re-crystallize the product and note its melting point**

Requirements

..... gms Phenol

..... ml liquid bromine

Procedure

Take ..... gm phenol in a 500 ml conical flask and dissolve it in 250 ml water. Keep the flask in an ice bath.

Now add drop by drop ..... ml previously cooled liquid Bromine and shake the flask vigourously, yellow colour 2, 4, 6-tribromophenol is obtained. [If the colour of the solution is dark yellow or orange decolourize by adding sodium bisulphate solution]

Filter, wash with excess water, dry and weigh the product.

Re-crystallize from rectified spirit and note its melting point

Show the product to the Examiner and get the signature of Examiner on the practical yield of the synthesized product.

Ask the Examiner for the theoretical yield of the product and then calculate the percentage yield of the synthesized product

Result:

Structure of the Product	MP ^{0}C	Theoretical Yield in gms	Practical Yield in gms	Percentage Yield

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Time: 3.00 Hrs **Marks: [30]**

Exercise No. 13 Preparation of Methyl Orange from sulphanilic acid

Aim: Synthesize Methyl Orange from Sulphanilic acid and determine the percentage yield of the product. Re-crystallize the product and note its melting point

Requirements

..... gm Sulphanilic acid
..... gm anhydrous Na_2CO_3
..... gm $NaNO_2$
..... ml N,N-dimethyl aniline
..... ml conc HCl
..... ml glacial acetic acid
..... ml 20% NaOH solution
..... gms NaCl

Procedure

In a 250 ml conical flask dissolve .....gm Sulphanilic acid and .....gm anhydrous Na_2CO_3 in 100 ml distilled water. Keep the flask in an ice bath and cool to below 5 ^{0}C.

Now add .....gm $NaNO_2$ (aqueous solution). Shake well and pour the mixture in a 500 ml beaker containing ..... ml conc HCl and 100 gms crushed ice. Stir well till the evolution of HNO_2 ceases and diazobenzene sulphonate precipitates.

Dissolve ..... ml N, N-dimethyl aniline in ..... ml glacial acetic acid and add it to the precipitates of diazobenzene sulphonate.

Shake the flask well and allow it to set for about 10 mins, red coloured acidic methyl orange separates out. Add ..... ml 20% NaOH solution and shake well, granules of sodium salt of methyl orange separates out (the mixture will be orange in colour).

Allow the mixture to stand for 15 mins and then cool in ice, Reddish orange couloured Methyl Orange will separate out.

Filter, wash, dry and weigh the product. Recrystallize from water and note its melting point

Show the product to the Examiner and get the signature of Examiner on the practical yield of the synthesized product.

Ask the Examiner for the theoretical yield of the product and then calculate the percentage yield of the synthesized product

Result:

Structure of the Product	MP ^{0}C	Theoretical Yield in gms	Practical Yield in gms	Percentage Yield

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ORGANIC SYNTHESIS

Time: 3.00 Hrs **Marks: [30]**

Exercise No. 14 Preparation of Methyl Red from anthranilic acid

Aim: Synthesize Methyl Red from Anthranilic acid and determine the percentage yield of the product. Re-crystallize the product and note its melting point

Requirements

..... gm Anthranilic acid ..... ml N,N-dimethyl aniline

..... ml conc HCl ..... ml 20% NaOH solution

..... gm $NaNO_2$ .....gm sodium acetate

Procedure

Take .....gm Anthranilic acid in a 100 ml beaker, add ..... ml conc HCl and 15 ml water to dissolve the anthranilic acid. Filter to remove impurities. Take the filtrate in a 250 ml beaker and place the beaker in an ice bath.

Now add .....ml conc HCl and about 25 gms crushed ice and stir well. The temperature reaches to about 3 ^{0}C; now add .....gms $NaNO_2$ and stir well till no vapours of HNO_2 evolves (Test with starch iodide paper).

Now rapidly add .....ml N, N-dimethyl aniline and stir the mixture well. (The temperature should not increase above 5 ^{0}C)

Dissolve .....gms sodium acetate in 10 ml aqueous solution. Now add this solution to the above reaction mixture with constant stirring and keep it in an ice bath for about half an hour. Then allow the reaction mixture to stand at room temperature.

Then add ..... ml 20% NaOH solution, shake well and keep the reaction mixture for 1 hour.

Filter the solid, dry in air and reflux, in a water bath, the reaction mixture with 50 ml methyl alcohol in a flask fitted with a water condenser. Cool in ice, wash with methyl alcohol, dry in air and weigh.

Show the product to the Examiner and get the signature of Examiner on the practical yield of the synthesized product.

Ask the Examiner for the theoretical yield of the product and then calculate the percentage yield of the synthesized product

Result:

Structure of the Product	MP ^{0}C	Theoretical Yield in gms	Practical Yield in gms	Percentage Yield

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ORGANIC SYNTHESIS

Time: 3.00 Hrs **Marks: [30]**

Exercise No. 15 Preparation of benzoic acid from benzaldehyde

Aim: Synthesize Benzoic acid from benzaldehyde and determine the percentage yield of the product. Re-crystallize the product and note its melting point

Requirements

..... gms Benzaldehyde

..... gms Na_2CO_3

.....gms $KMnO_4$

Conc HCl (to acidify)

Procedure:

Take ..... gms Benzaldehyde in a 500 ml two necked round bottom flask, add 250 ml distilled water shake it well and then add .....gms Na_2CO_3.

Put a few pieces of porcelain and fit a reflux condenser.

Add .....gms $KMnO_4$ solution slowly and gradually and reflux till the pink colour due to $KMnO_4$ vanishes (indicating complete oxidation).

Filter the product to separate solid MnO_2 from the filtrate.

Acidify the filtrate with conc HCl. White precipitates of Benzoic acid separates out.

Filter, wash, dry and weigh the product.

Recrystallize from hot water and note its melting point

Show the product to the Examiner and get the signature of Examiner on the practical yield of the synthesized product.

Ask the Examiner for the theoretical yield of the product and then calculate the percentage yield of the synthesized product

Result:

Structure of the Product	MP ^0C	Theoretical Yield in gms	Practical Yield in gms	Percentage Yield

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PHYSICOCHEMICAL EXERCISE

Time: 3.00 Hrs **Marks: [35+5]**

Exercise No. 16 pH metry

Aim: To determine Normality and gms/lit of given '*X*'N HCl by pH meter.

Requirements: '*X'* N HCl solution, Buffer solution, 0.5N NaOH solution, Glass and Calomel Electrodes

Procedure: Fill the burette with 0.5N NaOH solution. Standardize the pH meter with known pH buffer solution

Connect the electrodes to the pH meter and immerse both the electrode in a beaker containing 50ml '*X'*N HCl and measure the pH without adding NaOH. Then every time add 0.5ml NaOH from the burette, stir well and measure the pH. In the beginning the pH will be steady then it will increase suddenly and again become steady.

Observation No	Volume of 0.5 N NaOH ml	pH	Δ pH	Δ V	Δ pH / Δ V
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					

Graph

Draw the graph of

1. Volume of NaOH → pH
2. Volume of NaOH → Δ pH / Δ V

From the graphs find the neutralization point and hence determine the normality of HCl

Result

1. From Graph 1: Normality of HCl = ……
2. From Graph 2 : Normality of HCl = ……
3. Gms/litre of HCl = ……

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PHYSICOCHEMICAL EXERCISE

Time: 3.00 Hrs **Marks: [35+5]**

Exercise No. 17 pH metry

Aim: To determine the dissociation constant and Normality of given weak acid, CH_3COOH, by pH meter.

Requirements: '*X*'N CH_3COOH solution, Buffer solution, 0.5N NaOH solution, Glass and Calomel Electrodes

Procedure:

Fill the burette with 0.5N NaOH solution. Standardize the pH meter with known pH buffer solution.

Connect the electrodes to the pH meter and immerse both the electrode in a beaker containing 50ml '*X*'N CH_3COOH and measure the pH without adding NaOH. Then every time add 0.5ml 0.5N NaOH from the burette, stir well and measure the pH. In the beginning the pH will be steady then it will increase suddenly and again become steady.

Observation No	Volume of NaOH in ml	pH	Δ pH	Δ V	Δ pH / Δ V
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					

Graph

Draw the graph of

1. pH against volume of NaOH
2. Δ pH / Δ V against volume of NaOH

From the graph find half neutralization and hence determine the dissociation constant and normality of CH_3COOH

Result:

1. Dissociation constant of CH_3COOH = ……
2. Normality of CH_3COOH = ……

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PHYSICOCHEMICAL EXERCISE

Time: 3.00 Hrs **Marks: [35+5]**

Exercise No. 18 pH metry

Aim: To determine the dissociation constant and Normality of dibasic acid Oxalic acid / Malonic acid using 0.1N NaOH, by pH meter.

Requirements: 'X' N Oxalic acid / Malonic acid solution, 0.1N NaOH solution, Buffer solution, Glass and Calomel Electrodes

Procedure:

Fill the burette with 0.1N NaOH solution. Standardize the instrument with known pH buffer solution

Connect the electrodes to the pH meter and immerse both the electrode in a beaker containing 50ml '*X*'N Oxalic acid / Malonic acid and measure the pH without adding NaOH. Then every time add 0.5ml NaOH from the burette, stir well and measure the pH. In the beginning the pH will be steady then it will increase suddenly and again become steady.

Observation No	Volume of 0.5 N NaOH ml	pH	Δ pH	Δ V	Δ pH / Δ V
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					

Graph

Draw the graph of

1. Volume of NaOH → pH
2. Volume of NaOH → Δ pH / Δ V

From the graph find half neutralization and hence determine the dissociation constant and normality of the dibasic acid Oxalic acid / Malonic acid

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Result

1. Dissociation constant of the dibasic acid Oxalic acid / Malonic acid = ……
2. From Graph 1: Normality of the dibasic acid Oxalic acid / Malonic acid = ……
3. From Graph 2: Normality of the dibasic acid Oxalic acid / Malonic acid = ……

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PHYSICOCHEMICAL EXERCISE

Time: 3.00 Hrs **Marks: [35+5]**

Exercise No. 19 Potentiometry

Aim: To determine normality and dissociation constant of benzoic acid using 0.5 N NaOH solution by potentiometry

Given:

1. 'X' N Benzoic acid
2. Calomel electrode
3. Platinum Electrode
4. 0.5 N NaOH
5. Quinhydron powder

Procedure:

Construct the cell and show it to the Examiner before starting the experiment.

Take 50 ml of the given 'X' N Benzoic acid solution in a 150 ml beaker. Add about 0.5 gms Quinhydron powder. Connect calomel and Platinum electrodes with the potentiometer and dip the electrodes in the solution. Note the zero reading

Titrate the solution with 0.5 N NaOH solution by adding 0.5 ml from the burette each time. Take readings after every addition of 0.5 ml 0.5 N NaOH solution from the burette.

Take some more reading even after the end point. Tabulate your readings as shown below

No.	Vol of 0.5 N NaOH added 'V' ml	Potential in mv 'E' mv	ΔE	ΔV	$\Delta E / \Delta V$

Graphs:

1. Draw the graph of E against V, determine the end point and hence the normality and dissociation of benzoic acid, by using half neutralization point
2. Draw the graph of $\Delta E/\Delta V$ against V, determine the end point and hence the normality and dissociation of benzoic acid

Result:

1. Normality of Benzoic acid = _____
2. Dissociation constant of Benzoic acid = _____

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PHYSICOCHEMICAL EXERCISE

Time: 3.00 Hrs **Marks: [35+5]**

Exercise No. 20 Potentiometry

Aim : To determine normality of given acid 'x' N HCl by potentiometric titration against 0.5 N NaOH solution.

Requirements

1. 'x' N HCl
2. 0.5 N NaOH solution
3. Saturated KCl solution
4. KCl salt bridge
5. Quinhydron powder
6. Calomel electrode
7. Platinum/ Glass electrode

Procedure:

Construct the cell and show it to the Examiner before starting the experiment.

Standardize the potentiometer with Cd cell or Weston Cell

Take 25 ml of the given 'x' N HCl solution in a 150 ml beaker. Use glass electrode. If the glass electrode is not available, make it saturated with respect to quinhydron to prepare a quinhydron electrode.

Connect Calomel electrode (to the Positive end) and Platinum electrode (to the negative end) to the potentiometer and dip the electrodes in the solution. Determine the potential of this system and note the zero reading

Go on measuring the potential every time after adding 0.5 ml 0.5N NaOH solution from the burette each time and determine the reading. Take some more reading even after the end point Tabulate your readings as shown below

Similarly perform other titration, by adding 0.1 ml 0.5 N NaOH solution each time, near the end point. Tabulate your readings as shown below

No.	Vol of 0.5 N NaOH added 'V' ml	Potential in mv 'E' mv	ΔE	ΔV	ΔE / ΔV

Graphs:

1. Draw the graph of E against V, determine the end point and hence the normality of HCl
2. Draw the graph of ΔE/ΔV against V, determine the end point and hence the normality of HCl

Result:

1. Normality of HCl (from Graph -1) = _____
2. Normality of HCl (from Graph -2) = _____

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PHYSICOCHEMICAL EXERCISE

Time: 3.00 Hrs **Marks: [35+5]**

Exercise No. 21 Potentiometry

Aim: To determine concentration of 'x' N $FeSO_4(NH_4)_2SO_4.6H_2O$ solution by potentiometric titration against 0.5 N $K_2Cr_2O_7$ solution and determine the redox potential system.

Requirements

1. 'x' N $FeSO_4$ $(NH_4)_2SO_4.6H_2O$ solution
2. 0.5 N $K_2Cr_2O_7$
3. 2N H_2SO_4 solution
4. Calomel electrode
5. Platinum electrode

Procedure:

Construct the cell and show it to the Examiner before starting the experiment.

Calibrate the potentiometer with a standard cell

Take 25 ml of the given 'x' N $FeSO_4(NH_4)_2SO_4.6H_2O$ solution in a 150 ml beaker and add 25 ml 2N H_2SO_4 solution.

Connect the Calomel electrode and Platinum electrode to the potentiometer and dip the electrodes in the solution. Determine the potential of this system and note the zero reading

Go on measuring the potential every time after adding 0.5 ml 0.5 N $K_2Cr_2O_7$ solution from the burette each time. (Stir the solution well in the beaker). Take some more reading even after the end point. Tabulate your readings as shown below.

Similarly perform another titration, by adding 0.1 ml 0.5 N $K_2Cr_2O_7$ solution each time, near the end point. Tabulate your readings as shown below

No.	Vol of 0.5 N $K_2Cr_2O_7$ added 'V' ml	Potential in mv 'E' mv	ΔE	ΔV	$\Delta E / \Delta V$

Graphs:

1. Draw the graph of E against V, determine the end point and hence the normality of $FeSO_4$ $(NH_4)_2SO_4$. $6H_2O$ solution
2. Draw the graph of $\Delta E/\Delta V$ against V, determine the end point and hence the normality of $FeSO_4$ $(NH_4)_2SO_4$. $6H_2O$ solution

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Result:

1. Normality of $FeSO_4$ $(NH_4)_2SO_4$. $6H_2O$ solution (from Graph -1) = _____
2. Normality of $FeSO_4$ $(NH_4)_2SO_4$. $6H_2O$ solution (from Graph -2) = _____

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Time: 3.00 Hrs **Marks: [35+5]**

Exercise No. 22 Potentiometry

Aim: To determine normality of each halide in the mixture of KCl, KBr and KI by potentiometric titration against 0.5N $AgNO_3$ solution.

Requirements

1. Solution containing a mixture of 'x' N KCl, KBr and KI, 0.5 N $AgNO_3$ solution
2. Electrodes: Calomel electrode and Silver electrode

Procedure:

Construct the cell and show it to the Examiner before starting the experiment.

Calibrate the potentiometer with a standard cell

Take 50 ml of the given solution containing a mixture of 'x' N KCl, KBr and KI in a 150 ml beaker Connect the Calomel electrode and Silver electrode to the potentiometer and dip the electrodes in the solution. Keep the instrument on for 15 minutes and determine the potential of this system and note the zero reading

Go on measuring the potential every time after adding 0.5 ml 0.5 N $AgNO_3$ solution from the burette each time. (Stir the solution well in the beaker). Take some more reading even after the end point. Tabulate your readings as shown below. (Since the mixture contains three halides, three end points will be obtained).

No.	Vol of 0.5 N $AgNO_3$ added 'V' ml	Potential in mv 'E' mv	ΔE	ΔV	$\Delta E / \Delta V$

Graphs:

1. Draw the graph of E against V, determine the end point and hence the normality of KCl, KBr and KI in the mixture
2. Draw the graph of $\Delta E/\Delta V$ against V, determine the end point and hence the normality of KCl, KBr and KI in the mixture

Result:

1. Normality of KCl solution (from Graph -1) = _____
2. Normality of KBr solution (from Graph -1) = _____
3. Normality of KI solution (from Graph -1) = _____
4. Normality of KCl solution (from Graph -2) = _____
5. Normality of KBr solution (from Graph -2) = _____
6. Normality of KI solution (from Graph -2) = _____

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PHYSICOCHEMICAL EXERCISE

Time: 3.00 Hrs **Marks: [35+5]**

Exercise No. 23 Surface tension

Aim: To determine the surface tension of the liquids A, B and C by using Drop number method and hence calculate the value of Parachor of liquids and CH_2 group.

Requirements

1. Liquids A, B and C
2. Distilled water

Observation Table

No	Liquid	Specific density	Absolute density	No. of drops of liquid	Surface tension	Parachor
1	A					
2	B					
3	C					
4	H_2O					

Note

- ✓ Systematic and neat entry of all readings will be considered while assigning marks
- ✓ Details of all calculations should be clearly shown
- ✓ Ask the Examiner for all constants

Result

1. Surface tension of liquid A = _____
2. Surface tension of liquid B = _____
3. Surface tension of liquid C = _____
4. Parachor of liquid A = _____
5. Parachor of liquid B = _____
6. Parachor of liquid C = _____
7. Parachor of CH_2 = _____

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Time: 3.00 Hrs **Marks: [35+5]**

Exercise No. 24 Paper Chromatography [Ascending]

Aim: You are given three samples of amino acids and their mixture; separate them by ascending paper chromatography.

Requirements:

- ✓ Amino acid samples and their mixture
- ✓ **Developer:** n-butanol + Acetic acid + H_2O (4 :1: 5) use the upper layer
- ✓ **Spraying Agent:** 1 % Ninhydrin in 95% acetone

Procedure:

Saturate the chromatography chamber with a developer [n-butanol + Acetic acid + H_2O (4:1:5) use the upper layer]

With a pencil draw a starting line on the filter paper at a distance of about 2-3 cms from the edge of the paper.

Using a capillary put two drops of the given sample of amino acids and the mixture on the line. (Keep sufficient space between the spots). Allow it to dry

Now carefully place the filter paper in the Chromatography chamber previously saturated with the developer and allow it to develop for about one or one and a half hour.

Remove the filter paper from the Chromatography chamber; Mark the solvent front i.e. the distance traveled by the solvent and then allow it to dry.

Spray the dried filter paper with the spraying reagent 1 % Ninhydrin in 95% acetone. Allow it to dry; as a results colour spots develop on the filter paper.

Measure the distance of each coloured spot and the solvent front from the starting line. From that calculate R_f value and determine the constituents of the mixture

Result

No.	Sample	Rf Value	Constituent
1	A		
2	B		
3	C		
4	Mixture		

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PHYSICOCHEMICAL EXERCISE

Time: 3.00 Hrs **Marks: [35+5]**

Exercise No. 25 Paper Chromatography [Circular]

Aim: You are given three samples of amino acids and their mixture; separate them by circular paper chromatography.

Requirements:

- ✓ Amino acid samples and their mixture
- ✓ **Developer:** n-butanol + Acetic acid + H_2O (4 :1: 5) use the upper layer
- ✓ **Spraying Agent:** 1 % Ninhydrin in 95% acetone

Procedure:

Saturate the chromatography chamber with a developer [n-butanol + Acetic acid + H_2O (4:1:5) use the upper layer]

Cut a square sheet of Whatman Filter paper as per the size of the Chromatography chamber

With a pencil diagonally draw a line to join the corners of the filter paper. Mark the point of intersection i.e. the centre and draw a circle of radius about 2.5 cms.

Using a capillary put two drops of the given sample of amino acids and the mixture on the diagonal line. (Distance of 2.5 cms from the centre). Allow it to dry

Hold the paper by a wick at the centre and carefully place the filter paper in the Chromatography chamber previously saturated with the developer and allow it to develop for about one or one and a half hour.

Remove the filter paper from the Chromatography chamber; Mark the solvent front i.e. the distance traveled by the solvent and then allow it to dry.

Spray the dried filter paper with the spraying reagent 1 % Ninhydrin in 95% acetone. Allow it to dry; as a results colour spots develop on the filter paper.

Measure the distance of each coloured spot and the solvent front from the starting line. From that calculate R_f value and determine the constituents of the mixture

Result

No.	Sample	Rf Value	Constituent
1	A		
2	B		
3	C		
4	Mixture		

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PHYSICOCHEMICAL EXERCISE

Time: 3.00 Hrs **Marks: [35+5]**

Exercise No. 26 Thin Layer Chromatography

Aim: You are given three samples of amino acids and their mixture; separate them by TLC chromatography.

Requirements:

- ✓ Amino acid samples and their mixture
- ✓ **Developer:** n-butanol + Acetic acid + H_2O (4 :1: 5) use the upper layer
- ✓ **Spraying Agent:** 1 % Ninhydrin in 95% acetone

Procedure:

Saturate the chromatography chamber with a developer [n-butanol + Acetic acid + H_2O (4:1:5) use the upper layer]

With a pencil draw a starting line on the TLC plate at a distance of about 2-3 cms from the edge of the TLC plate.

Using a capillary put two drops of the given sample of amino acids and the mixture on the line. (Keep sufficient space between the spots). Allow it to dry

Now carefully place the TLC plate in the Chromatography chamber previously saturated with the developer and allow it to develop for about one or one and a half hour.

Remove the TLC plate from the Chromatography chamber; Mark the solvent front i.e. the distance traveled by the solvent and then allow it to dry.

Spray the dried TLC plate with the spraying reagent 1 % Ninhydrin in 95% acetone. Allow it to dry; as a results colour spots develop on the TLC plate.

Measure the distance of each coloured spot and the solvent front from the starting line. From that calculate R_f value and determine the constituents of the mixture

Result

No.	Sample	Rf Value	Constituent
1	A		
2	B		
3	C		
4	Mixture		

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PHYSICOCHEMICAL EXERCISE

Time: 3.00 Hrs **Marks: [35+5]**

Exercise No. 27 Paper Chromatography [Ascending]

Aim: You are given three samples of metal ions and their mixture; separate them by ascending paper chromatography.

Requirements:

- ✓ Metal ions samples and their mixture
- ✓ **Developer:** Acetone + Ethyl acetate + 6N HCl (9:9:2)
- ✓ **Spraying Agent:** 0.1 % Rubinic acid in Acetone

Procedure:

Saturate the chromatography chamber with a developer [Acetone + Ethylacetate + 6N HCl (9:9:2)]

With a pencil draw a starting line on the filter paper at a distance of about 2-3 cms from the edge of the paper.

Using a capillary put two drops of the given sample of metal ion and the mixture on the line. (Keep sufficient space between the spots). Allow it to dry

Now carefully place the filter paper in the Chromatography chamber previously saturated with the developer and allow it to develop for about one or one and a half hour.

Remove the filter paper from the Chromatography chamber; Mark the solvent front i.e. the distance traveled by the solvent and then allow it to dry.

Pass ammonia vapours over the dried filter paper and then spray it with the spraying reagent 0.1 % Rubinic acid in Acetone. Allow it to dry; as a results coloured spots develop on the filter paper.

Measure the distance of each coloured spot and the solvent front from the starting line. From that calculate R_f value and determine the constituents of the mixture

Result

No.	Sample	Rf Value	Constituent
1	A		
2	B		
3	C		
4	Mixture		

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PHYSICOCHEMICAL EXERCISE

Time: 3.00 Hrs **Marks: [35+5]**

Exercise No. 28 Paper Chromatography [Circular]

Aim: You are given three samples of metal ions and their mixture; separate them by circular paper chromatography.

Requirements:

- ✓ Metal ions samples and their mixture
- ✓ **Developer:** Acetone + Ethylacetate + 6N HCl (9:9:2)
- ✓ **Spraying Agent:** 0.1 % Rubinic acid in Acetone

Procedure:

Saturate the chromatography chamber with a developer [Acetone + Ethylacetate + 6N HCl (9:9:2)]

Cut a square sheet of Whatman Filter paper as per the size of the Chromatography chamber

With a pencil diagonally draw a line to join the corners of the filter paper. Mark the point of intersection i.e. the centre and draw a circle of radius about 2.5 cms.

Using a capillary put two drops of the given sample of metal ions and the mixture on the diagonal line. (Distance of 2.5 cms from the centre). Allow it to dry

Hold the paper by a wick at the centre and carefully place the filter paper in the Chromatography chamber previously saturated with the developer and allow it to develop for about one or one and a half hour.

Remove the filter paper from the Chromatography chamber; Mark the solvent front i.e. the distance traveled by the solvent and then allow it to dry.

Pass the dried filter paper over ammonia vapours and then spray it with the spraying reagent 0.1 % Rubinic acid in Acetone. Allow it to dry; as a results coloured spots develop on the filter paper.

Measure the distance of each coloured spot and the solvent front from the starting line. From that calculate R_f value and determine the constituents of the mixture

Result

No.	Sample	Rf Value	Constituent
1	A		
2	B		
3	C		
4	Mixture		

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Chemistry Practical Examination

Centre : _____

Place : _____

The purpose of the practical examination, the division of the batches and distribution of the practical work, candidates are divided into the following groups:

DATES	SEAT NOS. OF BATCH		GROUPS	
	FROM	TO	'A' GROUP	'B' GROUP

DAY	TIME	'A' GROUP	'B' GROUP
FIRST DAY	10.00 am to 1.00 pm	Inorganic Qualitative Analysis (Viva)	Physicochemical(viva)
	2.00 to 5.00 pm	Physicochemical(viva)	Inorganic Qualitative Analysis (Viva)
SECOND DAY	10.00 am to 1.00 pm	Organic Synthesis	Project
	2.00 to 5.00 pm	Project	Organic Synthesis

Note:

**** The arrangement of above practical session will be finalize by concerned examiner.***

The Candidates are informed to be present at he examination centre at least 15 mins before the commencement of the Examination. The following are to be brought by the candidate at the time of the Examinations:

- Certified Journal
- Fee Receipt
- College Identity Card
- Apron / Lab Coat
- Calculator
- Match Box
- Small Knife
- Cloth Duster

Senior Examiner
TYBSc Chemistry Practical Examination
March / April / 20