Bhakta Kavi Narsinh Mehta University

BSc Semester V
Chemistry Practicals
With effect from June-2018
[CBCS]

BHAKTA KAVI NARSINH MEHTA UNIVERSITY
BSc Semester V
Chemistry Practical Examination

ORGANIC SEPARATION

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

Exercise No. 1

Organic Separation

In the container bearing the **Capsule No ……** you are given a mixture of two organic compounds with distinct chemical and physical properties. The given mixture belongs to one of the types given below.

By preliminary observations/ tests decide the type of the mixture.

Get the signature of the Examiner before proceeding to separate the mixture.

Separate the two components of the mixture and identify each components of the mixture.

Perform all the tests and identify each component of the mixture

Keep both the purified components for inspection by the Examiners

Type of Mixture:…….

Final result:

No.	Nature	Elements	Functional Group	MP/BP	Name	Structural Formula
1						
2						

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Chemistry Practical Examination
INORGANIC VOLUMETRIC ANALYSIS

Time: 3.00 Hrs **Marks: 30**

Iodometry / Iodimetry

Exercise No. 2

Estimation of Cu

Aim: To determine by iodometric method the amount of Cu^{+2} and $CuSO_4.5H_2O$ in the given $CuSO_4.5H_2O$ using 0.05N $Na_2S_2O_3.5H_2O$ solution.

Requirements: Solid $Na_2S_2O_3.5H_2O$, 10% KI, 2N Na2CO3 solution, 2N acetic acid , fresh starch solution.

Procedure:

Step –I: Preparation of 0.05 M $Na_2S_2O_3.5H_2O$ solution

Weigh 3.1025 gm $Na_2S_2O_3.5H_2O$ in a previously weighed watch glass and dissolve in distilled water making up the volume to 250 ml in a measuring flask. Fill this 0.05 M $Na_2S_2O_3.5H_2O$ solution in a burette.

Step –II: Estimation of Cu^{+2} and $CuSO_4.5H_2O$

Dilute, with distilled water, the given acidic copper sulphate solution to 250 ml in a measuring flask. Take 25 ml of the diluted solution in a conical flask and add dilute Na_2CO_3 or NH_4OH drop wise with constant stirring. (To remove mineral acid) Continue adding 2 N Na_2CO_3 till complete precipitation (i.e. till the ppts no longer dissolve on stirring)

Now add 2N CH_3COOH to this solution till ppts completely dissolve. The solution turns clear blue. Add 20 ml 10% KI solution and immediately titrate the liberated iodine against $Na_2S_2O_3$ solution filled in the burette; when the solution in the flask turns light yellow add approximately 1 ml fresh starch solution (the solution turns blue). Continue adding $Na_2S_2O_3$ solution from the burette till the solution turns white (stable). The white ppt obtained is Cu_2I_2. Note the burette reading.

Calculations:

		$2CuSO_4.5H_2O$	≡	I_2	≡	$2Na_2S_2O_3.5H_2O$
Cu^{+2}	≡	$CuSO_4.5H_2O$	≡	I	≡	$Na_2S_2O_3.5H_2O$
63.5 g	≡	249.7 g	≡	127 g	≡	248.2 g

Result

1. 25ml dil. $CuSO_4.5H_2O$ solution requires ……. ml. 0.05 M $Na_2S_2O_3$ $5H_2O$ solution

2. The amount of Cu^{+2} in the given solution = ……. g

3. Amount of $CuSO_4.5H_2O$ in the solution = …… g

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INORGANIC VOLUMETRIC ANALYSIS

Time: 3.00 Hrs **Marks: 30**

Iodometry / Iodimetry

Exercise No. 3

Estimation of As

Aim: To determine by iodimetric method the amount of As^{+3} and As_2O_3 in the given As_2O_3 using 0.05M $Na_2S_2O_3.5H_2O$ solution.

Requirements: Solid $Na_2S_2O_3.5H_2O$, 0.05N I_2 solution, solid $NaHCO_3$, fresh starch solution.

Procedure:

Preparation of 0.05 M $Na_2S_2O_3.5H_2O$ solution

Weigh 3.1025 gm $Na_2S_2O_3.5H_2O$ in a previously weighed watch glass and dissolve in distilled water making up the volume to 250 ml. Fill this 0.05 M $Na_2S_2O_3.5H_2O$ solution in a burette.

Part-I: Standardization of Iodine solution

Take 25 ml of the given iodine solution and titrate it against $Na_2S_2O_3.5H_2O$ solution filled in the burette. When the solution turns yellow add approximately 1 ml starch solution.The solution turns blue. Add $Na_2S_2O_3.5H_2O$ solution from the burette with constant stirring till the solution turns colourless. Note the burette reading.

Part –II: Estimation of As_2O_3

Note: Arsenic salts are highly poisonous; hence instead of using a pipette, use a burette for taking Arsenic. Avoid contact of arsenic salts if skin is cut or bruised.

Dilute with distilled water the given As_2O_3 solution to 250 ml in a measuring flask. Fill this diluted As_2O_3 solution in the burette.

Take 25 ml I_2 solution in a conical flask; add approximately 50ml (2 test tubes) distilled water and approximately 3 gms pure $NaHCO_3$ (solid). Shake the solution to dissolve the solid $NaHCO_3$. Titrate this solution against As_2O_3 solution filled in the burette. When the solution in the flask turns light yellow add approximately 1 ml fresh starch solution (the solution turns blue). Continue adding As_2O_3 solution from the burette till the solution turns colourless. Note the burette reading.

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Calculation: Estimation of Arsenic

1000 ml 1N I_2 solution ≡ 49.45 g As_2O_3 ≡ 37.45 g As^{+3}

Result

1. Normality of I_2 solution = ……. N
2. Volume of '*X* 'N I_2 solution required for 25 ml diluted As_2O_3 solution is ……. ml
3. Amount of As_2O_3 in the given solution is …… g
4. Amount of As^{+3} in the given solution is …… g

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INORGANIC VOLUMETRIC ANALYSIS

Time: 3.00 Hrs **Marks: 30**

Complexometric

Exercise No. 4(a)

Estimation of Ni

[Direct Titration Method using Murexide as an indicator]

Aim: To determine the amount of Ni^{+2} in the given $NiSO_4.7H_2O$ solution using 0.01 M EDTA solution.

Requirements: EDTA disodium salt (solid), 1M NH_4Cl solution, liquor ammonia, distilled water, Murexide indicator (solid mixture from or freshly decanted solution).

Procedure:

Step –I: Preparation of 0.01 M EDTA disodium salt solution

Dissolve 0.9306 g EDTA salt in distilled water and make up the volume to 250 ml in a measuring flask. Fill this solution in the burette.

Step –II: Estimation of Ni^{2+} [Using Murexide as an indicator]

Dilute with distilled water the given Ni^{2+} solution to 250ml in a measuring flask. Take 25 ml of the diluted Ni^{+2} solution, add 150 ml distilled water and add about 40-50 mg (approximately 1 pinch) murexide indicator (indicator + solid KNO_3 mixture) and shake vigorously. Now add 10 ml Buffer solution (Mixture of 1 M NH_4Cl solution and 1 M Ammonia) till distinct yellow colour is obtained. Titrate the solution against 0.01M EDTA disodium salt solution with constant stirring. At the end point the yellow colour of the solution turns violet. Note the burette reading

Calculations

1000 ml 1M EDTA-salt ≡ 58.7 g Ni^{2+} ≡ 280.8 g $NiSO_4\ 7H_2O$

Result

1. Volume of 0.01M EDTA solution required for 25 ml diluted Ni^{2+}solution is …….. ml
2. Amount of Ni^{2+} in the given solution is …….. g
3. Amount of $NiSO_4.\ 7H_2O$ in the given solution is …… g

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Inorganic Volumetric Analysis

Time: 3.00 Hrs **Marks: 30**

Complexometric

Exercise No. 4(b)

Estimation of Ni

Direct Titration Method using Bromopyrogallol Red as an indicator

Aim: To determine the amount of Ni^{+2} in the given $NiSO_4.7H_2O$ solution using 0.01 M EDTA solution.

Requirements: EDTA disodium salt (solid), Buffer solution, Bromopyrogallol Red indicator, distilled water

Buffer solution: Mixture of equal volume of 1M NH_4Cl solution and 1M NH_3 solution.

Procedure:

Step –I: Preparation of 0.01 M EDTA disodium salt solution

Dissolve 0.9306 g EDTA salt in distilled water and make up the volume to 250 ml in a measuring flask. Fill the solution in the burette.

Step –II: Estimation of Ni^{2+} [Using Bromopyrogallol Red as an indicator]

Dilute with distilled water the given Ni^{2+} solution to 250 ml in a measuring flask. Take 25 ml of the diluted Ni^{+2} solution, add 150 ml distilled water and 1 ml Bromopyrogallol Red indicator. Now add 10 ml buffer solution and shake vigourously. Titrate the solution against 0.01M EDTA disodium salt solution from the burette. At the end point the blue colour of the solution turns red. Note the burette reading

Calculations

1000 ml 1M EDTA-salt ≡ 58.7 g Ni^{2+} ≡ 280.8 g $NiSO_4$ $7H_2O$

Result

1. Volume of 0.01M EDTA solution required for 25 ml diluted Ni^{2+}solution is …….. ml
2. Amount of Ni^{2+} in the given solution is …….. g
3. Amount of $NiSO_4.7H_2O$ in the given solution is ……. g

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Inorganic Volumetric Analysis

Time: 3.00 Hrs **Marks: 30**

Complexometric

Exercise No. 4(c)

Estimation of Ni

[Back Titration Method using Eriochrome Black T indicator]

Aim: To determine the amount of Ni^{+2} in the given $NiSO_4.7H_2O$ solution using 0.01 M EDTA solution.

Requirements: EDTA disodium salt (solid), Buffer solution pH 10, Eriochrome Black T indicator, 0.01 M $MgSO_4$ solution, distilled water

Procedure

Step –I: Preparation of 0.01 M EDTA disodium salt solution

Dissolve 0.9306 g EDTA salt in distilled water and make up the volume to 250 ml

Step –II: Preparation of 0.01 M (exact) $MgSO_4$ solution

Dissolve 0.616 g $MgSO_4$ in distilled water and make up the volume to 250 ml

Step –III: Blank Reading

Fill the prepared 0.01 M $MgSO_4$ solution in the burette. Take 25 ml EDTA solution in a conical flask add 25 ml distilled water and 3 ml buffer solution. Add 2-3 drops Eriochrome Black T indicator and titrate against 0.01 M $MgSO_4$ solution from the burette. At the end point the blue coloured solution turns wine red.

Step – IV: Estimation of Ni^{2+}

Dilute with distilled water the given Ni^{2+} solution to 250ml in a measuring flask. Take 25 ml of the diluted Ni^{+2} solutions (if the solution is acidic neutralize by adding NH_4OH – then heat to remove excess NH_3; cool the flask under tap water). Add a definite volume (25 ml) of 0.01M EDTA solution. Now add 25 ml distilled water, 3 ml buffer solution and 2-3 drops Eriochrome Black T indicator. Shake and titrate the solution against $MgSO_4$ solution from the burette. At the end point the blue colour of the solution turns red. Note the burette reading

Calculation

1000 ml 1M EDTA-salt ≡ 58.7 g Ni^{2+} ≡ 280.8 g $NiSO_4$ $7H_2O$

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Result

1. Volume of 0.01M EDTA solution required for 25 ml diluted Ni^{2+} solution is …… ml
2. Amount of Ni^{2+} in the given solution is …….. g
3. Amount of $NiSO_4.7H_2O$ in the given solution is …….. g

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Inorganic Volumetric Analysis

Time: 3.00 Hrs **Marks: 30**

Complexometric

Exercise No. 5

Estimation of Pb and Mg

Aim: To determine the amount of Mg^{+2} and Pb^{+2} in the given solution containing a mixture of Mg^{+2} and Pb^{+2} using 0.01 M EDTA solution

Requirements: EDTA– disodium salt (solid), distilled water, indicators Eriochrome Black-T and Xylenol Orange, 10% hexamine solution or solid powder, buffer solution (pH = 10).

Procedure:

Step -I: Preparation of 0.01M EDTA-disodium salt solution:

Dissolve the 0.9306 gm EDTA in distilled water & make up the total volume to 250 ml

Step- II: Dilute with distilled water the given solution containing a mixture of Mg^{+2} and Pb^{+2} to 250 ml with distilled water.

Step –III: Determination of the total amount of Pb^{+2} and Mg^{+2} in the given mixture

Take 25 ml of the diluted mixture in a conical flask. Add 50 ml distilled water and 5 ml (definite volume) of buffer solution (pH=10). Now add 3-4 drops Eriochrome Black-T as an indicator and titrate against 0.01M EDTA solution from the burette with constant stirring. At the end point the wine red coloured solution turns blue. Note the burette reading.

Step – IV: Determination of the amount of Pb^{+2} in the solution

Take 25ml diluted mixture in a conical flask and add 50ml distilled water and 3-4 drops Xylenol Orange indicator. Now add 3 ml dil. HNO_3 (till solution turns yellow). Then slowly add 5 ml 10% hexamine solution or the solid powder with constant shaking till the yellow solution turns red (pH=6). Now titrate the solution against 0.01M EDTA solution from the burette. At the end point the red solution turns distinct yellow. Note the burette reading.

Calculations

1000 ml 1M EDTA-salt ≡ 207.2 g Pb^{2+} ≡ 331.2 g $Pb(NO_3)_2$

1000 ml 1M EDTA-salt ≡ 24.3 gm Mg^{2+} ≡ 148.3 gm $Mg(NO_3)_2$

Result

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1. Amount of Pb^{+2} in the given solution is ……. g
2. Amount of $Pb(NO_3)_2$ in the given solution is ….. g
3. Amount of Mg^{+2} in the given solution is …… g
4. Amount of $Mg(NO_3)_2$ in the given solution is …….. g

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Chemistry Practical Examination
Inorganic Volumetric Analysis

Time: 3.00 Hrs **Marks: 30**

Complexometric

Exercise No. 6

Estimation of the amount of Ca and Zn

Aim: To determine the amount of Ca^{+2} & Zn^{+2} in the given solution containing a mixture of $CaCl_2\,2H_2O$ and $ZnCl_2$ using 0.01 M EDTA solution

Requirements: EDTA – disodium salt (solid), distilled water, indicators Eriochrome Black-T and Xylenol Orange, 10% hexamine solution or solid powder, buffer solution (pH = 10).

Procedure:

Step -I: Preparation of 0.01M EDTA-disodium salt solution:

Dissolve the 0.9306 gm EDTA in distilled water & make up the total volume to 250 ml

Step- II: Dilute with distilled water the given solution containing a mixture of Ca^{+2} and Zn^{+2} to 250 ml with distilled water.

Step –III: Determination of the total amount of Ca^{+2} and Zn^{+2} in the given mixture

Take 25 ml of the diluted mixture in a conical flask. Add 25 ml distilled water and 5 ml (definite volume) of buffer solution (pH= 10). Now add 3-4 drop Eriochrome Black-T as an indicator and titrate against 0.01M EDTA solution from the burette with constant stirring. At the end point the wine red coloured solution turns blue. Note the burette reading.

Step – IV: Determination of the amount of Zn^{+2} in the solution

Take 25ml diluted mixture in a conical flask and add 25 ml distilled water and 3-4 drops Xylenol Orange indicator (solution turns yellow). Now add 5 ml 10% hexamine solution drop wise or slowly add the solid powder with constant shaking till the yellow solution turns red (pH=6). Now titrate the solution against 0.01M EDTA solution from the burette. At the end point the red solution turns distinct yellow. Note the burette reading.

Calculations

1000 ml 1M EDTA-salt ≡ 65.4 g Zn^{+2} ≡ 136.4 g $ZnCl_2$

1000 ml 1M EDTA-salt ≡ 40 gm Ca^{2+} ≡ 147 gm $CaCl_2.2H_2O$

Result

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1. Amount of Zn^{+2} in the given solution is …….. g
2. Amount of $ZnCl_2$ in the given solution is …….. g
3. Amount of Ca^{+2} in the given solution is …… g
4. Amount of $CaCl_2.2H_2O$ in the given solution is …… g

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Inorganic Volumetric Analysis

Time: 3.00 Hrs **Marks: 30**

Complexometric

Exercise No. 7

Estimation of the amount of Fe and Cr

Aim: To determine the amount of Fe^{+3} & Cr^{+3} in the given solution containing a mixture of $FeCl_3 \cdot 6H_2O$ and $CrCl_3 \cdot 6H_2O$ using 0.01 M $Pb(NO_3)_2$ solution

Requirements: EDTA – disodium salt (solid), distilled water, indicators Xylenol Orange, 10% hexamine solution or solid powder, 0.01 M $Pb(NO_3)_2$.

Procedure:

Step -I: Preparation of 0.01M EDTA-disodium salt solution:

Dissolve 0.9306 gm EDTA in distilled water & make up the total volume to 250 ml.

Step – II: Preparation of 0.01M $Pb(NO_3)_2$ solution:

Dissolve the 0.828 gm $Pb(NO_3)_2$ in distilled water & make up the total volume to 250 ml.

Step- III: Dilute with distilled water the given solution containing a mixture of Fe^{+3} and Cr^{+3} to 250 ml with distilled water.

Step –III: Determination of the total amount of Fe^{+3} in the given mixture

Take 25 ml of the diluted solution of mixture containing Fe^{+3} and Cr^{+3} in a conical flask. Add 50 ml distilled water and 25 ml 0.01M EDTA solution. Now add 12-15 ml 10% hexamine solution (pH between 5-6 use pH paper) and 3-4 drops xylenol orange as an indicator (solution turns yellow) and titrate against 0.01M $Pb(NO_3)_2$ solution from the burette with constant stirring. At the end point the yellow coloured solution turns first orange then red-violet**. (Do not throw away this solution and use it for part-II)** Note the burette reading (A ml).

Step – IV: Determination of the amount of Cr^{+3} in the given mixture

To the red-violet solution obtained in part I, add 25 ml 0.01 M EDTA solution and 10 ml 1N HNO_3 solution (pH should be between 1 & 2). Boil the solution for 15 to 20 min, cool and add 25 ml distilled water. Now add 10 ml 10% hexamine solution and 3-4 drops xylenol orange as an indicator. The solution turns yellow. Now titrate the solution against 0.01M $Pb(NO_3)_2$ solution from the burette with constant stirring. At the end point the yellow solution will turn orange and then red-violet. Note the burette reading (B ml).

Calculations

1000 ml 1M EDTA-salt ≡ 55.83 g Fe^{+3} ≡ 270.3 g $FeCl_3.6H_2O$

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1000 ml 1M EDTA-salt ≡ 52 g Cr^{+3} ≡ 266.5 g $CrCl_3.6H_2O$

Result

1. Amount 0.01M EDTA solution used for Fe^{+3} in 25 ml diluted solution of the mixture _____ (25-A) ml
2. Amount 0.01M EDTA solution used for Cr^{+3} in 25 ml diluted solution of the mixture _____(25-B) ml
3. Amount of Fe^{+3} in the given solution is …….. g
4. Amount of Cr^{+2} in the given solution is …… g

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Inorganic Volumetric Analysis

Time: 3.00 Hrs **Marks: 30**

Redox

Exercise No. 8

Estimation of NO_2^{-1} ions

Aim: To determine, by redox titration, the amount of NO_2^{-1} ions in the given $NaNO_2$ solution, with the help of 0.1N $KMnO_4$ solution.

Requirement: Solid $KMnO_4$, 2N H_2SO_4

Procedure:

Step – I : Preparation of 0.1N $KMnO_4$ solution.

Dissolve 0.79gm of $KMnO_4$ in distilled water and make the volume to 250 ml.

Step –II: Estimation of NO_2^{-1}

Dilute, with distilled water, the given $NaNO_2$ solution to 250ml in a measuring flask. Fill this solution in the burette.

Now take 25ml of 0.1 N $KMnO_4$ solution in a beaker. Add 25ml of 2N H_2SO_4 and 25ml distilled water. Heat it upto 50^oC and then titrate against $NaNO_2$ from burette. [Precaution: During titration take care to see that the jet of the burette is dipped in the beaker used for titration] At the endpoint the light pink colour becomes colourless. Note the burette reading.

Calculation

1000ml 1N $KMnO_4$ ≡ 23 gm NO_2^{-1} ≡ 34.5 gm $NaNO_2$

Result

1.Amount of NO_2^{-1} in the given solution = …… gm

2.Amount of $NaNO_2$ in the given solution = …… gm

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Inorganic Volumetric Analysis

Time: 3.00 Hrs **Marks: 30**

Water analysis

Exercise No. 9

Determination of Chloride

Aim: To determine the amount of chloride in the given sample of water using '*X*' N $AgNO_3$ solution.

Requirements: '*X*' N $AgNO_3$ solution, 0.02 N NaCl, K_2CrO_4 indicator solution

Procedure

Standardization of $AgNO_3$

Take 10 ml 0.02 N NaCl in a porcelain dish, add 2 drops K_2CrO_4 indicator. Titrate, with constant stirring, against '*X*' N $AgNO_3$ solution until a slight reddish colour ppt is obtained. Note the burette reading.

Estimation of Cl^{1-} ions in the water sample (tap water)

Take 25 ml of the water sample (tap water) in a porcelain dish, add 2 drops K_2CrO_4 indicator. Titrate, with constant stirring, against '*X*' N $AgNO_3$ solution from the burette until a slight reddish colour ppt is obtained. Note the burette reading.

Calculations

1000 ml 1N $AgNO_3$ ≡ 35.5 gms Chloride

Result:

1. The Normality of the given $AgNO_3$ solution is ……..... N
2. Volume of $AgNO_3$ solution required for 25 ml sample of water is …….. ml
3. Amount of chloride in the given sample of water is …….... mg/litre

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Inorganic Volumetric Analysis

Time: 3.00 Hrs **Marks: 30**

Exercise No. 10

Determination of Purity of $NaHCO_3$

Aim: To determine the purity of $NaHCO_3$ in the given sample of antacid by volumetric analysis.

Requirements: Sample of antacid, 0.05 N HCl, methyl orangeindicator.

Procedure:

Method: Dissolve the given sample of antacid in distilled water and makeup volume to 250ml. If necessary filter so as to get a clear solution.

From that pipette out 25ml solution in conical flask and add 2-3 drops methyl orange indicator and titrate against 0.05N HCl solution. At the end point color change will be from yellow to orange. (A) ml.

Calculations:

Amount of 0.05N HCl solution used for 25ml sample solution = _____(A) ml.

For 250ml diluted solution used up amount of 0.05N HCl = A × 10 = ____ (B) ml

Ask the Examiner for the weight of the sample of antacid

$$\text{Practically equivalent wt. of antacid (Ep)} = \frac{\text{Wt. of antacid} \times 1000}{\text{'B' ml} \times \text{'N' of HCl}}$$

Theoretically equivalent wt. of antacid (Ec) = 84 gm for 100 % pure

$$\text{Practically equivalent wt. of antacid (Ep)} = \frac{Ep \times 100}{Ec}$$

= 'X'

= 'X' % purity of antacid.

Result:

1. Amount of 0.05N HCl used for the neutralization of antacid= ____ (A) ml
2. Practically equivalent weight (Ep) of antacid = _____ (Ep)
3. Purity of antacid = ____ (X) %.

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Chemistry Practical Examination
Physico Chemical Exercise

Time: 3.00 Hrs **Marks: [30 +5]**

Exercise No. 11 Conductometry

Aim: Determine the cell constant of the given conductivity cell by using 0.1N KCl solution and also determine normality of the given HCl solution by conductometric titration

Requirements: 0.5N NaOH, '*X*'N HCl, solid KCl.

Procedure:

1. Prepare 100ml 0.1N KCl solution and use it to determine the cell constant of the conductivity cell.

2. Take 25ml of the given HCl solution in a beaker (or use the volume as per the capacity of the cell) and dip the conductivity cell in it. Determine the conductivity of the cell and also determine the given HCl and the conductivity after each addition of 0.5 ml NaOH, till the neutralization point is reached. Take similar readings after the neutralization point is reached.

Observation No	Volume of added 0.5N NaOH solution [V ml]	Conductance 'C' mho
1	0.0 ml	
2	0.5 ml	
3	1.0 ml	
4	1.5 ml	
5	2.0 ml	
6	2.5 ml	
7	3.0 ml	
8	3.5 ml	
9	4.0 ml	
10	4.5 ml	
11	5.0 ml	
12	5.5 ml	
13	6.0 ml	
14	6.5 ml	
15	7.0 ml	
16	7.5 ml	

17	8.0 ml	
18	8.5 ml	
19	9.0 ml	
20	9.5 ml	

Graph: Plot the graph of conductance against the volume of alkali added and use it to determine the point of neutralization. Calculate the normality of the given HCl solution.

Result: 1. Cell constant of the conductivity cell = ---------

2. Normality of HCl = …….

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Chemistry Practical Examination
Physico Chemical Exercise

Time: 3.00 Hrs **Marks: [30 +5]**

Exercise No. 12 Conductometry

Aim: Determine the cell constant of the conductivity cell by using 0.1N KCl solution and determine the concentration of each component in the given mixture of HCl + CH_3COOH in terms of normality by conductometric tiration.

Requirements: 0.5N NaOH, *X*N HCl, 'Y' N CH_3COOH, 0.1 N KCl

Procedure: Take 25ml of the given '*X'* N HCl + 25 ml '*Y* 'N CH_3COOH solution in a beaker (or use the volume as per the capacity of the cell) and dip conductivity cell in it. Determine the conductivity of the given mixture of HCl + CH_3COOH solution. Similarly take more readings by adding 0.5 ml 0.5 N NaOH solution. Take about 30 readings. Continue the titration and take the reading till it gives the conductance for the strong alkali

Observation No	Vol. of added 0.5N NaOH solution [V ml]	Conductance 'C' mho
1	0.0 ml	
2	0.5 ml	
3	1.0 ml	
4	1.5 ml	
5	2.0 ml	
6	2.5 ml	
7	3.0 ml	
8	3.5 ml	
9	4.0 ml	
10	4.5 ml	
11	5.0 ml	
12	5.5 ml	
13	6.0 ml	
14	6.5 ml	
15	7.0 ml	
16	7.5 ml	
17	8.0 ml	
18	8.5 ml	
19	9.0 ml	
20	9.5 ml	

Graph: Plot the graph of volume of alkali added against conductance and use it to determine the point of neutralization.

Calculate the normality of each component in the given mixture of HCl + CH_3COOH.

Result

1. Cell constant of the conductivity cell = ----------
2. Normality of HCl = …….
3. Normality of CH_3COOH = …….

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Chemistry Practical Examination
Physico Chemical Exercise

Time: 3.00 Hrs **Marks: [30 +5]**

Exercise No. 13 Conductometry

Aim: Determine the Cell constant of the given conductivity cell and determine the normality of the given CH_3COOH by conductometric titration

Requirements: 0.5N NaOH, '*X'* N CH_3COOH, Prepare 0.1 N KCl solution

Procedure: Take 25ml of the given '*X'* N CH_3COOH solution in a beaker (or use the volume as per the capacity of the cell) and dip conductivity cell in it. Determine the conductivity of the given *X* N CH_3COOH solution. Similarly take more readings by adding 0.5 ml 0.5 N NaOH solution. Take about 30 readings.

Observation No	Vol of added 0.5N NaOH solution [V ml]	Conductance 'C' mho
1	0.0 ml	
2	0.5 ml	
3	1.0 ml	
4	1.5 ml	
5	2.0 ml	
6	2.5 ml	
7	3.0 ml	
8	3.5 ml	
9	4.0 ml	
10	4.5 ml	
11	5.0 ml	
12	5.5 ml	
13	6.0 ml	
14	6.5 ml	
15	7.0 ml	
16	7.5 ml	
17	8.0 ml	
18	8.5 ml	
19	9.0 ml	
20	9.5 ml	

Graph: Plot the graph of conductance against the volume of alkali added and use it to determine the point of neutralization. Calculate the normality of the given CH_3COOH solution

Result

1. Cell constant of the conductivity cell = ----------
2. Normality of CH_3COOH = ……..

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Time: 3.00 Hrs **Marks: [30 +5]**

Exercise No. 14 Conductometry

Aim: Determine the Cell constant of the given conductivity cell and also determine normality of the given Ni^{+2} solution by conductometric titration.

Requirements: 0.1 M EDTA, 0.1 M $NiSO_4$

Procedure: Take 50 ml of the given 0.1 M $NiSO_4$ solution in a beaker (or use the volume as per the capacity of the cell) and dip conductivity cell in it. Determine the conductivity of the given $NiSO_4$ solution and also determine the conductivity after each addition of 0.5 ml EDTA. Take similar readings after the end point is reached.

Observation No	Vol of added 0.1 M EDTA solution [V ml]	Conductance 'C' mho
1	0.0 ml	
2	0.5 ml	
3	1.0 ml	
4	1.5 ml	
5	2.0 ml	
6	2.5 ml	
7	3.0 ml	
8	3.5 ml	
9	4.0 ml	
10	4.5 ml	
11	5.0 ml	
12	5.5 ml	
13	6.0 ml	
14	6.5 ml	
15	7.0 ml	
16	7.5 ml	
17	8.0 ml	
18	8.5 ml	
19	9.0 ml	
20	9.5 ml	

Graph: Plot the graph of conductance against the volume of EDTA added and use it to determine the point of end point. Calculate the normality of the given $NiSO_4$ solution.

Result

1. Cell constant of the conductivity cell = ----------
2. Normality of $NiSO_4$ = …… N

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Time: 3.00 Hrs **Marks: [30 +5]**

Exercise No. 15 Conductometry

Aim: To determine the cell constant of the conductivity cell and the normality of '*X'* N $AgNO_3$ using 0.5 N NaCl by conductometric titration

Requirements: 0.5N NaCl , '*X'* N $AgNO_3$, Solid KCl

Method: Prepare 100 ml solution of 0.1 N KCl and use it to determine the cell constant of the conductivity cell.

Take 25 ml '*X'* N $AgNO_3$ solution in a beaker and dip the conductivity cell in it. Titrate the solution against 0.5 N NaCl solution. Similarly take more readings by adding 0.5 ml 0.5 N NaCl solution. After the neutralization point take more reading to determine the end point accurately.

Observation Table:

Observation No.	Vol of added 0.5N NaOH solution [V ml]	Conductance 'C' mho
1	0.0 ml	
2	0.5 ml	
3	1.0 ml	
4	1.5 ml	
5	2.0 ml	
6	2.5 ml	
7	3.0 ml	
8	3.5 ml	
9	4.0 ml	
10	4.5 ml	
11	5.0 ml	
12	5.5 ml	
13	6.0 ml	
14	6.5 ml	
15	7.0 ml	
16	7.5 ml	
17	8.0 ml	

18	8.5 ml	
19	9.0 ml	
20	9.5 ml	

Graph: Plot the graph of conductance against the volume of NaCl added and use it to determine the point of neutralization. Calculate the normality of the given $AgNO_3$ solution

Result

1. Cell Constant = _____

2. Normality of $AgNO_3$ = _____ N

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Time: 3.00 Hrs **Marks: [30 +5]**

Exercise No. 16 Thermodynamics

Aim: To calculate entropy of vapourisation (ΔSv) of a given liquid by Kinetic approach i.e. from the graph of log (1/time) against (1/temperature).

Requirements

Benzene / $CHCl_3$ / CCl_4 / n-hexane

Constant temperature bath

Procedure

With the help of a micropipette, take 0.1ml of the given liquid [Benzene / $CHCl_3$ / CCl_4 / n-hexane] and transfer it to a clean evaporating dish kept floating in a constant temperature bath. Note the time for complete evaporation of the liquid. Repeat the process atleast thrice.

Similarly perform the experiment at four different temperatures (with a difference of 5^0C)

Tabulate your readings as follows:

Temperature for Experiment = T^0C

No	Temp ^{0}C	Temp T ^{0}K	1/T Express in the power of 10^{-3}	Time in seconds (t)				Log 1/t	ΔH_v kcal/mol
				t_1	t_2	t_3	Average 't' sec		
1									
2									
3									
4									
5									

Note: Ask the Examiner for the boiling point of the given liquid

Graph

- ✓ Plot the graph of log 1/t against 1/T
- ✓ From the slope determine the Heat of Vapourization and hence calculate Entropy of Vapourization

Result

1. Heat of Vapourization of the given liquid is _____Kcal/mol deg K

2. Entropy of Vapourization of the given liquid is _____ cal/mol deg K

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Exercise No. 17 Refractometry

Aim: Determine the refractive index of given liquid A, B, C, D. and thus calculate the specific refractive index and molecular refractive index.

Procedure: Clean specific gravity bottle with distilled H_20, alcohol, ether and dry with dryer.

Weigh given liquid A, B, C & D and water and find out specific density and absolute density. Arrange the instrument in sunlight expose the prism and clean with water, alcohol and ether in that order and then with cotton. Lastly clean the prism with the liquid to be used for the experiment. Then put 2-3 drops of that liquid on prism and close prism. Insert the liquid by glass tube or pipette through the hole on the side of prism box. Adjust the mirror in such a manner that by the reflection of light rays maximum surface of prism is brightened. Rotate the prism with the screw so half of the circle gets darken remaining becomes brighter which can be observed through telescope. If colour bands are observed than rotate the compensator on the prism to remove colour bands. Rotate the prism screw so that the limit of the bright and dark parts touches at the centre point of cross wire inside the telescope. At this time read the refractive index on the scale, note the reading upto four digits. Similarly take three readings of each liquid, determine average and calculate the specific refractive index and molecular refractive index.

Observation Table:

No.	Liquid	Specific Density	Absolute Density	Refractive Index				Specific Refractive Index	Molecular Refractive Index
				1	2	3	Average		
1	A								
2	B								
3	C								
4	D								
5	H_2O								

Note: Ask the Examiner for the Molecular Weight of the Liquids

Result:

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Liquid	A	B	C	D	Water
Specific Refractive Index					
Molecular Refractive Index					

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Time: 3.00 Hrs **Marks: [30 +5]**

Exercise No. 18 Refractometry

Aim: To prepare glycerine solution with a concentration of 10%, 5% & 2.5 % from the given glycerine solution and Determine the refractive index of given liquid of water and glycerol and thus calculate the specific refractive index and molecular refractive index.

Procedure:

Clean specific gravity bottle with distilled H_2O, alcohol, ether and dry with dryer.

Weigh 10%, 5% & 2.5 % glycerine solutions and water and determine the specific density and absolute density. Arrange the instrument in sunlight expose the prism and clean with water, alcohol and ether in that order and then with cotton. Lastly clean the prism with the liquid to be used for the experiment. Then put 2-3 drops of that liquid on prism and close prism. Insert the liquid by glass tube or pipette through the hole on the side of prism box. Adjust the mirror in such a manner that by the reflection of light rays maximum surface of prism is brightened. Rotate the prism with the screw so half of the circle gets darken remaining becomes brighter which can be observed through telescope. If colour bands are observed than rotate the compensator on the prism to remove colour bands. Rotate the prism screw so that the limit of the bright and dark parts touches the centre point of cross wire inside the telescope. At this time read the refractive index on the scale, note the reading upto four digits. Similarly take three readings of each liquid, determine average and calculate the specific refractive index and molecular refractive index.

Observation Table:

No.	Liquid	Density	Refractive Index	Specific Refractive Index	Molecular Refractive Index
1	A (Water)				
2	B (Glycerol)				
3	10 %				
4	5 %				
5	2.5 %				
6	Unknown				

Graph

Plot the graph of % composition against specific refractive index.

Determine the concentration of the unknown solution from the graph

Result:

No.	Liquid	Specific Refractive Index	Molecular Refractive Index
1	A (Water)		
2	B (Glycerol)		
3	10 %		
4	5 %		
5	2.5 %		

2. Concentration of the unknown solution is ……

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Time: 3.00 Hrs **Marks: [30 +5]**

Exercise No. 19 Viscosity

Aim: Find relative and absolute viscosity of given pure liquids A, B, C & D by Ostwald's viscometer

Requirement: Viscometer, Liquid A, B, C & D and distilled H_2O, Stopwatch, Specific gravity bottle

Procedure:

1. Clean specific gravity bottle respectively with chromic acid, water, alcohol and ether. Dry it with dryer. Find specific density and absolute density by weighing liquid A, B, C & D in specific gravity bottle.

2. Clean viscometer with chromic acid, water, alcohol and ether. Dry it with dryer.

Take the definite volume of liquid A [e.g. l0 ml or 15ml] in the broad part of viscometer. Suck the liquid by rubber tube in the capillary upto the upper marker. Then allow the liquid to flow up to the lower mark. Note the time in seconds for flowing the liquid down through both the marks. Use stopwatch for this. Similarly for liquid B, C, D and distilled H_2O take three readings for each. The average of all the three readings is to be used for calculation. Calculate the relative and absolute viscosity.

Observation

No	Liquid	Specific Density	Absolute Density	Time in secs			Average 't' secs	Relative Viscosity	Absolute Viscosity
				t_1	t_2	t_3			
1	A								
2	B								
3	C								
4	D								
5	H_2O								

Result:

1) Relative Viscosity of the liquids :

A= …….

B= ……

C=……

D= …..

2) Absolute Viscosity of the liquid:

A= ……

B=……

C=……

D=……..

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Time: 3.00 Hrs **Marks: [30 +5]**

Exercise No. 20 Viscosity

Aim: To prepare glycerine solution with a concentration of 10%, 5% & 2.5 % from the given glycerine solution and determine relative and absolute viscosity of given glycerine solution and unknown concentration by Ostwald's viscometer

Requirement: Viscometer, Glycerine and distilled H_2O, Stopwatch, Specific gravity bottle

Procedure:

1. Clean specific gravity bottle respectively with chromic acid, water, alcohol and ether. Dry it with dryer. Find specific density and absolute density by weighing liquid A, B, C & D in specific gravity bottle.
2. Clean viscometer with chromic acid, water, alcohol and ether. Dry it with dryer.

Take the definite volume [10 or 20 ml] of 10 % glycerine solution in the broad part of viscometer. Suck the solution a little above the upper mark by means of a rubber tube joined to the capillary. Then release the liquid to flow down to the lower mark. Use stopwatch to note the time in seconds for the liquid to flow down through both the marks. Similarly find the flowing time for 10%, 5% & 2.5 % for liquid B, C, D and distilled H_2O take three readings for each. The average of all the three readings is to be used for calculation. Calculate the relative and absolute viscosity.

Observation

No	Liquid Conc	Specific Density	Absolute Density	Time in secs			Average 't' secs	Relative Viscosity	Absolute Viscosity
				t_1	t_2	t_3			
1	10 %								
2	5 %								
3	2.5 %								
4	unknown								

Graph

Plot Graph of Concentration against Absolute Viscosity and determine the concentration of the unknown from the graph

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Result:

1. Concentration of the unknown solution = ……

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Time: 3.00 Hrs **Marks: [30 +5]**

Exercise No. 21 Colorimetry

Aim: Determine the amount of Ni^{+2} in the given solution using DMG by colorimetric method.

Requirements: Ni^{+2} solution, Saturated Br_2 solution, 1% alcoholic solution of DMG (Dimethylglyoxine), 1: 1 NH_3 solution

Procedure: Dilute with distilled water the given Ni^{+2} solution in 100ml measuring flask.

Take 2, 4, 6, 8 and 10ml of the above Ni solution in five different measuring flask each of 100 ml capacity. Add 2ml of saturated Br_2 solution, 2ml of 1: 1 NH_3 solution and 5ml DMG solution into each of the 100ml measuring flask and make up the volume to 100ml with distilled H_2O. Similarly add 2ml of saturated Br_2 solution, 2ml of 1: 1 NH_3 solution and 5ml DMG solution into the unknown solution given in a 100 ml measuring flask and make up the volume to 100ml.Shake each flask well and keep them for 10 - 15 minutes.

Standardize the calorimeter for zero and 100 positions using with distilled H_2O as standard.

[Use green filter or 480 μ wavelength] Measure the transmittance T% and optical density (O.D) of all the above prepared solution and also for the unknown solution.

Observation No.	Volume taken ml	Concentration C mg/ml	Optical Density OD	Transmittance T %
1	2.0 ml			
2	4.0 ml			
3.	6.0 ml			
4	8.0 ml			
5	10.0 ml			
6	Unknown			

Graphs

Draw the graph of

1. Concentration against T%

2. Concentration against OD

Find the concentration of unknown solution from the graphs.

Note: 1 ml $NiSO_4.7H_2O$ contains 0.01mg of Ni

Result

Concentration of unknown solution:

1. From graph I concentration = _____ mg/ml

2. From graph II concentration = _____ mg/ml

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Time: 3.00 Hrs **Marks: [30 +5]**

Exercise No. 22 Colorimetry

Aim: To determine the amount of Fe^{+3} in the given solution by colorimetric method.

Requirement: Ferric Ammonium Sulphate solution (0.3mg Fe /ml) in a 100 ml flask, 20% KCNS solution, 2N HCl, Dropping pipette/burette, 100ml measuring flask

Method: Dilute with distilled water the given Fe^{+3} solution to 100ml in measuring flask. From this diluted solution take 2, 4, 6, 8 and 10ml in five different measuring flask each of 100ml capacity. Add 1ml 2N HCl solution and 5ml 20% KCNS solution into each measuring flask and dilute it up to 100ml with distilled H_20. Similarly add 1ml 2N HCl and 5ml 20% KCNS solution into unknown solution given in a 100 ml measuring flask and make up the volume to 100ml. Shake each flask well and keep them for 10 -15 minutes. Keep shaking each flask after few minutes.

Measure the Optical Density and Transmittance of the known and unknown solution. [Use green filter or 480 μ wavelength]

Observation No.	Volume taken ml	Concentration C mg	Optical Density OD	Transmittance T %
1	2.0 ml			
2	4.0 ml			
3.	6.0 ml			
4	8.0 ml			
5	10.0 ml			
6	Unknown			

Graphs

Draw the following graphs

1. Graph of Transmittance against concentration
2. Graph of Optical density against concentration

Find the concentration of unknown solution from the graph and note the results

Result

Concentration of unknown solution:

1. From graph I concentration =_____ mg/ml

2. From graph II concentration = _____ mg/ml

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Time: 3.00 Hrs **Marks: [30 +5]**

Exercise No. 23 Polarimeter

Aim : To determine specific rotation of the given dextrose solution of three different concentration (10%, 5%, 2.5%). Plot the graph of specific rotation against concentration and hence determine the unknown concentration.

Candidates should prepare 10%, 5% and 2.5% concentration of the solution.

No.	Concentration	Reading of Distilled water	Reading of solution	Angle of Deviation θ	Specific Rotation α
1	10 %				
2	5 %				
3	2.5 %				
4	Unknown				

Note

1. Systematic and neat entry of the results will be considered while assigning marks
2. Details of the calculations should be clearly shown

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Time: 3.00 Hrs **Marks: [30 +5]**

Exercise No. 24 Polarimeter

Aim: To study the inversion rate of sugar in presence of 1N HCl and hence determine the rate constant for the inversion of cane sugar.

Requirements

1. Cane sugar
2. 1N HCl

Procedure

Prepare 20% solution of pure cane sugar by dissolving 20 gms cane sugar in 100 ml distilled water. Pipette out 25 ml of the cane sugar solution in a dry conical flask. Take 25ml 1N HCl solution in a clean dry beaker. Keep both containers i.e. the conical flask (containing sugar solution) and the beaker (containing 1N HCl solution) in a water bath so that the two solutions attain the same temperature.

Mix the cane sugar solution and 1N HCl solution, shake the mixture and immediately fill it in the observation tube; and measure the angle of rotation. Consider this reading as the zero reading.

Take readings for angle or rotation at an interval of 10 minutes.

Take six readings other than the zero reading. Tabulate your readings as shown below

Observation Table

No.	Time in Mins	Rotation angle 'r'	Inversion $r_o - r_t$	$r_t - r_\infty$	$\log (r_t - r_\infty)$	k
1	0					
2	10					
3	20					
4	30					
5	40					
6	50					
7	60					

Ask the Examiner for infinity reading i.e. r_∞

- ✓ Give the equation to calculate the rate of the reaction
- ✓ Plot graph of $(r_o - r_t)$ against t
- ✓ Plot graph of $\log(r_t - r_\infty)$ against t
- ✓ Deduce the order of the inversion of cane sugar from the graphs

Result

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1. The rate constant for the inversion of cane sugar, k =_____
2. From the graphs the order of the reaction is _____

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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	Session	'A' GROUP	'B' GROUP
FIRST DAY	10.00 AM, Session-1	Organic Separation	Physico Chemical
	Session-2	Physico Chemical	Organic Separation
SECOND DAY	Session-3	Volumetric Analysis & Viva	

Note:

The Candidates are informed to be present at the 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:

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

Senior Examiner
TYBSc Chemistry Practical Examination
March / April /Oct / Nov 20