GENERAL GUIDELINES FOR THE CONDUCT OF PRACTICALS AND PRACTICAL EXAMINATIONS

AN2P01/AP2P01/CH2P01/PH2P01/PO2P01
INORGANIC CHEMISTRY PRACTICAL - 1

TOTAL WEIGHT FOR THE COURSE : 20 **CREDIT : 3**

COMPONENTS

Part	Q.No	Question Type	Weight
I	1	Principle of separation and identification of cations (groups I-VI)	1
	2	Qualitative separation and identification of a cation mixture containing two less familiar cations such as Tl, W, Se, Mo, Ce, Th, Ti, Zr, V, U and Li	5
II	3	Write the theory and Principle for the estimation metal ions by Colorimetric method.	1
	4	Quantitative analysis of any of the metal ions or nitrate or phosphate ions given in the syllabus	5
III	5	Write the theory and principle of preparation of any one metal complex given in the syllabus and its characterization by spectroscopic methods.	1
	6	Preparation of Metal complex , purification and determination of m.p of ligand and metal complex.(Ligand and metal complex)	5
	7	Viva	**2**
		TOTAL	**20**

MODEL QUESTION PAPER

TIME : 6 Hrs **TOTAL WEIGHT: 20**

1. Write down in 15 minutes the principle for the separation of group I to VI cations (Weight: 1)
2. Separate and identify the rare earth cations in the given binary mixture. (Weight: 5)
3. Write down in 15 minutes the theory and principle for the estimation of nitrate ion by colorimetric method. (Weight: 1)
4. Estimate the mass of manganese in the whole of the given solution by colorimetric method. (Weight: 5)
5. Write down in 15 minutes, theory and principle for the preparation of the metal complex and its characterization by UV-Visible, IR and NMR spectroscopic methods. (Weight: 1)
6. Prepare the metal complex Potassium tris (oxalate) Aluminium (III) , dry and purify it by re crystallization. Determine the melting point of ligand and metal complex. Display the crude and recrystallised samples. (Weight: 5)
7. Viva based on the Inorganic Chemistry practical topics. (Weight:2)

AN2P02/AP2P02/CH2P02/PH2P02/PO2P02
ORGANIC CHEMISTRY PRACTICALS - 1

TOTAL WEIGHT FOR THE COURSE : 20 **CREDIT : 3**

COMPONENTS

Part	Q.No	Question Type	Weight
I	1	Write the theory and experimental method for any one of the separation technique given in the syllabus	1
II	2	Write the theory and experimental method for the separation of organic binary mixtures by chemical or physical methods	2
	3	Quantitative separation of the organic binary mixture by chemical/physical method	6
	4	Qualitative analysis of binary organic mixtures by TLC	2
	5	Purification of an organic compound by column chromatography.	5
III	6	Draw the structures of organic molecules and reaction schemes by ChemDraw/ SymixDraw/Chemsketch etc. (2 exercises)	2
	7	Viva	**2**
		TOTAL	**20**

MODEL QUESTION PAPER

TIME : 6 Hrs **TOTAL WEIGHT: 20**

1. Write the theory and experimental method of soxhlet extraction technique. (Weight: 1)
2. Write the theory and experimental method for the separation of the given organic binary mixture by chemical/physical methods. Report the method of separation in the first 30 minutes. (Weight: 2)
3. Separate the given organic binary mixture quantitatively by chemical/physical method. Record the yield of the components, determine the melting point of solid component and boiling point of the liquid component and display the purified samples for inspection. (Weight: 6)
4. Separate the given organic mixture by TLC, calculate the R_f values of the components and display the developed TLC plate for inspection. (Weight: 2)
5. Prepare a column made up of silica gel and purify the given organic compound by column chromatography. Record the yield and display for inspection. (Weight: 5)
6. (a) Draw the structure of the organic compound (IUPAC name is given), and
   (b) Draw the given organic reaction scheme by ChemDraw/ SymixDraw/Chemsketch. Submission of the printout of the above for inspection. (Weight: 2)
7. Viva based on the organic chemistry practical topics. (Weight: 2)

DESCRIPTION

PART I : Total Weight: 1

General Methods of Separation And Purification Organic Compounds Such as:

1. *Solvent extraction*
2. *Soxhlet extraction*
3. *Fractional crystallization*
4. *TLC and Paper chromatography*
5. *Column chromatography*
6. *Membrane dialysis*

Student must familiarize with the principle, materials and experimental procedure for the above separation techniques. The record of these experiments should be submitted and the student shall be asked to write the theory and experimental method of any one of these techniques at the time of examination.

PART II: Total Weight: 15

1. **Separation of organic binary mixtures by chemical/solvent separation methods.**
2. **Separation of organic mixtures by TLC.**
3. **Separation/purification of organic mixtures by column chromatography.**

1. Separation of Organic Binary Mixtures by Chemical/Solvent Separation Methods.

1.1. At least 10 experiments should be done by the students. Both chemical (eg. by using sodium bicarbonate, sodium hydroxide, hydrochloric acid, sodium bisulphite etc.) and physical (eg. Solvent separation of organic binary mixtures with differing polarity by organic solvents such as diethyl ether) methods shall be adopted. (Mixtures can be solid-solid, solid liquid or liquid- liquid- **No need to use bifunctional organic compounds for separation).**

Students may be asked to write detailed theory and experimental procedure of the method of separation of the given experiment at the time of examination. Method of separation should be reported in writing within the first 30 minutes.

Weight	**= 2**
Method and Theory	= 1
Procedure	= 1

1.2. Quantitative separation of the organic binary mixture (preferably one solid and one liquid) by chemical or physical methods.

Weight	**= 6**
Component 1: Yield	= 1
Quality	= 1
Boiling point or melting point	= 1
Component 2: Yield	= 1
Quality	= 1
Boiling point or melting point	= 1

2. Separation of Organic Mixtures by TLC

At least 5 experiments should be done in this category. At least 2 experiments (preferably coloured) should be given in a batch of 5-8 students at the time of examination. The students should determine the Rf values of the components. Developed TLC plates should be exhibited at the time of examination.

Weight = 2

Display of the developed plate = 1
Rf values of 2 components = 1

List of Mixtures- Some Examples

1. o-Chloroaniline and p-Chloroaniline (use iodine chamber or UV detector).
2. o-Nitroaniline and p-Nitroaniline.
3. Benzoin and Benzil.
4. o-Nitroaniline and o-Chloroaniline (use iodine chamber or UV detector).
5. o-Nitroaniline and Azo dye (formed from the reaction of benzene diazonium chloride with beta-naphthol)

3. Purification of Organic Compound by Column Chromatography

Students should conduct at least 4 experiments in this category. Organic compound (preferably colored) and the solvent system may be given to the student. Activated silica can be used for separation. The organic compound should be quantitatively purified and the yield should be reported soon after the separation. Display the purified sample for inspection. Purity may be ascertained by the examiner.

Weight = 5

Preparation of column = 1 (Approx. 20 cm height, diameter of column 2 cm)
Quality of the column = 1
Purity of the compound = 1
Yield of compound = 2

List of Organic Compounds - Some Examples

1. o-Nitroaniline
2. o-Chloroaniline
3. Benzil
4. Azodye (formed from the reaction of benzene diazonium chloride with beta-naphthol)

PART III: Drawing the Structures

Weight = 2

Drawing the structures of the organic molecule (IUPAC name should be given) and reaction schemes of any one of the following reactions by ChemDraw, SymixDraw and Chemsketch and submission of the printout of the same.

1. Cycloaddition of diene and dienophile (Diels-Alder reaction)
2. Oxidation of primary alcohol to aldehyde and then to acid.
3. Benzoin condensation.
4. Esterification of simple carboxylic acids.
5. Aldol condensation.

Part IV: Viva-Voce

Weight = 2

A comprehensive viva-voce based on the organic chemistry practical topics may be conducted at the time of examination.

ANNEXURE

EXPERIMENTS FOR TLC AND COLUMN CHROMATOGRAPHY

1. Separation of mixture of benzil and 2-nitrophenol

Stationary phase: Silica gel
Eluent: 94/6: Hexane/ethyl acetate
R_F for Benzil is 0.2 and for 2-nitrophenol is 0.4

2. Separation of mixture of benzil and naphthalene

Stationary phase: Silica gel
Eluent: 94/6: Hexane/ethyl acetate
R_F for Benzil is 0.2 and for naphthalene is 0.6

3. Separation of mixture of anthracene and anthraldehyde

Stationary phase: Silica gel
Eluent: 94/6: Hexane/ethyl acetate
R_F for anthraldehyde is 0.3and for anthracene is 0.5

4. Separation of mixture of benzophenone and naphthalene

Stationary phase: Silica gel
Eluent: 94/6: Hexane/ethyl acetate
R_F for Benzophenone is 0.4 and for naphthalene is 0.6

5. Separation of mixture of 2-nitrophenol and 4-nitrophenol

Stationary phase: Silica gel
Eluent: 94/6: Hexane/ethyl acetate
R_F for 4-nitrophenol is is 0.2 and for 2-nitrophenol is 0.4

This experiment serves the additional purpose of demonstrating effect of intra *vs* intermolecular *H*-bonding on properties of molecules.

AN2P03/AP2P03/CH2P03/PH2P03/PO2P03
PHYSICAL CHEMISTRY PRACTICAL - 1

TOTAL WEIGHT FOR THE COURSE : 20 **CREDIT : 3**

COMPONENTS

Part	Q.No	Question Type	Weight
I	1	Physical Chemistry Experiment	
		Brief Procedure	2
		Data	11
		Graph	
		Calculation	
		Unknown calculation	
		Accuracy	
		Total	13
II	2	Computational chemistry experiment	
		Input file or brief procedure	2
		Setting up input file and running the calculation	1
		Tabulation of data	1
		Results and analysis	1
		Total	**5**
III	3	**Viva based on part A and B**	**2**
		TOTAL	**20**

MODEL QUESTIONS

TIME : 6 Hrs **TOTAL WEIGHT: 20**

Attempt the marked questions from Part A and Part B

PART A : Questions based on Physical Chemistry experiments **Weight: 13**

01. Verify the Langmuir adsorption isotherm and hence determine the concentration of the given oxalic acid solution.
02. Verify the Freundlich adsorption isotherm and hence determine the concentration of the given oxalic acid solution.
03. Construct the solid-liquid equilibrium diagram for the system formed by two substances A and B. Find out the eutectic temperature and eutectic composition of the system.
04. Melting points of two compounds A and B are ---°C and ---°C respectively. The two compounds form a two component system with eutectic temperature, eutectic composition---°C and ---% by mass of A respectively. Construct the phase diagram and determine the composition of the given mixture.
05. Determine by distribution method the equilibrium constant for the formation of I_3^-.

06. Construct the isothermal ternary phase diagram of the ternary liquid system A-B-C and determine the composition of the given mixture.
07. Determine the concentration of the given KI solution by distribution method. Collect the value of the equilibrium constant from the record book.
08. Study the variation of miscibility temperature of phenol-water system by the addition of KCl and determine the composition of the given KCl solution.
09. Study the variation of surface tension of acetic acid with concentration and determine the composition of the given acid.
10. Collecting data from your record book draw the phase diagram of the ternary system A-B-C and draw the tie line using the given mixture.

PART B: Questions based on computational Chemistry **Weight: 5**

1. Determine the optimized geometrical parameters of ……. using … method and ……basis set

   Examples of molecules: NH_3, H_2O, CH_4, C_2H_6, HCN, O_2 , ethane, ethyne etc.

   method: RHF, ROHF,UHF, B3LYP

   Basis set: STO-3G, 6-31G, 6-31G(d), 6-31G(d,p), 6-31G+(d,p) aug-cc-pVZ, semiempirical basis sets like PM3 , AM1 etc.
2. Optimize the given structure using …… basis set . Identify the molecule. Find the energy. Find the HOMO and its energy. What is its IE or EA? (The Cartesian coordinates of the molecule should be given from literature or from a structure drawing program.)
3. Calculate the rotational energy barrier for ethane / CH_2ClCH_2Cl / hydrogen peroxide using RHF method with … basis set.
4. Calculate the correlation energy of … molecule using MP2 method with …. basis set.
5. Calculate the vibrational frequencies of ……. molecule … using … basis set. Identify the vibrational mode associated with each frequency.
6. Optimize the …. molecule with …. basis set and identify the geometrical parameters , dipole moment, Mulliken atomic charges and number of basis functions on each atom.
7. Calculate the strain energy of cyclopropane/cyclobutane/cyclopentane using an isodesmic reaction at …….. level. (the z-matrix or Cartesian coordinates of the molecule may be given).
8. Do a single point calculation of the given molecule and identify whether it is a stationary point or transition state. (the z-matrix or Cartesian coordinates of the molecule should be given).
9. Calculate the heat of formation of …. from the following reaction using ab initio method. The experimental heat of formation are given below.

   Eg. CO_2; $CO_2 + CH_4$ ---> 2HCHO; heat of formation of methane and formaldehyde are -74.5 and -115.90 kJ/mol respectively.

Part C : A viva-voce based on the practical topics **Weight: 2**