UNIVERSITY OF KERALA

IV Semester M.Sc. Degree Practical Examination, November 2018
Branch III Chemistry – CH 235: Organic Chemistry - II

Time: 9.30 to 3.30
Maximum marks: 75

I. In the first 10 minutes,

a. Write the brief theory and procedure for the identification of a given amino acid by paper chromatography you are provided with pure samples of 5 amino acids---------- (5 marks)

b. Write the brief theory and procedure for the preparation of **p-bromoacetanilide, / dihydropyrimidinone/ 1, 1-bis-2-naphthol** by green chemistry protocol.

(5 marks)

II. Estimate the mass of aniline/phenol/glucose/ascorbic acid in the whole of the given solution using a standard solution of sodium thiosulphate/Fehlings solution of normality ……. (20 marks)

III. Estimate colorimetrically the amount of paracetamol/protein/ascorbic acid in the whole of the given solution (20 marks + graph 5 marks)

IV. Identify the organic compound number …….. of molecular formula …… from the IR, ^{1}H NMR, ^{13}C NMR and mass spectra attached. Write your spectral interpretation in detail. (20 marks)

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SCHEME OF VALUATION

1.	(a) Paper Chromatography	Theory and working: 3 marks Figure and Rf explanation: 2 marks Procedure: 3 marks- equation-1 mark	
	(b) Organic preparation	Green aspects-1 mark	
II.	Estimation of	Anilline/phenol/ascorbic acid	Glucose
	Upto	1 % 20 marks	2 % 20 marks
	Deduct 0.5 marks for every	0.1 % error upto 2%	0.1 % error upto 3%
	Deduct 1 marks for every	0.1 % error upto 2.5 %	0.1 % error upto 3.5 %
		≥ 2.5 % (5 marks)	≥ 3.5 % (5 marks)
III	Colorimetry (Paracetamol)	Upto 3.5 % error	20 marks
		Deduct 1 marks for every 0.1 % ≥ 5 % (5 marks)	
		Graph with five points 5 marks	

IV	Identification of compound from spectra	20 marks
	Identification of four prominent bands in IR spectrum	4 marks
	All peaks in ^{1}H NMR spectrum (correct multiplicity, δ, intensity ratio)	4 marks
	All peaks in ^{13}C NMR	3 marks
	Mass spectrum	
	M+ peak	1 mark
	Base peak	1 mark

Identification of other two peaks	2 marks
Correct final structure	1 mark
Oral discussion	4 marks

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ESTIMATION OF ANILINE / PHENOL

Make up the given solution 100 ml. Pipette out 20 ml into an iodine flask. Add exactly 20 ml N/5 or 40 ml N/20 bromate-bromide solution. Dilute with 25 ml water. Add 5 ml conc. HCl and close the flask immediately. Shake well for a minute. Allow to stand for 30 minutes with occasional swirling. Cool the flask under the tap. Add 10 ml 10 % KI solution carefully, shake and allow to stand for a few minutes. Remove the stopper and titrate with sodium thiosulphate from the burette using starch as indicator. Conduct a duplicate and blank.

ESTIMATION OF GLUCOSE

Make up the given solution to 250 ml. Pipette out 20 ml Fehling's solution into a clean conical flask. Dilute with 20 ml distilled water. Heat to boil and titrate with glucose solution from the burette until the blue colour just discharges. Repeat the titration using methylene blue indicator near the endpoint. Repeat.

ESTIMATION OF ASCORBIC ACID

a. Standardisation of iodine solution

Pipette out 20 ml of standard sodium thiosulphate solution into a conical flask. Add 2 ml of starch solution and titrate with iodine solution from the burette. The end point is the appearance of blue colour. Repeat the titration for concordant values.

b. Estimation of ascorbic acid

Make up the given solution to 100 ml. Pipette out 20 ml of the solution to a conical flask. Add 2 ml of starch solution dilute to about 150 ml and titrate with iodine solution from the burette. The end point is the appearance of blue colour. Repeat the titration for concordant values.

PREPARATION OF SOLUTIONS

a. Estimation of Aniline/ Phenol

i. Aniline: Weigh 1.9 g of aniline, dissolve in sufficient HCl and dilute to 250 ml. Give 20, 21, 22, 22ml.
ii. Phenol: Weigh 1.9 g of phenol, dissolve in sufficient NaOH solution and dilute to 250 ml. Give 20, 21, 22, 22ml.
iii. N/5 Bromate-bromide solution: Dissolve 5.567 g of AR potassium bromate and 35 g potassium bromide in distilled water and make up to 1 L.
iv. N/10 Sodium thiosulphate: Prepare by dissolving 6.2 g of thiosulphate in water and make up the solution to 1 L. Standardise by titrating with standard potassium dichromate.
v. 10 % potassium iodide: Dissolve 100g of KI in distilled water and make up to 1 L.

b. Estimation of glucose

i. Glucose solution: Dissolve 6.3 g of glucose in distilled water and make up to 250 ml. Give 20, 21, 22, 22ml.
ii. Fehling's solution A: Dissolve 34.64 g of powdered A R copper sulphate in water and make upto 500 ml.
Fehling's solution B: Dissolve 173 g crystalline sodium potassium tartarate (Rochelle salt) in warm water. Dissolve 60 g pure NaOH in water. Mix the two solutions, cool and make upto 500 ml. Transfer equal volumes of A and B to a dry flask and mix thoroughly by shaking.

c. Estimation of ascorbic acid

i. Sodium thiosulphate (M/20) solution: Prepare by dissolving 6.2 g of thiosulphate in water and make up the solution to 500 ml. Standardise by titrating with standard potassium dichromate.
ii. Iodine (M/40) solution: Weigh 10 g of KI and 6.5 g of iodine into a100 ml beaker. Dissolve in distilled water, transfer quantitatively to 1000 ml standard flask and make up to the mark by distilled water.
iii. Ascorbic acid (M/8) solution: Weigh about 5.5 g of ascorbic acid accurately into a 250 ml standard flask. Add about 12.5 g of oxalic acid to prevent oxidation by air. Pipette out 20, 21, 22, 23 ml for estimation.

COLORIMETRIC ESTIMATION OF PARACETAMOL

Procedure

a. Weigh 0.1 g of paracetamol and dissolve in deionized water. Transfer qualitatively to a 500 ml standard flask and make up to the mark.
b. Dilute 25 ml of this stock solution to 250 ml.
c. Add 1, 2, 4, 6, 8 and 10 ml of this solution to 100 ml standard flasks labeled S1, S2, S3, S4, S5 and S6. Add 9, 8, 6, 4, 2 and 0 ml deionized water respectively to these flasks to make them to 10 ml.
d. Make up the given unknown paracetamol solution to 250 ml. Add 5 and 7 ml of this solution to flasks labeled T1 and T2, add 5 and 3 ml of deionized water to make the solution to 10 ml.
e. To each flask, add 4 ml of iron(III)chloride solution and 8 ml of potassium hexacyanoferrate(III) solution. Leave for 10 minutes and then add 2 ml 5M HCl. Make up to the mark by deionized water.
f. After 20 minutes, measure the absorbance at 700 nm.
g. Plot a graph of absorbance against concentration (calibration curve) of paracetamol.
h. Find out the concentration of unknown paracetamol from the calibration curve.

PREPARATION OF SOLUTIONS

a. Paracetamol solution: Dissolve 0.1 g of pacacetamol in deionized water and make up to 500 ml.
b. Iron(III) chloride (0.02 M): Dissolve 1.62 g iron(III) chloride in water and make up to 500 ml.
c. Potassium hexacyanoferrate(III) solution (0.002 M): Dissolve 0.66 g potassium hexacyanoferrate(III) in water and make up to 1000 ml.
d. 5 M HCl: Dilute 225 ml Conc. HCl to 500 ml.