RAJARATA UNIVERSITY OF SRI LANKA
FACULTY OF APPLIED SCIENCES

B.Sc. in Applied Sciences
B.Sc. Third Year Semester II Examination – July 2020

CHE 3311 – ADVANCED ANALYTICAL CHEMISTRY

Time: Three (03) hours.

Answer any six questions.
The use of a non-programmable calculator is permitted.

1. a) Explain clearly the difference between a distribution coefficient, K_D and a distribution ratio, D.

(30 marks)

b) A chemist dissolves 155.13 mg of iodine in 1.0 L of distilled water. The solution is equilibrated at 25 °C with 50.0 mL of carbon tetrachloride. Titration of the organic solvent requires 22.35 mL of 0.04474 M $Na_2S_2O_3$ solution. Calculate the distribution coefficient, K_D for I_2.
(I = 127)

(30 marks)

c) How much more effective a sample of 0.5 g in 10.0 mL water is extracted (i) four extractions using 5.0 mL of 1-hexanol (ii) one extraction 20.0 mL portion? Comment your answers. Derive any equation you have used in this calculation. D =2

(40 marks)

2. a) Name two chelating reagents and explain how you would determine the Ni(II) presents in aqueous solution at pH between 5-12, using the solvent extraction involves metal chelates.

(35 marks)

b) Discuss the effect of pH and the reagent concentration on the solvent extraction of metal chelates.

(30 marks)

b) List the limitations in solvent extraction technique and briefly discuss the solid phase extraction (SPE) technique used to overcome the limitations.

(35 marks)

3. a) Write down the van Deemter equation. Explain the constant terms in the equation.

(35 marks)

b) Define the following :

i. R_f value.
ii. Retention time.
iii. Cation exchange resins
iv. Anion exchange resins.

(20 marks)

c) Describe the principles of gas chromatography and explain the following type of detectors used in gas chromatography.

i. Thermal conductivity detector.
ii. Flame ionization detector.
iii. Electron capture detector.

(45 marks)

4. a) Describe the following terms:

i. Capacity factor.
ii. Resolution.
iii. Theoretical plates.

(15 marks)

b) Discuss the steps involved in separation of the analytes using thin layer chromatography. Compare advantages and disadvantages of paper chromatography and thin layer chromatography.

(40 marks)

c) Explain briefly the sample injection method and two commonly used detectors in high performance liquid chromatography.

(45 marks)

5. a) Distinguish between two types of monochromators used in spectrophotometers and list one advantage and one disadvantage of each.

(30 marks)

b) Compare the operations of single-beam spectrophotometer and a double beam spectrophotometer.

(25 marks)

c) Phosphorus in urine can be determined by treating with molybdenum (VI) and then reducing with the phosphomolybdo complex with aminonaptholsulfonic acid to give the characteristic molybdenum blur colour. This absorbs at 690 nm. A patient excreted 1270 mL urine in 24 hours and the pH was 6.5. A 1.00 mL aliquot of the urine was treated with molybdate reagent and aminonaphtholsulphonic acid and was diluted to a volume of 50.0 mL. A series of phosphate standard was similarly treated. The absorbance of solutions at 690 nm, measured against a blank, were as follows:

Solution / ppm P	Absorbance
1.00	0.205
2.00	0.410
3.00	0.615
4.0	0.820
Urine sample	0.625

i. Calculate the number of grams of phosphorus excreted per day.

ii. Calculate the phosphate concentration in the urine as millimoles per liter. (P = 31, O = 16)

(45 marks)

6. a) Draw a block diagram of atomic absorption spectroscopy (AAS) and briefly discuss the applications of AAS.

(40 marks)

b) Explain the mechanism and operation of hollow-cathode lamp.

(20 marks)

c) A constant current of 0.800 A is used to deposit copper at the cathode and oxygen is evolved at the anode of an electrolytic cell. Calculate the number of grams of each product formed in 15.2 min. Cu = 63.55, O = 16 and F = 96485 C.

(40 marks)

7. a) List three applications of polarography.

(30 marks)

b) Ilkovic equation used in polarography is given by $i_d = k\ n\ D^{1/2}m^{2/3}t^{1/6}C$. Define the terms with their units. The diffusion coefficient of O_2 in dilute aqueous solution is 2.6×10^{-5} cm^2/s . A 0.25×10^{-3} M solution of O_2 in an appropriate supporting electrolyte gives a polarographic wave with a diffusion current 5.8×10^{-6} A. The DME constants m and t are 1.85×10^{-3} g/s and t = 4.09 s respectively. Calculate the value of n.

(40 marks)

c) Discuss the limitations of polarography.

(30 marks)

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