THE OPEN UNIVERSITY OF SRI LANKA
B.Sc. Degree Programme / Stand alone courses in chemistry
Level 5 – FINAL EXAMINATION – 2006/2007

CHU 3129/CHE 5129 – INSTRUMENTAL METHODS OF CHEMICAL ANALYSIS

Duration: Two and half-hours
Date and time: 09th of May 2007, from 1.30 p.m. to 4.00 p.m.

Instructions to students

This question paper consists of 05 pages and six questions. Answer any four questions only.

$R = 8.314\ J\ K^{-1}\ mol^{-1}$, $F = 96485\ C\ mol^{-1}$

1.
(i) A student separated a mixture containing three components using gas chromatography and obtained the chromatogram with a report. Briefly explain in words the general method of determining the percentage of each component.

(25 marks)

(ii) With respect to the detector of the gas chromatograph, under which condition your answer to part (i) is correct?

(10 marks)

(iii) In order to obtain correct quantitative results from the chromatogram what parameter you have to calculate? How do you calculate the parameter? Then how do you find out the corrected peak areas?

(15 marks)

(iv) Write down five properties that a good gas chromatographic detector should have in order to obtain quantitative results. Briefly define each of the properties.

(50 marks)

2.

(i) Distinguish between adsorption and partitioning mechanisms using gas-liquid chromatography and gas-solid chromatography as examples.

(20 marks)

(ii) What kind of equilibrium would you try to achieve by saturating a TLC tank with vapor of the mobile phase?

(10 marks)

(iii) Suppose you try to obtain the chromatogram of one organic compound using high performance liquid chromatography. Which wavelength do you select in the UV absorbance detector to obtain high sensitivity in the determination of the organic compound?

(10 marks)

(iv) Write down two requirements that a pump used in high performance liquid chromatography should satisfy.

(10 marks)

(v) What is the basic difference in introduction of the sample in gas chromatography and in high performance liquid chromatography?

(10 marks)

(vi) What is the purpose of using a guard column in high performance liquid chromatography?

(20 marks)

(vii) Briefly explain why longitudinal diffusion of sample molecules in high performance liquid chromatography is much smaller than that in gas chromatography?

(10 marks)

(viii) What is the difference between gradient elution and isocratic elution in high performance liquid chromatography?

(10 marks)

3.

(i) Define "absorbance" and "transmittance" in terms of the intensity of incident light I_o and intensity of transmitted light I_f.

(20 marks)

(ii) What are the units of "absorbance"?

(05 marks)

(iii) Suppose that a solution of $Co(NO_3)_2$ has a molar absorptivity of 51 $dm^3\ mol^{-1}\ cm^{-1}$ at 505 nm. Plot a graph of absorbance versus concentration (in mol dm^{-3}) for $Co(NO_3)_2$ solutions having concentrations 0.002, 0.004, 0.006, 0.008 and 0.010 in mol dm^{-3}, each solution in a 1 cm cell. (Take absorbance axis to the long side of your graph paper) **Round off all absorbance values to two decimal places. Tabulate all the data that you use to plot the graph.**

(35 marks)

(iv)

(a) **Using the equation of the straight line** find out the concentration of a $Co(NO_3)_2$ solution having an absorbance of 0.35 in the same cell.

(10 marks)

(b) **Using the graph** find out a value for the molar absorptivity of $Co(NO_3)_2$ at 505 nm. **Show your calculations clearly along with the derivation of units.**

(15 marks)

(c) What is the absolute error in your result in part (b)?

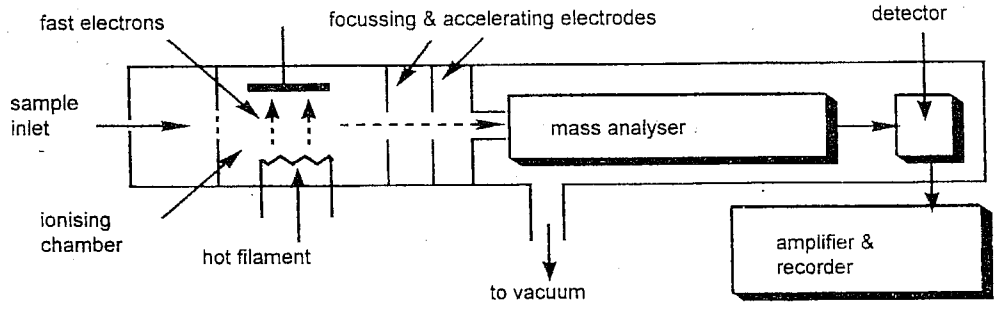
(05 marks)

(v) Why do you need a wavelength selector in a UV-visible spectrophotometer?

(10 marks)

4.

(i) A schematic diagram of a mass spectrometer is given below.



(a) What is the part fixed opposite to the hot filament?

(10 marks)

(b) How are fast moving electrons generated within the ionizing chamber?

(10 marks)

(c) Why can't you analyze neutral molecules using this mass spectrometer?

(20 marks)

(d) How are neutral molecules ionized within the ionizing chamber?

(10 marks)

(e) Why do we need to have a vacuum inside a mass spectrometer?

(10 marks)

(ii) We cannot get an IR spectrum of a sample of nitrogen gas. Clearly explain why?

(25 marks)

(iii) Draw a schematic diagram of a flame photometer.
(15 marks)

5. (i)
Consider a cell which consists of Cu wire and a saturated calomel electrode dipping into a 0.05 M $CuSO_4$ solution. Both calomel electrode and the Cu wire are connected to a potentiometer at 25°C.

$E^{\circ}_{Cu^{2+}/Cu} = +0.337\ V, \qquad E^{\circ}_{Calomel} = +0.244\ V$

(a) Write down the standard cell notation for the above cell.
(10 marks)

(b) Calculate the half cell potential for the Cu electrode.
(20 marks)

(c) Write down the spontaneous cell reaction.
(20 marks)

(d) Calculate the cell potential.
(10 marks)

(ii) (a)

In electrogravimetry, what are the four factors that can affect the properties of the deposit? Explain.
(20 marks)

(b) List three advantages and disadvantages of potentiometric titrations.
(20 marks)

6. **This question has two parts. <u>Answer only one part</u>.**

<u>Part A</u>

(i)

100 mA current is passed through a 2 L solution of silver ions for three minutes. Calculate the concentration of silver formed in this reduction process. Use all SI units for calculations.
(40 marks)

(ii)
Constant potential coulometry requires a three electrode cell. Discuss.

(20 marks)

(iii)
Describe the function of each electrode.

(20 marks)

(iv) Briefly explain three applications of voltametry.

(20 marks)

Part B

(i) Write complete nuclear equations for the following

(*a*) β decay by $^{131}_{53}I$

(*b*) Electron capture by $^{7}_{4}Be$

(*c*) α – decay by $^{210}_{84}Po$

(15 marks)

(ii) Cobalt-60, which undergoes β decay has a half life of 5.26 years.

(a) What is the initial activity of a 50.0 μg sample of ^{60}Co?

(b) The cobalt-60 in a radiotherapy unit must be replaced when its radioactivity falls to 75% of the original sample. How long can this radiation source be used?

(25 marks)

(iii) Briefly describe how radiation can be detected using liquid scintillation detectors.

(20 marks)

(iv)

(a) Write the <u>three</u> conditions that must be fulfilled for an element to be capable of being determined by neutron activation analysis.
(b) Briefly explain the principal behind neutron activation analysis.
(c) What are the advantages and disadvantages of activation analysis?

(40 marks)