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MAIN EXAMINATION

SEPTEMBER –DECEMBER 2021

FACULTY OF SCIENCE

DEPARTMENT OF CHEMISTRY

REGULAR PROGRAMME

CHEM 408: ANALYTICAL CHEMISTRY II

Date: DECEMBER 2021	**Duration: 2 Hours**
INSTRUCTIONS: Answer Question ONE and any TWO Questions	

Q1.

(a) Define the following terms:

(5marks)

(i) Mass Spectrometry
(ii) Chromatographic column
(iii) Karl Fischer Titration
(iv) Particle sizing
(v) Bioanalysis

(b) Name two sources of x-rays, and explain how the x-rays are generated in each case.

(3marks)

(c) What two energy states of spinning–processing nuclei present in a magnetic field are involved in an NMR experiment? Which state represents the higher energy?

(2marks)

(d) What do GC-MS and LC-MS refer to?

(2marks)

(e) The distribution coefficient for a given extraction experiment is 98.0. If the concentration in the extracting solvent is 0.0127 *M*, what is the concentration in the original solvent? **(3marks)**

(f) How does partition chromatography differ from adsorption chromatography?

(2marks)

(g) Why must the size of a liquid sample injected into the GC be small? What would happen if it were too large? **(3marks)**

(h) What type of HPLC should be chosen for each of the following separation applications?

(3marks)

(i)All mixture components have formula weights less than 2000, are molecular and polar, and are soluble in non-polar organic solvents.

(ii) Mixture components have formula weights varying from very large to rather small and are non-ionic.

(iii) Mixture components have formula weights less than 2000, are molecular and polar, and are water soluble.

(i) Distinguish between an electrolytic cell and a galvanic cell. **(2marks)**

(j) A given calibration liquid is known to have a kinematic viscosity of 12.72 cS at 25°C.

(3marks)

(k) Why are both nucleotides and derivatized amino acids difficult to separate via reverse phase chromatography?

(2marks)

Q2. Study the figure(s) below and answer the questions that follow:

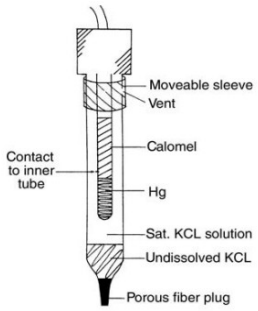


Figure 3: Left, a drawing of a commercial saturated calomel electrode

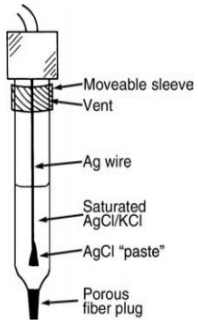


Figure 4: A drawing of a commercial silver–silver chloride reference electrode.

(a) Write the half-cell reaction and Nernst Equations for Figure 3 and Figure 4 above respectively.

(4marks)

(b) Identify the purpose served by each of the chemical or part labelled in figure 3 and 4 above.

(4marks)

(c) What is the E for the Fe^{3+}/Fe^{2+} half-cell if $[Fe^{3+}] = 10^{-4}$ M and $[Fe^{2+}]$ is 10^{-1} M at $25^{O}C$?

(3marks)

(d) Explain the make and working principle of the pH Electrode. **(2marks)**

(e) Write down the Nernst equation involved. How does it compare with that of Reference Electrodes in question 1(a) above?

(5marks)

(f) State the importance of buffer solution in pH measurement process. **(2marks)**

Q3.

(a) Sketch a diagram for an HPLC system that is capable of an isocratic elution. Label each part. Specify a detector that you can use for your system.

(10marks)

(b) What is a guard column and why is it used in HPLC but not in GC systems?

(4marks)

(c) Using and example, describe what is gradient elution and when is it advantageous to
(d)use gradient elution instead of isocratic elution in HPLC?

(4marks)

(d) Explain the difference in the detector systems suited for GC and HPLC.

(2marks)

Q4.

(a) Look at the NMR spectrum in Figure 2. How many different kinds of hydrogen are represented? Explain your answer.

(3marks)

(b) What does the integrator trace in Figure 2 tell you about the number of the different kinds of hydrogen present in the structure?

(2marks)

(c) If you were told that the spectrum in Figure 2 was either ethyl alcohol or diethyl ether, what evidence would you cite to conclude that it is ethyl alcohol and not diethyl ether? **(3marks)**

(d) Explain briefly three applications of NMR spectroscopy in modern scientific world.

(3marks)

(e) If you were told that the spectrum in Figure 2 resulted from either ethyl alcohol or methyl ethyl ether, what evidence would you cite to conclude that it is ethyl alcohol and not methyl ethyl ether? **(3marks)**

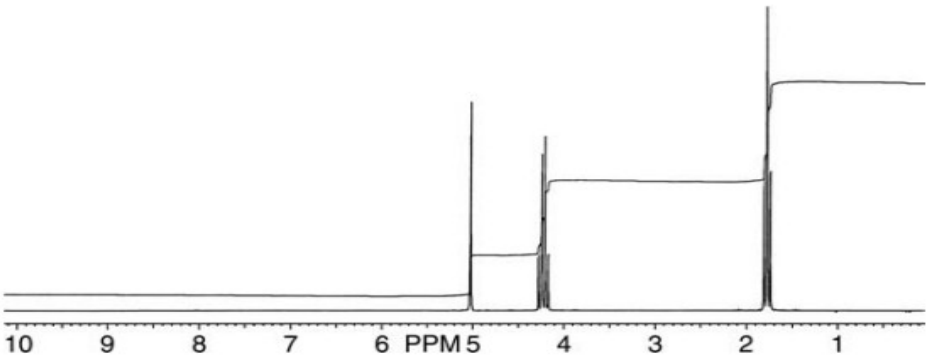


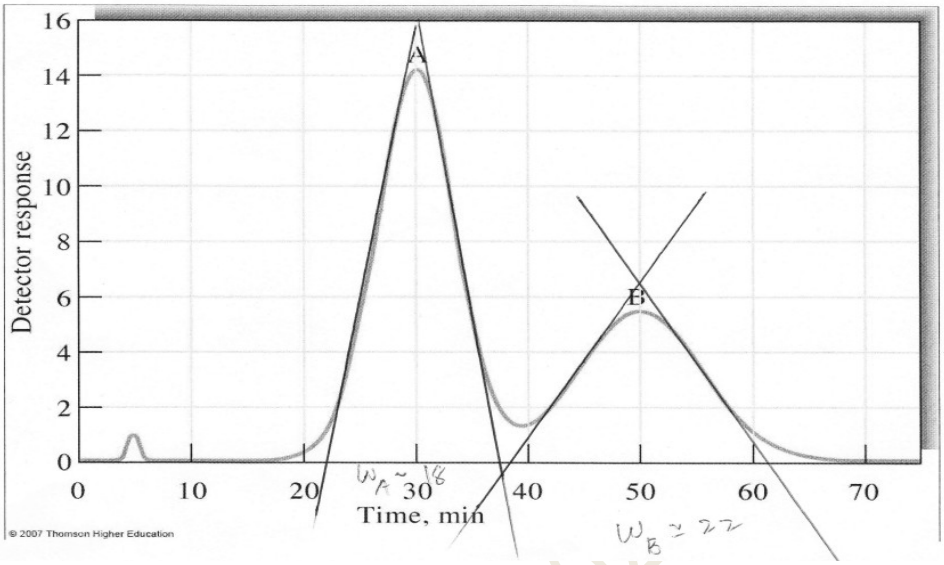
Figure 2: The NMR spectrum for problems (a) to (d).

Q5.

(a) There are three terms in the van Deemter equation: the A term, the B term, and the C term, each describing a different type of contribution to the band broadening. Explain the type of band broadening described by 2of the 3 terms.

(6marks)

(b)A chromatograph of a two component mixture is shown next page. The LC column is 25-cm long. The flow rate was 0.40 ml/min. Using the chromatograph determine/calculate the following:



(i) The time components A and B spends on the stationary phase.

(4marks)

(ii) The retention factor for components A and B.

(3marks)

(iii) The resolution between the two peaks

(4marks)

(iv) What column length would be required to achieve a resolution of 1.75?

(3marks)

END