



KENYATTA UNIVERSITY

UNIVERSITY EXAMINATIONS 2011/2012

SECOND SEMESTER EXAMINATION FOR THE DEGREE OF MASTER OF SCIENCE

SCH 555: ANALYTICAL SPECTROSCOPY II

DATE: THURSDAY, 5TH APRIL 2012

TIME: 9.00 A.M. – 12.00 P.M.

INSTRUCTIONS:

Answer all questions. Show working where necessary.

1 (a) Explain how Fourier transform NMR works. What are its advantages over normal NMR spectroscopy(5 marks).

(b) What is the purpose of double resonance NMR? How does it work (5 marks).

(c) A natural product chemist extracted some organic compounds from some plant roots using hexane. He would like to identify some of the components of the extract. What analytical instruments would you recommend him to use and why. How does the instrument work? (5Marks).

(d) A high resolution NMR instrument operated at a proton frequency of 400 MHz. What is the magnetic field strength of the instrument? If ^{31}P has gyromagnetic ratio of 2.261, at what frequency would you expect it to absorb under the same conditions. (5 marks)

Note: gyro magnetic ratio for proton is 5.585, Bohr magneton is 5.05×10^{-27} G/TS, Planck's constant 6.623×10^{-34} JS.

2 (a) Explain the two problems that made it difficult to obtain NMR spectra of solids until recently. How were the problems minimized? (5 marks).

(b) Predict the appearance of high resolution NMR spectrum for the following compounds

(i) Ethyl-benzene, (ii) acetic acid, (iii) 1,2-dimethoxyethane ($\text{CH}_3\text{OCH}_2\text{CH}_2\text{OCH}_3$), in each case indicate number of different protons and the relative peak area for each proton (4 marks).

(c) Compare and contrast matrix assisted laser desorption ionization and fast atom bombardment (5 marks).

2 (d) Explain two ways of introducing samples in the ionization chamber of a mass spectrometer and the conditions necessary for each type of introductions(4 marks).

3 (a) What resolution is required to separate two masses of 58.9959 and 59.0012 in a mass spectrometer (3 marks).

(b) Explain chemical shift in NMR and its role in structural determination of organic compounds.(5 marks).

(c) Define the following terms:

- (i) Spin-spin coupling.
- (ii) Magnetic anisotropy (4 marks).

(d) Explain two ways in which a nuclear in the excited state can relax without emitting any radiation (4 marks).

4 (a) Calculate in Bohr magneton spin only magnetic moments for a compound with three unpaired electrons (4 Marks).Note: magneto-gyric ratio g for electron is 2.000.

(b) Differentiate between the following terms:

- (i) Ferromagnetism and anti-ferromagnetism
- (ii) Para magnetism and diamagnetism (4 marks)

(C) Draw a diagram showing the effect of temperature on paramagnetic ferromagnetic and anti-ferromagnetic properties of substances.(4 marks)

(d) For a long time liquid chromatography –mass spectrometry was not possible until recently why? How was the problem sorted out?(4 marks),
