



TECHNICAL UNIVERSITY OF MOMBASA

FACULTY OF APPLIED AND HEALTH SCIENCES

DEPARTMENT OF PURE & APPLIED SCIENCES

UNIVERSITY EXAMINATION FOR:

MASTERS OF SCIENCE IN CHEMISTRY

ACH 5108: ADVANCED SPECTROSCOPIC TECHNIQUES

END OF SEMESTER EXAMINATION

SERIES: DECEMBER 2016

TIME: 3 HOURS

DATE: Pick Date Dec 2016

Instructions to Candidates

You should have the following for this examination

-Answer Booklet, examination pass and student ID

This paper consists of **SIX** Question(s). Attempt any **FOUR** questions.

Do not write on the question paper.

Question ONE

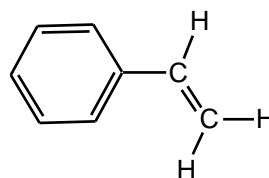
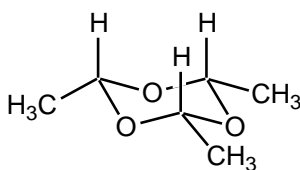
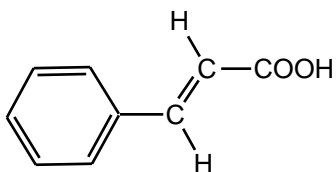
(a) (i) What is spin-spin coupling as applied in NMR spectroscopy? [1 marks]

(ii) Make use of the relevant structures of organic molecules below and sketches of proton spectra to describe the following coupling systems in proton NMR spectroscopy.

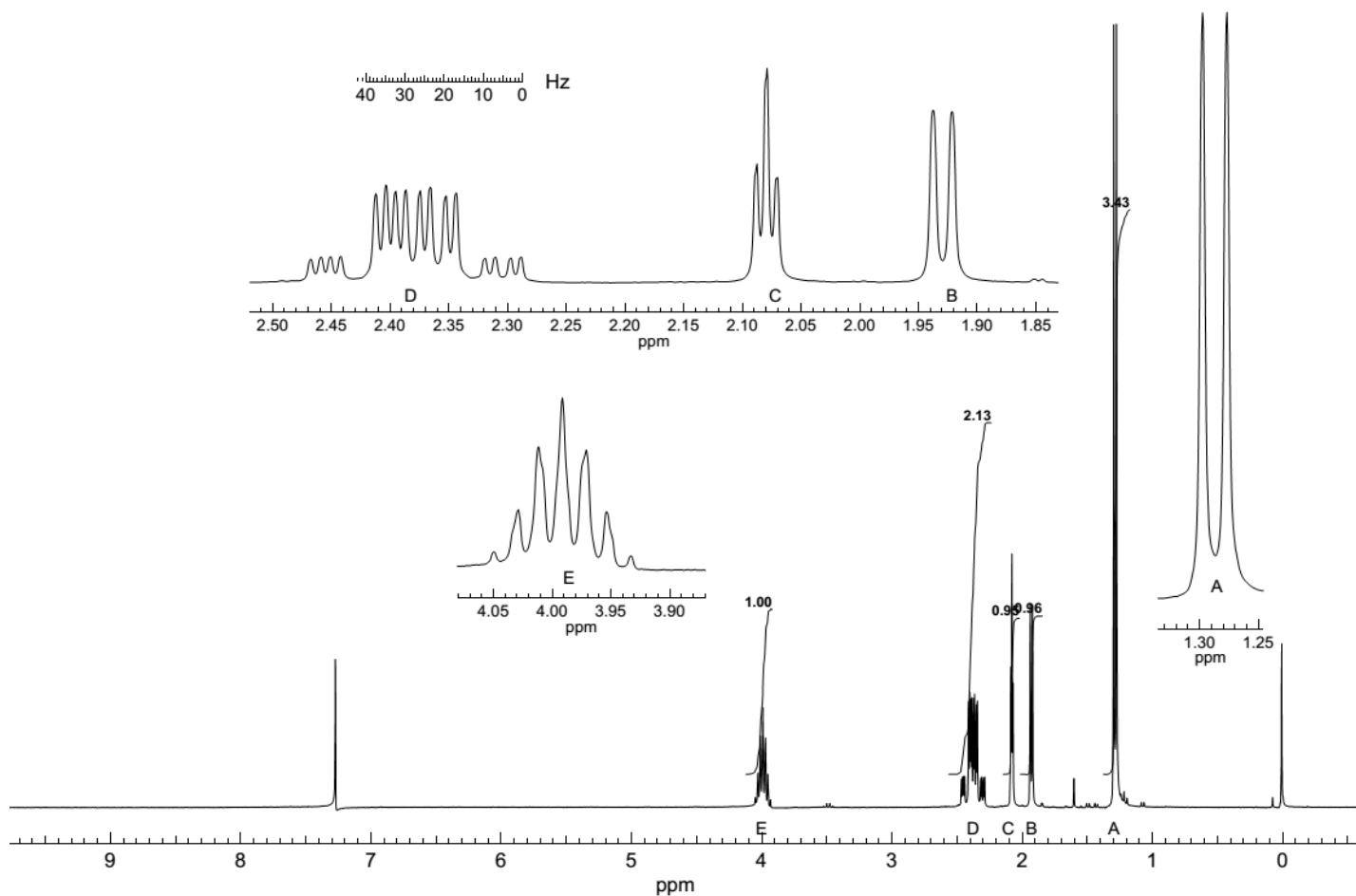
I. AX coupling system [2 marks]

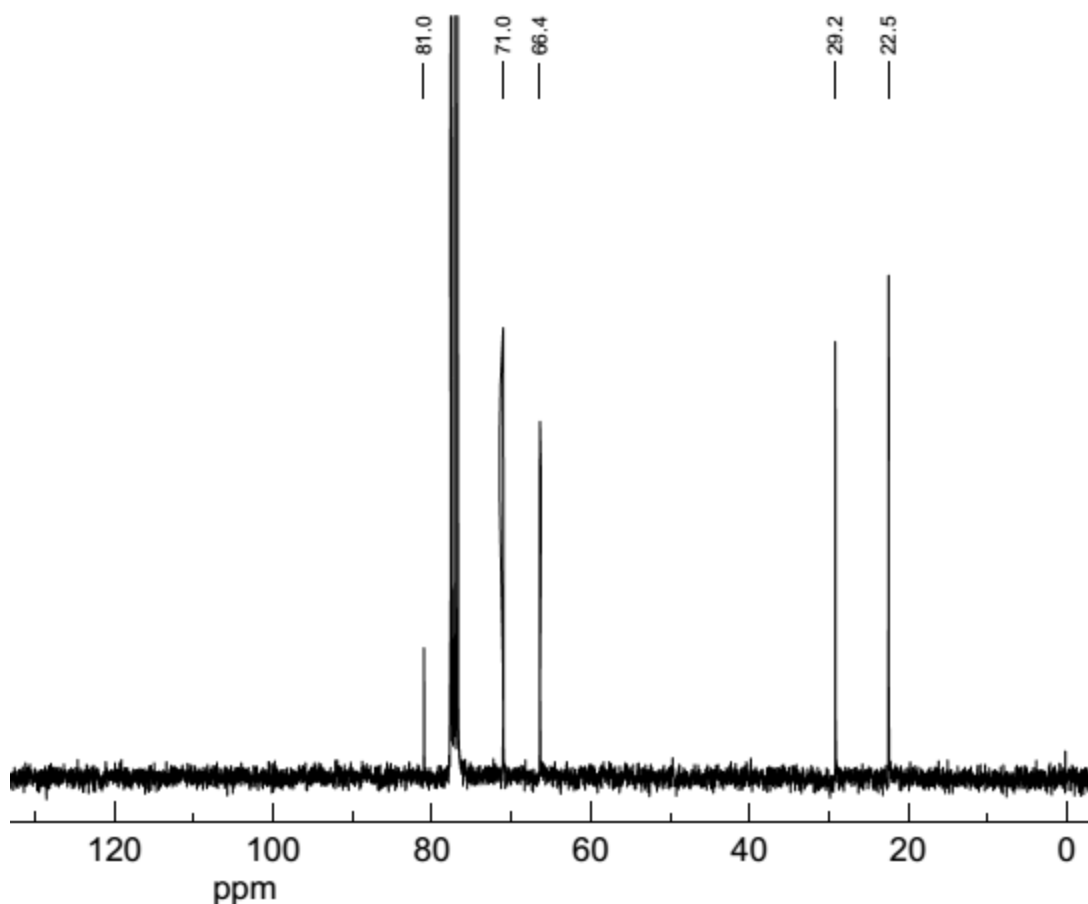
II. AX₃ coupling system [2 marks]

III. AMX Coupling system [2 marks]



(b) An organic molecule has the molecular formula C_5H_8O and register IR absorption signals due to a O-H stretch, carbon-carbon triple bond stretch and a C-H stretch of a sp hybridized carbon. The 1H -NMR and ^{13}C -NMR of the molecule is given below:





- i. Compute the DBE of the molecule. [1 mark]
- ii. Analyze the ^1H -NMR spectrum. For each of the groups of signals marked A, B, C, D and E on the spectrum report the multiplet structure in the standard format (e.g., 0.0 δ , dtd, $J = 0.0, 0.0, 0.0$ Hz, 2H), and any part of structure of the molecule you could obtain from the signal(s). [10 marks]
- iii. Draw the structure of the molecule and write the chemical shift values of the carbon atom on your proposed structure. [7 marks]

Question TWO

- (a) What are NMR shift reagents? Outline their utility in study of complex spectra in NMR spectroscopy and provide any two examples. [8 marks]
- (b) State and explain four factors that affect the chemical shift values registered by protons in ^1H -NMR spectroscopy. [8 marks]
- (c) Discuss spin-spin splitting and coupling in ^{13}C -NMR. [4 marks]
- (d) Outline basic difference between ^1H -NMR and ^{13}C -NMR. [5 marks]

Question THREE

- (a) Which kind of information is obtained from 2D NMR to aid in structural elucidation of organic molecules? [2 marks]
- (b) State six 2D NMR techniques. [3 marks]
- (c) Describe DEPT technique in detail clearly indicating piece of information that can be obtained from the experiment to aid in structure elucidation. [7 marks]
- (d) Describe gated decoupled methodology and inverse gated decoupled methodology as applied in ^{13}C – NMR. [8 marks]
- (e) Outline Nuclear overhauser effect (NOE) as applied in NMR spectroscopy. [5 marks]

Question FOUR

- (a) Outline the principle of Atomic fluorescence spectrometry (AFS) and account for its application to a great variety of environmental, biological and food samples. [8 marks]
- (b) State the three main types of atomic fluorescence and outline when each stated type occurs in Atomic fluorescence spectrometry (AFS) [6 marks]
- (c) State four factors which determine the intensity of the fluorescence radiation in Atomic fluorescence spectrometry (AFS). [4 marks]
- (d) Use a schematic diagram to outline the instrumentation in HG-AFS. [7 marks]

Question FIVE

Resonance ionization spectroscopy (RIS) involves use of resonant laser light at different wave lengths.

- (a) What is Laser? State four classes of laser [5 marks]
- (b) Explain principle involved in Resonance ionization spectroscopy (RIS) and outline four main advantages of RIS. [9 marks]
- (c) What is population inversion as applied in Resonance ionization spectroscopy (RIS)? Explain why laser pump is required in RIS and how the pumping energy is delivered. [6 marks]
- (d) Outline the application of Resonance ionization spectroscopy (RIS) [5 marks]

Question SIX

- (a) Describe hyphenated techniques in advance spectroscopic techniques by making use of relevant example and outline their advantages. [8 marks]
- (b) Make use of a schematic presentation to outline the principle of excitation, signal generation and detection in a photoacoustic experiment. [7 marks]
- (c) State any ten areas of application of photoacoustic spectroscopy. [10 marks]