



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: AUGUST 2017

TIME: 3 HOURS

DATE: Pick Date Aug 2017

Instructions to Candidates

You should have the following for this examination

-Answer Booklet, examination pass and student ID

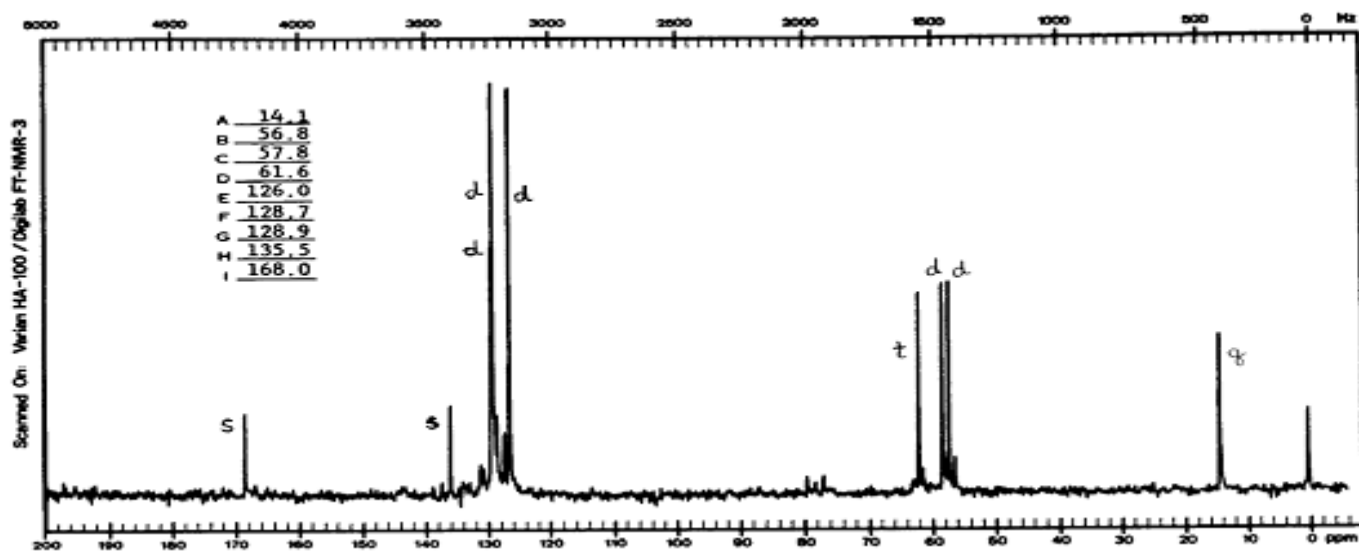
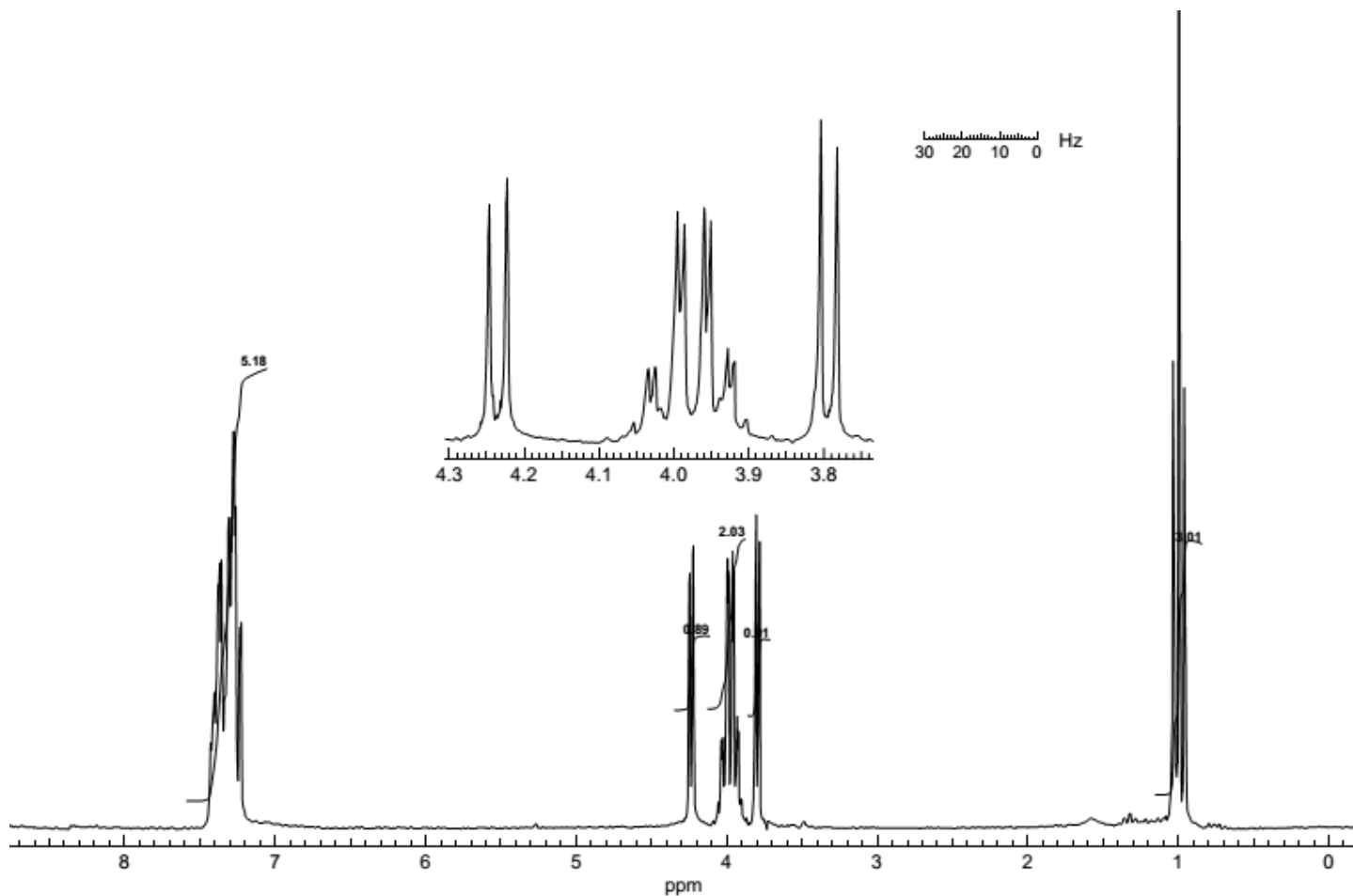
This paper consists of **SIX** Question(s). Attempt any **FOUR** questions.

Do not write on the question paper.

Question ONE

(a) What is peak resolution as applied in ^1H -NMR Spectroscopy? State four main factors which affect peak resolution in NMR spectroscopy. **[5 marks]**

(b) An organic molecule which has the molecular formula $\text{C}_{11}\text{H}_{12}\text{O}_3$ registered IR absorption signals associated with a carbonyl group and Aromatic ring. The ^1H -NMR and ^{13}C -NMR spectra of the molecule are given below:



- i. Compute the DBE of the molecule. [2 marks]
- ii. Identify significant peaks in the ^{13}C -NMR spectrum of the compound and describe the structural information you obtain from them. [6 marks]

- iii. Draw the structure of the molecule and, label the structure with ^1H chemical shifts and multiplicity of the protons in the same magnetic environment. [10 marks]
- iv. What feature(s) of the spectra would you use to determine the stereochemistry of the molecule? [2 marks]

Question TWO

- (a) Outline the theory of Electron spin resonance spectrometry. [6 marks]
- (b) Give detailed account on instrumentation of electron spin resonanace spectrometry. [6 marks]
- (c) Discuss the two relaxation methods in Electron spin resonance spectrometry. [8 marks]
- (d) Outline the applications of Electron spin resonance spectrometry in in nutraceutical and food research. [5 marks]

Question THREE

- (a) What is photoacoustic spectroscopy? Outline its main difference from conventional optical techniques. [3 marks]
- (b) Outline the difference between thermally thick sample and thermally thin sample as applied in Photoacoustic spectroscopy. [4 marks]
- (b) Describe the sample cell and detectors used in Photoacoustic spectroscopy. [5 marks]
- (c) Outline the properties of sample which affect photoacoustic signal [6 marks]
- (d) Outline the principle and advantages of gas phase photoacoustic spectrometry. [5 marks]

Question FOUR

- (a) Outline the role of various components of an NMR spectrometer. [7 marks]
- (b) Describe proton decoupled and off resonance decoupled techniques used in ^{13}C NMR. [6 marks]
- (d) Explain why
- i. In ^{13}C NMR proton less carbon exhibits low intensity. [3 marks]
- ii. CDCl_3 exhibits a triplet at δ 76, 77 and 78 in its ^{13}C NMR spectrum. [2 marks]
- (e) Give an account of deuterium substitution used in ^{13}C NMR. [7 marks]

Question FIVE

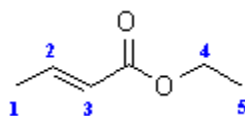
(a) What is two dimensional NMR? [2 marks]

(b) Describe the following two dimensional spectroscopy techniques

i. HETCOR [5 marks]

ii. COSY [6 marks]

(c) Make use of a sketch to describe COSY spectrum of ethyl 2-butenoate (X). Label the proton signals with the numbers provided on the structure of the molecule. [8 marks]



X

(d) Outline the two main relaxation processes in NMR spectroscopy [4 marks]

Question SIX

(a) Explain why hyphenated techniques usually involve chromatographic and spectroscopic techniques. [4 marks]

(b) Describe the following hyphenated techniques clearly indicating their instrumentation and application.

i. LC – NMR [7 marks]

ii. GC – NMR [7 marks]

iii. GC-MS [7 marks]