UNIVERSITY OF JAFFNA, SRI LANKA
**BACHELOR OF PHARMACY**
FOURTH YEAR FIRST SEMESTER EXAMINATION – NOVEMBER 2014
PHAPA 4101 PHARMACEUTICAL ANALYSIS
PAPER II

Date: 24/11/2014   **Time: 02 Hours**

1 1.1 What is the principle of high performance liquid chromatography (HPLC)? (20 Marks)
   1.2 What are the strengths and limitations of the HPLC? (20 Marks)
   1.3 Draw a schematic diagram to illustrate the Diode Array Detector (DAD) and Refractive Index Detector (RID). (40 Marks)
   1.4 The retention time of the acidic drug naproxen on an octadecylsilyl (ODS) column with a (t_o) of 2.3 minutes in a mixture containing acetonitrile/0.05M acetate buffer pH 5.2 (40:60) is 9.07minutes. The pKa of naproxen is 4.2; what would be the effect of reducing the pH of the mobile phase to 4.2? (20 Marks)

2. 2.1 What is the basic principle of the mass spectrometry? (20 Marks)
   2.2 Describe the electron ionization method and the electrospray ionization method used in the mas-spectrometry (30 Marks)
   2.3
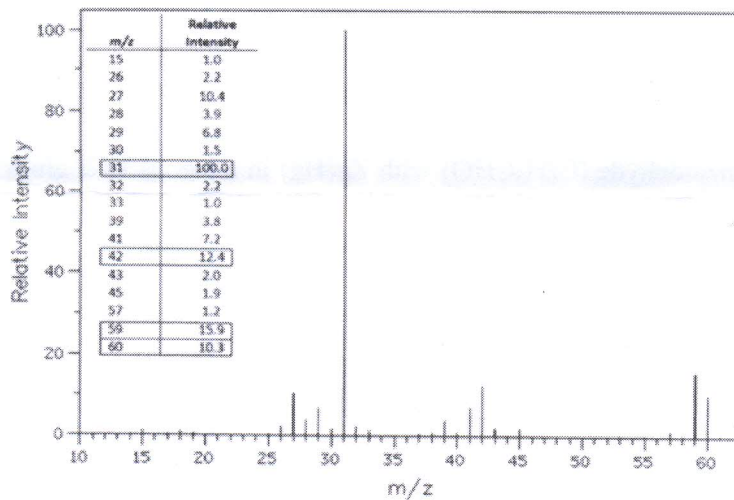
The above data obtained from electron ionization mass spectrometry for the molecule of 1-propanolol.
2.3.1 Draw the structure of the most likely molecular ion. (20 Marks)
2.3.2 Draw the structure of ion responsible for the base peak at $m/z = 31$. Provide a reaction mechanism of the fragmentation that will lead to that ion. (20 Marks)
2.3.3 Why does the peak at $m/z = 15$ have such a low intensity? (10 Marks)

3 3.1 Draw a schematic diagram to illustrate the electron transition that occurs in the atomic emission spectroscopy (AES). (20 Marks)
   3.2 Explain the interferences in AES analysis? (30 Marks)
   3.3 A pharmaceutical product is suspected to be contaminated with strontium (Sr). A research student in a pharmaceutical laboratory has conducted an AES experiment to determine the concentration of strontium in the pharmacetutical product. The data obtained as follows.

		Sr Conc (mg/L)	Sr Emission (nm)
Standards		0.00	0.0
		1.00	16.6
		2.00	37.8
		3.00	43.2
		4.00	68.7
		5.00	95.2
Sample		A	45.6
Sample		B	102.7

Determine the strontium concentration (mg/L) in each of the samples.　　　(50 Marks)

4　4.1　Using H_2O as a molecule, explain vibrational modes in the Infra-Red (IR) spectroscopy.　　　(20 Marks)

4.2　What are the strengths and weakness of IR spectroscopy?　　　(30 Marks)

4.3
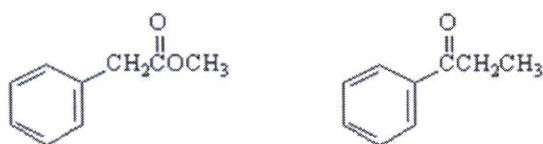
Explain how IR spectroscopy could be used to spcifically distinguish between the above two compounds.　　　(30 Marks)

4.4　Treatment of benzaldehyde (C_6H_5CHO) with Zn(Hg) in aqueous HCl gives a product Z that has a molecular ion at m/z = 92 and IR absorptions at 3150-2950, 1605, and 1496cm^{-1}. Draw the structure of Z.　　　(20 Marks)
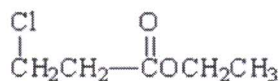
5　5.1　How can you obtain structural informations from ^1H NMR?　　　(20 Marks)

5.2
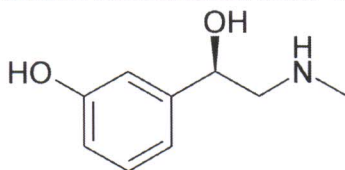
The above structure represent the phenyephrine. Predict the ^1H NMR spectrum and assign the protons in the ^1H NMR spectrum.　　　(60 Marks)

5.3

Predict the multiplicity of each hydrogen enviroments in the above compound.　　　(20 Marks)

6　6.1　Define the term "Quality Control"　　　(20 Marks)

6.2　List the factors that determine the drug quality.　　　(40 Marks)

6.3　Explain the basic requirements of the quality control.　　　(40 Marks)