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RAJARATA UNIVERSITY OF SRI LANKA

FACULTY OF APPLIED SCIENCES

B.Sc. (Special) Degree in Chemistry
Third Year – Semester II Examination – April / May 2016

CHE 3218 ADVANCED ORGANIC CHEMISTRY - LABORATORY

Answer ALL Questions **Time: Six (06) hours.**

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01). Follow the below procedure to determine the fatty acid (FA) composition of the oil provided.

a. Weighted about 50 mg of oil sample (___) into screw cap tube.

b. Add 1.0 ml of 0.5 mol dm^{-3} methanolic sodium hydroxide and tight the cap firmly.

c. Place the tubes in a boiling water bath for until the oil dissolved (about 5 mins).

d. Cool the solution to room temperature (**do not open the lid while in hot**), add 1.5 ml of Boron trifluoride reagent and 0.5 ml of 0.1% hydroquinone solution into the test tube. Tight the cap firmly.

e. Place in boiling water bath for 5 mins.

f. Cool the solution to room temperature **(do not open the lid while in hot)**, 5.0 ml of saturated sodium chloride and 2.5 ml of n- heptane to the test tube, then close the cap

g. Shake the mixture vigorously for 30s and keep until separate the phases. Transfer the organic phase in to an injection vial and label with index number.

h. Hand over the vial for GC analysis.

1.1 Identify your sample by compared with standard chromatograms given.

1.2 Write the relevant equations of the above procedure.

02).

a. Add 1 g of provided sample A/B in to test tube and add 2 mL of provided solution C/D to the above test tube and stir the mixture using a glass road.

b. Add 10 drops of provided solution E to the above mixture and continue stirring for further 10 minutes.

c. Place the test tube containing above mixture in a beaker of hot water for 20 minutes to dissolve solid material

d. Then transfer solution in to a 125 mL conical flask containing 40 mL of water, rinse the test tube with distilled water and stir well.

e. Cool thoroughly in ice to induce crystallization.

f. Collect the crystalline solid using a Buchner funnel.

g. Re-crystalize the product using suitable solvent (F /G).

2.1

i. Calculate the % yield of the product.

ii. Run the TLC of the product obtained at section (g) using solvent (H) and (I) separately and calculate the R_f values for each solvent.

iii. Dissolve small quantity of your sample (f) in 3ml of ~~distilled water~~ ethanol and obtain the UV-Visible spectrum, and identify your product by comparing with the standard UV-Visible spectra given bellow

iv. Write the IUPAC name of your product

v. Identify the A/B, C/D and E.

vi. Write the reaction mechanism for the synthesis of your products.

vii. Select the suitable FTIR spectrum for your product from the three spectra given below and assign the relevant bands.

UV-Visible spectra of products given below

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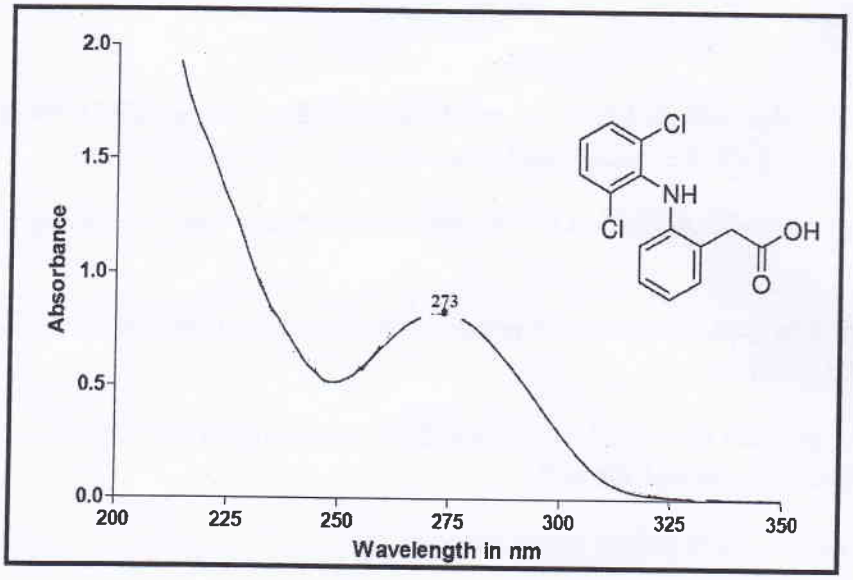
(P)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Wavelength in nm
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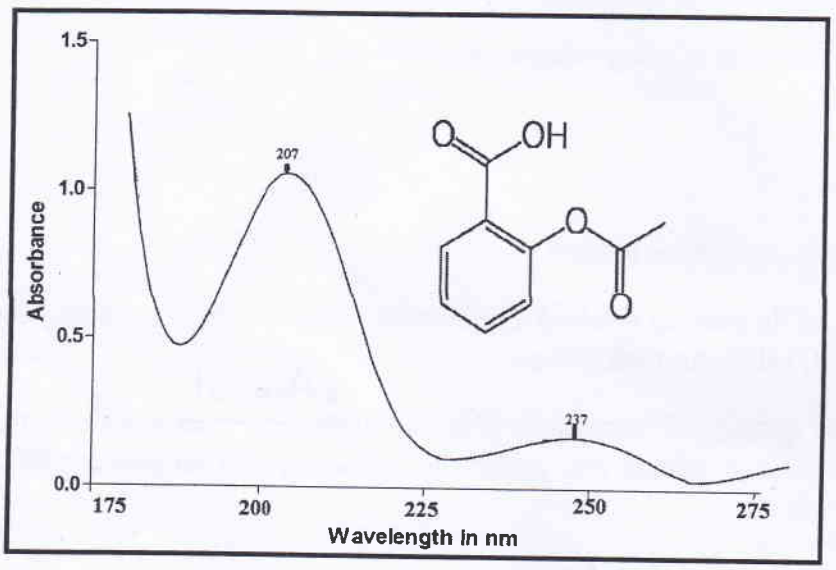
NH
Cl
OH
O

(Q)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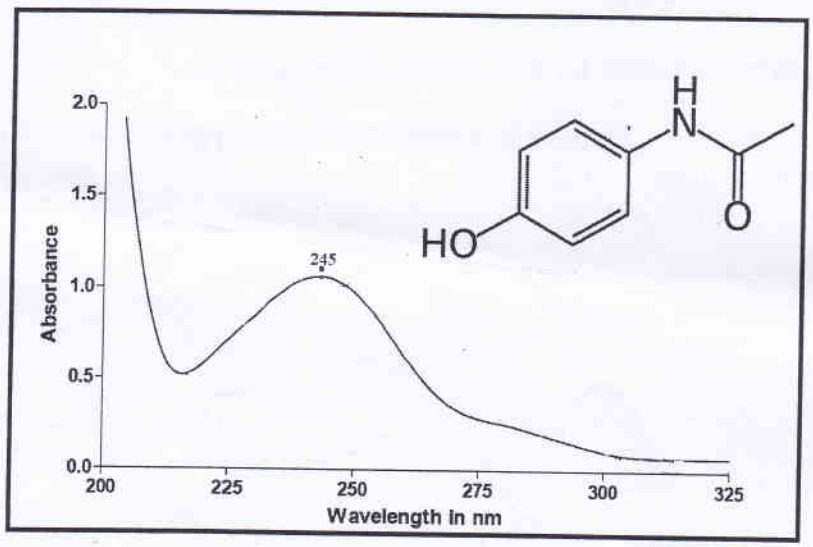
OH
O
O

(R)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200
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250
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300
325
Wavelength in nm
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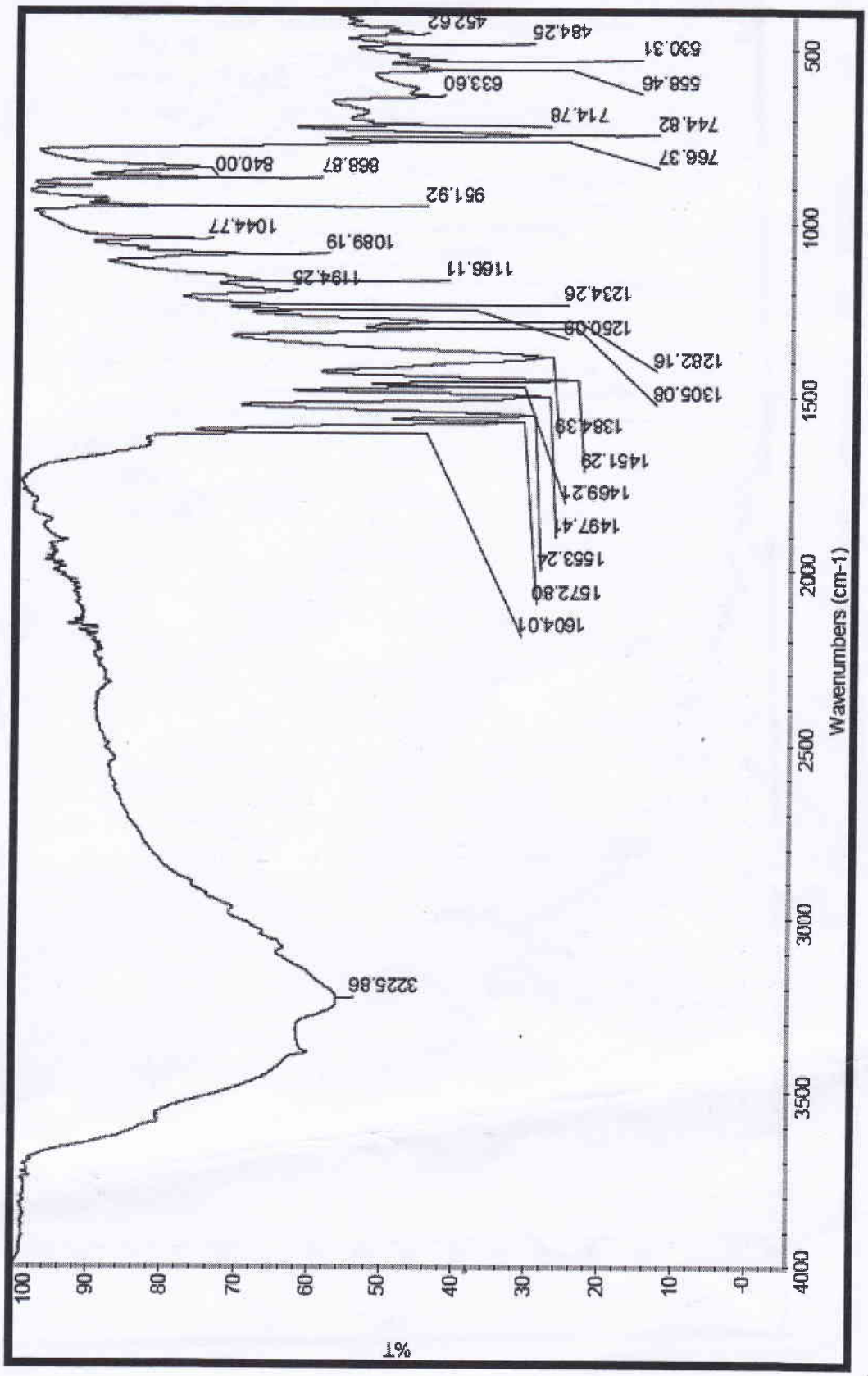
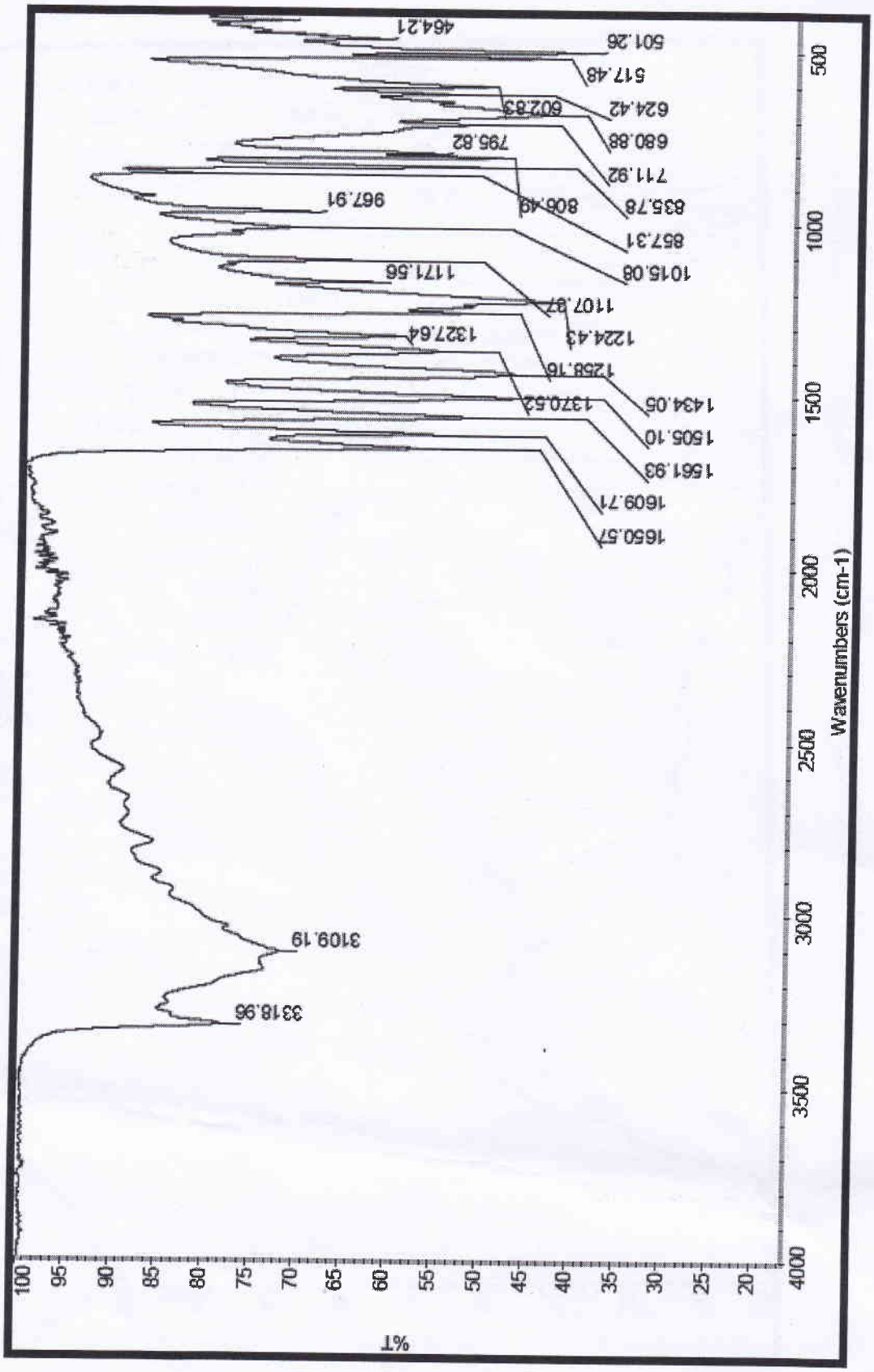
H
N
HO
O

Related FTIR spectra of above suggested compounds

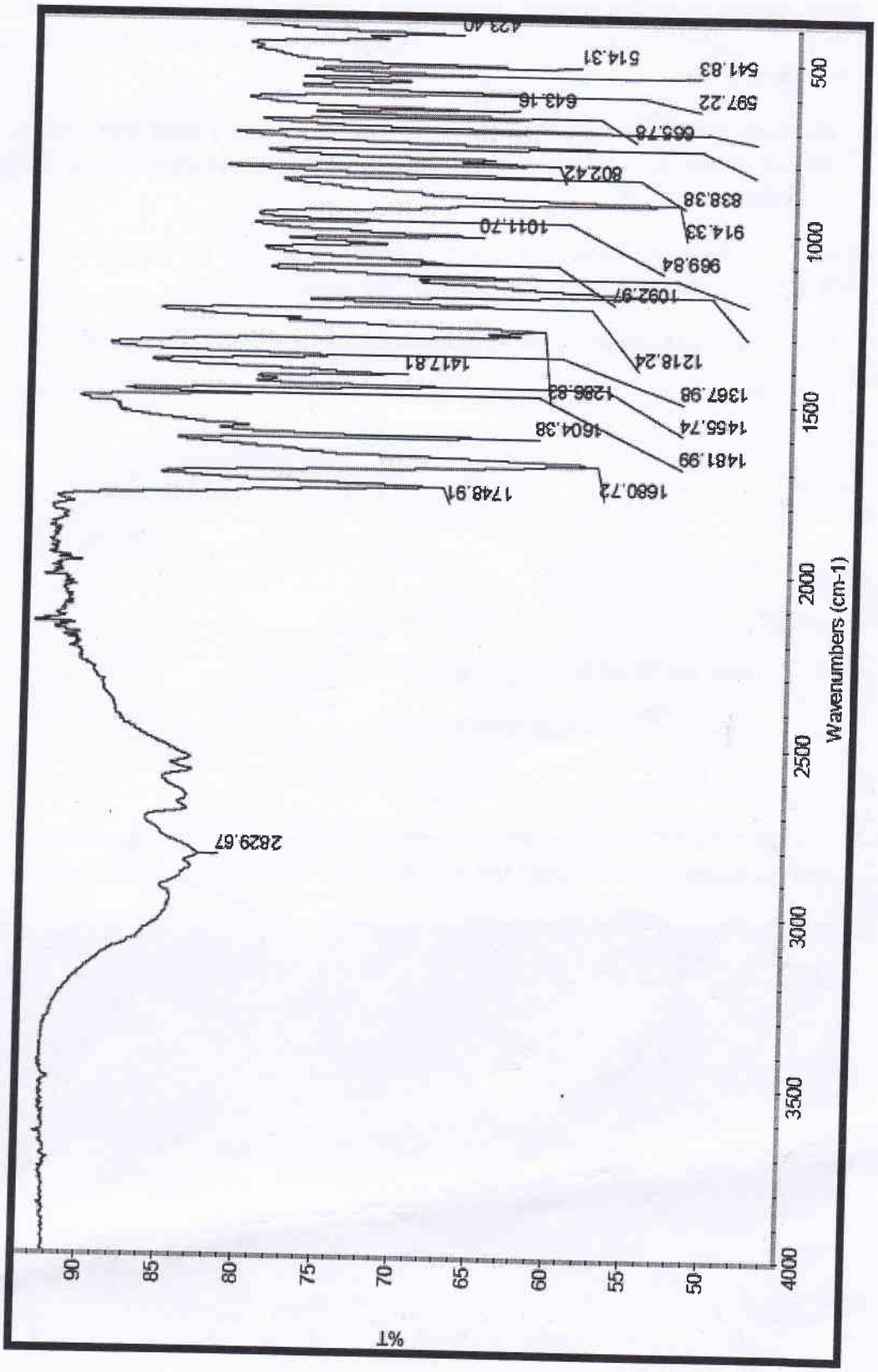
FTIR Spectrum of .....(P/Q/R)



FTIR Spectrum of.....(P/Q/R)



FTIR Spectrum of .....(P/Q/R)



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03). Last week, one of your friend placed six chemical bottles on your chemical rack. Those are Alkaline Potassium Permanganate solution, Cinnamic Acid ($C_6H_5(CH)_2C(O)_2H$), Distilled water, Aqueous Chloride solution, Bromine solution and Chloroform. However, out of these six bottles four labels were removed from respective chemical bottles except in Bromine and Chloroform bottles.

a. By using your laboratory experience, design simple experiment in order to qualitatively identify above four unknown samples by using any above chemicals and separation and analytical techniques.

Note:- You are not allow to use any other chemicals and pH papers/ meters except HCl, H_2SO_4, EtOH, MeOH, Hexane, Heptane and Dichloromethane.

b. How many possible reactions presence in your experiment design?

c. Draw all possible reaction mechanisms of them

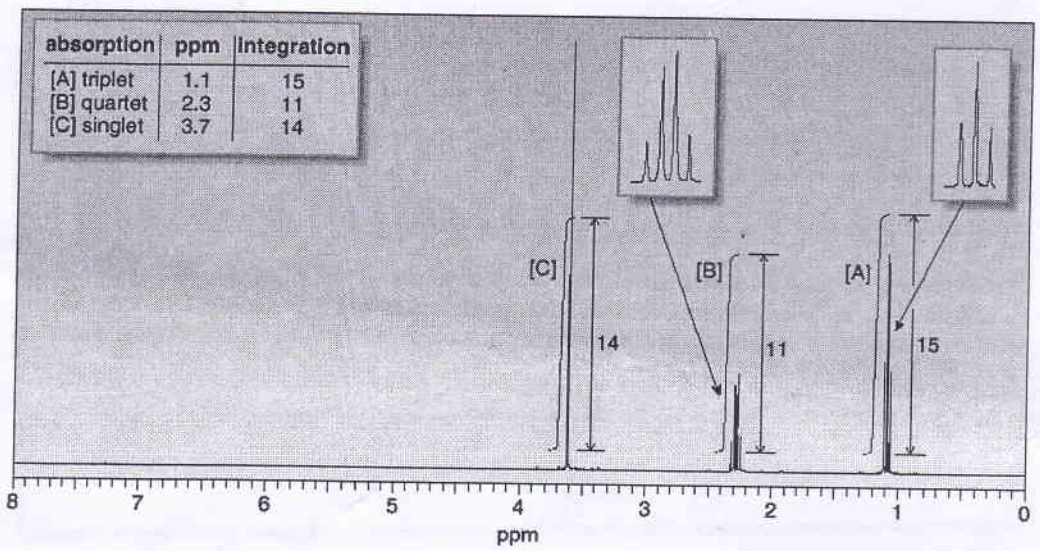
04).

4.1

a). Draw the ^1H NMR of Benzoic acid

b). Draw the ^{13}C NMR of Ethyl Acetate

4.2 Using the its 1H NMR spectrum, determine the structure of an unknown compound X that has molecular formula C_4H_8O and contains a C=O absorption in its IR spectrum.



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