MEGA LECTURE

TOPIC 20 ANSWERS TO EXERCISES

Topic 20 Exercise 1

1. To separate components in a mixture and identify them
2. TLC – solvent moves up plate coated with solid; CC – a solvent moves down a column packed with solid; gas, under pressure at high temperature, is passed through a column, packed with a solid or coated by a liquid
3. The different components have different relative solubilites in the mobile phase and are retained by the stationary phase to different extents
4. Using Rf values
5. Using retention times and by mass spectrometry of the separated components
6. Separated by thin layer chromatography, located using developing agents such as ninhydrin or ultraviolet light, and identified by their Rf values

Topic 20 Exercise 2

1. a) Propanal has three peaks, propanone has two
   b) Both have two peaks, one with chemical shift between 0 and 50. However the second peak in propanone will have a chemical shift at 160 – 220, but the second peak in propan-2-ol will have a chemical shift at 50 - 90
   c) i) 4 ii) 5 iii) 3
2. Peak at 60 ppm C-O and peak at 160 ppm O=C-O so ester is most likely
   $C_nH_{2n}O_2$ = 116 so n = 6
   Five peaks so two C atoms are in identical environments, likely $-C(CH_3)_2$
   $HCOOCH_2CH_2CH(CH_3)_2$ or $HCOOCH(CH_3)CH(CH_3)_2$ or
   $CH_3COOCH_2CH(CH_3)_2$ or $CH_3CH_2COOCH(CH_3)_2$ or
   $(CH_3)_2CHCOOCH_2CH_2CH_3$ or $(CH_3)_2CHCOOCH_2CH_3$ or
   $(CH_3)_2CHCH_2COOCH_3$
3. Peak at 60 ppm C-O so alcohol is most likely
   $C_nH_{2n+2}O$ = 74 so n = 4
   Two peaks so three C atoms are in identical environments, likely $-(CH_3)_3$
   $(CH_3)_3COH$
4. Peak at 210 ppm = C=O so carbonyl most likely
   $C_nH_{2n}O$ = 84 so n = 5
   Five peaks so no C atoms in identical environments
   $CH_3CH_2CH_2CH_2CHO$ or $CH_3CH_2CH(CH_3)CHO$ or $CH_3CH_2CH_2COCH_3$
5. Peak at 180 ppm = O=C-O so carboxylic acid most likely
   $C_nH_{2n}O_2$ = 102 so n = 5
   Four peaks so two C atoms in identical environments, likely $-C(CH_3)_2$
   ($(CH_3)_2CHCH_2COOH$

Topic 20 Exercise 3

1. peak at 1.1 is CH_3- adjacent to $-CH_2$-
   peak at 2.2 is $-CH_2CO$-, adjacent to CH_3-

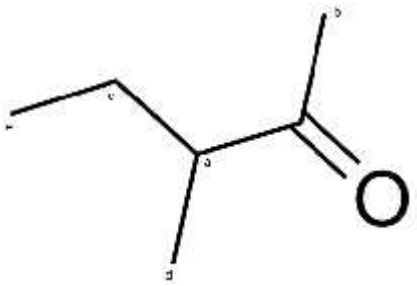
peak at 11.8 is –COOH
so molecule is propanoic acid, CH_3CH_2COOH

2. a) peak at 1.2 is CH_3-, adjacent to -CH_2-
peak at 1.3 is also CH_3-, adjacent to -CH_2-
peak at 2.3 is -CH_2CO-, adjacent to CH_3-
peak at 4.1 is -CH_2O-, adjacent to CH_3-
so molecule is ethyl propanoate, $CH_3CH_2COOCH_2CH_3$

b) $CHCl_3$ is not used as a solvent because it contains a proton which will interfere with the spectrum of the substance being analysed.

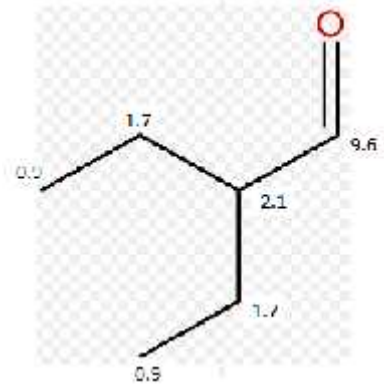
c) TMS is a good standard because
- it contains 12 identical protons, giving a single intense peak
- it contains highly shielded protons, which do not interfere with the spectrum
- it is cheap and non-toxic

3. e is -C<u>H</u>$_3$ adjacent to -C<u>H</u>$_2$- (c)
b is -C<u>H</u>$_3$ adjacent to -C=O
d is -C<u>H</u>$_3$ adjacent to -C<u>H</u>- (a)
a is -C<u>H</u>- adjacent to -C=O



3-methylpentan-2one

4. 9.6 is -C<u>H</u>O adjacent to -C<u>H</u>- (2.1)
2.1 is -C<u>H</u>- adjacent to -C<u>H</u>O (9.6)
0.9 is 2 x -C<u>H</u>$_3$ adjacent to -C<u>H</u>$_2$- (1.7)



ethylbutanal

5. 4.1 is O-C<u>H</u>$_2$- adjacent to -C<u>H</u>$_3$ (1.3)
   2.1 is C<u>H</u>$_3$C=O
   $CH_3COOCH_2CH_3$ (ethyl ethanoate)
   (2.1) (4.1)(1.3)
6. 2.4 is O=CC<u>H</u>$_2$ adjacent to C<u>H</u>$_3$ (1.1)
   2.2 is C<u>H</u>$_3$C=O
   $CH_3COCH_2CH_3$ (butanone)
   (2.2) (2.4)(1.1)
7. 0.9 is C<u>H</u>$_3$ adjacent to C<u>H</u>$_2$ (1.4)
   1.2 is C<u>H</u>$_3$ adjacent to C<u>H</u> (3.6)
   3.6 is O-C<u>H</u>-
   2.3 is -O<u>H</u>
   $CH_3CH_2CH(OH)CH_3$ (butan-2-ol)
   (0.9)(1.4)(3.6)(2.3)(1.2)
8. 11.6 is -COOH
   2.2 is O=CCH_2- adjacent to -CH_2- (1.9)
   1.0 is -CH_3 adjacent to -CH_2- (1.9)
   $CH_3CH_2CH_2COOH$ (butanoic acid)
   (1.0)(1.9)(2.2) (11.6)
9. 9.6 is -C<u>H</u>O adjacent to -C<u>H</u>- (2.3)
   2.3 is -C<u>H</u>C=O
   1.1 is 2 x CH_3 adjacent to -CH- (2.3)
   $(CH_3)_2CHCHO$ **(**methylpropanal)
   (1.1) (2.3)(9.6)

Topic 20 Exercise 4

1. empirical formula = $C_5H_{10}O_2$
   from mass spectrum mr = 102, efm = 102 so mf = $C_5H_{10}O_2$
   infra-red spectrum:
   peak at 1710 cm^{-1} indicates a carbonyl
   proton nmr spectrum:
   peak at 0.8 is CH_3- adjacent to -CH_2-
   peak at 1.1 is -CH_2- adjacent to CH_3- and -CH_2-
   peak at 2.3 is -CH_2CO- adjacent to -CH_2-
   peak at 3.7 is CH_3O-
   so molecule is methyl butanoate, $CH_3CH_2CH_2COOCH_3$
2. from mass spectrum mr = 60
   From ^{13}C nmr 60 ppm = C-O (alcohol)
   3300 cm^{-1} in IR spectrum confirms -OH alcohol
   $C_nH_{2n+2}O$ = 60 so n = 3
   No identical C environments so must be $CH_3CH_2CH_2OH$ (propan-1-ol)
3. from mass spectrum mr = 86
   From ^{13}C nmr 220 ppm = C=O (carbonyl)
   1700 cm^{-1} in IR spectrum confirms C=O
   $C_nH_{2n}O$ = 86 so n = 5

Three C environments only so either $(CH_3)_3CCHO$ (dimethylpropanal) or $CH_3CH_2COCH_2CH_3$ (pentan-3-one)