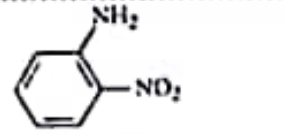
34.5

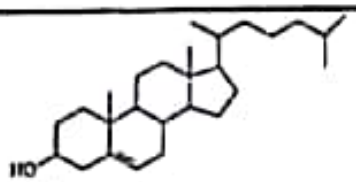
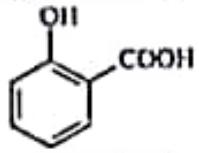
Organic Chemistry Laboratory | First Exam | Department of Chemistry
60 MIN. | 24 November 2016
NAME ..... SECTION 14 REGISTRATION No. 0130662

1 > (3 pts) Given the following table

| | |
|---|---|
|  *p*-Nitroaniline | M.p = 147-149 °C<br>$R_f$ value<br>Hexane 0.1 · $CH_2Cl_2$ 0.2 · ether 0.3 · $CH_3OH$ (0.8) |
|  *o*-Nitroaniline | M.p = 70-72 °C<br>$R_f$ value<br>Hexane 0.2 · $CH_2Cl_2$ 0.3 · ether 0.8 · $CH_3OH$ (0.9) |

- The best solvent for seperating a mixture of *ortho* and *para*-nitroaniline by TLC is methanol ($CH_3OH$)
- Which is more soluble in methanol *ortho* or *para*-nitroaniline? para-nitroaniline
- Which is more strongly adsorbed on the silica *o*-nitroaniline or *p*-nitroaniline Para nitroaniline

4 > (6 pts) Given the following table

| | |
|---|---|
|  cholesterol | M.p = 148-152 °C<br>Solubility (g/ 100 mL)<br>$H_2O$: Cold 0.1, Hot 0.3 · $CH_3OH$: Cold 0.3, Hot 3.2 · $CH_2Cl_2$: Cold 1.7, Hot 4.7 |
|  salicylic acid | M.p = 158-160 °C<br>Solubility<br>$H_2O$: Cold -ve, Hot -ve · $CH_2Cl_2$: Cold +ve, Hot +ve · $CH_3OH$: Cold +ve, Hot +ve |

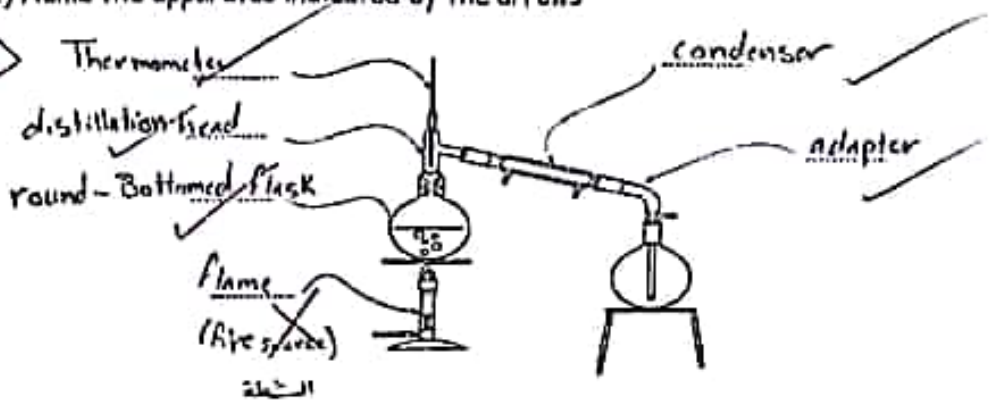
- The best solvent for recrystallization of cholesterol is $CH_2Cl_2$
- The best solvent for recrystallization of salicylic acid Solvent pair of $H_2O$ and $CH_3OH$
- A 1:1 mixture from cholesterol salicylic acid would start to melt at lower than 148 (higher that 161 / lower than 148 / between 148 and 160/ exactly at (160 + 148 )/2 °C).
- The melting point range of the cholesterol salicylic acid mixture would be more than 5 °C (1-2 °C, more than 5 °C, (160 + 140)/2, (160- 140)/2 °C).

2 - 20 => 5%

5

2.5

- (3 pts) Name the apparatus indicated by the arrows




7

- (7 pts) Given the following data concerning the fractional distillation for mixture of water (b.p 100°C) and acetone (b.p 56°C)

| Fraction No. | Temperature °C | Volume of distillate | Composition |
|---|---|---|---|
| 1 | 50-62 | 5 | Acetone |
| 2 | 62-72 | 3 | Acetone & water |
| 3 | 72-82 | 3 | Acetone & water |
| 4 | 82-95 | 2 | Acetone & water |
| 5 | residue | 8 | Water |

Answer the following questions:

- Write the composition of the distillate for each fraction (see the table)
- Which fraction (3 or 4) is more richer in water? 4
- If the thermometer is not kept moist during distillation, then the boiling point reading tends to be higher than (higher than/ lower than/ same as) the actual.

- At 70°C the vapor pressure of acetone is 400 (A) mm Hg and that of water is 100 (B) mm Hg, Calculate the vapor pressure of a mixture of acetone and water in 1 : 3 molar ratio

⊕ miscible in each other

$P_T = P^\circ_A x_A + P^\circ_B x_B$

$= 400 \times 0.25 + 100 \times 0.75$ #

$P_T = 175$ mm.Hg

$x_A = \frac{n_B}{n_T} = \frac{1}{4} = 0.25$

$x_B = \frac{n_B}{n_T} = \frac{3}{4} = 0.75$

1) (1 pt) During the isolation of caffeine from tea leaves, sodium carbonate is added to convert tannic acid to its salt (to precipitate caffeine/ convert tannic acid to its salt/ to prevent emulsions / as drying agent).

1) (1 pt) The reason the seperatory funnel is not shaken vigorously in the extraction of caffeine is to avoid formation of emulsion

3

11.5

4 > (4 pts) Assume the following solubilities (g/100 mL solvent) at room temperature of compounds A and B in the given solvents:

| | water | ether | benzene | chloroform | acetone |
|---|---|---|---|---|---|
| A | 1.0 | 5.0 | 3.0 | (9.0) | 13.0 |
| B | 3.0 | 6.0 | 3.0 | 1.5 | 1.0 |

- Calculate the distribution coefficient $K_D$ ($\frac{S_{ether}}{S_{water}}$) for compound A.

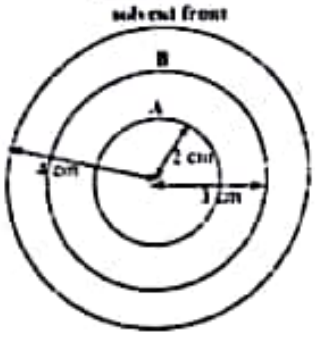
$$K_D = \frac{S_{ether}}{S_{water}} = \frac{5}{1} = 5$$

- The best solvent to extract compound A from water is Chloroform
  (Not acetone, Because acetone is soluble in water) ⊙
- Compound B has distribution coefficient ($K_D$)= 2, between ether and water. If 5.0 g of B are dissolved in 100 mL of water, calculate the weight of B that extracted with 50 mL ether.

$$K_D = \frac{C_w}{C_o} \Rightarrow K_D R = \frac{m_w/V_w}{m_o/V_o} \Rightarrow 2 = \frac{\frac{x}{50}}{\frac{5-x}{100}}$$

$x = 2.5$ g of B

2 > (2 pts) A mixture of two compounds A and B was chromatographed on a cellulose paper (filter paper) using isopropanol-water (2:1) as a mobile phase (eluent).




- The $R_f$ value for compound A is $\frac{2}{4} = 0.5$
- The compound which is more soluble in water is B

6 > (7 pts) Name the best technique, method, and (or) reagent which can be used to perform each of the following:

- Check the purity of a solid substance. Melting Point
- Decrease the solubility of the essential oil in the aqueous layer. Salting out (e.g. $Na_2CO_3$ or NaCl)
- Heating a flammable liquid. Water Bath
- Separate cyclohexane (b.p. 81 °C) from hexane (b.p. 69 °C). fractional distillation
- Separate dichloromethane and water Extraction ✗
- Separate a green dye into its components Paper Chromatography
- Remove colored impurities in recrystallization use Charcoal

12

- In the melting point determination, too large sample would increase (increase / decrease / has no effect) on the melting point range.

- Which of the following will not affect the measured melting point range of salicylic acid?

a) using too large amount of sample.

b) when rate of heating of the oil bath is slow.

c) the presence of pin-hole (ثقب) in the capillary tube which allows the oil liquid to enter the capillary.

d) using thick wall capillary tube.

(1) (1 pt) A water-soluble organic liquid decomposes upon heating to its normal boiling point. What technique you use to purify this liquid vacuum distillation

(1) (1 pt) Rapid cooling results in tiny (Large, tiny, medium sized) crystals.

(1) (1 pt) Insoluble impurities are removed by (simple filtration of hot solution / adding more solvent / evaporation of the solvent / suction filtration). gravity filtration of hot solution

(1) (1 pt) Filtration by "fluted filter paper" is recommended because:

a) it results in medium size crystals ↑ Rate of filtration

b) it slows down filtration giving large crystals

c) it prevents formation of crystals inside the filter paper because of rapid filtration.

(1) (1 pt) Colored impurities are removed by charcoal

(2) (2 pts) The following data refer to the vapor pressure of the two immiscible liquids *p*-xylene and water at temperatures shown:

| Temp. °C | 10 | 40 | 50 | 70 | 75 | 85 |
|---|---|---|---|---|---|---|
| P° $H_2O$ (mm Hg) | 74 | 122 | 294 | 437 | 540 | 940 |
| P° toluene (mm Hg) | 25 | 30 | 68 | 170 | 220 | 338 |

$P_T = 760$

- The boiling point of 1:3 mixture $H_2O$ : *p*-xylene at 760 mm Hg will be at:

a) 40°C b) 50°C c) 70°C d) 75°C e) 85°C

- During the distillation of the $H_2O$ / *p*-xylene mixture which fraction is richer in *p*-xylene

a) first fraction b) second fraction

c) the last fraction d) all fractions have the same composition