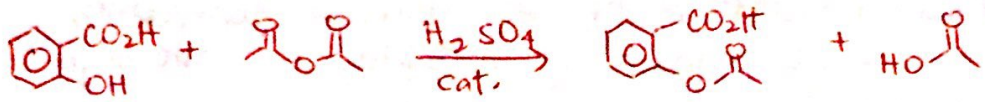
PRACTICE PROBLEMS

① A STUDENT PERFORMED THE EXPERIMENT ON THE SYNTHESIS OF ASPIRIN USING 6.90g salicylic acid (MW 138) and 3.00 mL acetic anhydride (MW 102, density 1.082 g/mL). If the RXN gave an 85.0% yield, HOW many GRAMS OF ASPIRIN DID THE STUDENT actually OBTAIN?

A) WRITE THE Balanced equation:



B) WHAT IS THE LIMITING REAGENT? EXPLAIN BY SHOWING:

* # OF MOLE OF SA:

$$6.90\,g\ SA \times \frac{1\ mol\ SA}{138\,g} = 0.0500\ mol\ SA$$

* # OF MOLE OF AA:

$$3.00\,mL \times \frac{1.082\,g\ AA}{mL} \times \frac{mol\ AA}{102\,g} = 0.0318\ mol\ AA$$

* LIMITING REAGENT

① $0.0500\ mol\ SA \times \frac{1\ mol\ ASPIRIN}{1\ mol\ SA} = 0.0500\ mol\ ASPIRIN$

② $0.0318\ mol\ AA \times \frac{1\ mol\ ASPIRIN}{1\ mol\ AA} = 0.0318\ mol\ ASPIRIN$ → ASA

smaller!

Thus, AA is the limiting reagent.

C) WHAT IS THE THEORETICAL YIELD? (MW of ASA)

$$0.0318\ mol\ ASA \times \frac{180.2\,g\ ASA}{mol} = 5.73\,g\ ASA$$

F) HOW MANY GRAMS OF ASPIRIN WERE OBTAINED IF THE YIELD IS 85%?

$$\frac{\text{actual yield}}{\text{Theo yield}} \times 100 = X\% \Rightarrow \text{actual } \% = \frac{(x\%)(\text{the } \%)}{100} \Rightarrow \text{Actual} = \frac{(85)(5.73)}{100}$$

$$= 4.87\,g$$

(2) RECRYSTALLIZATION:

YOU HAVE 10 GRAMS OF AN IMPURE COMPOUND & YOU WISH TO PURIFY BY RECRYSTALLIZATION. EIGHT GRAMS IS SOLUBLE IN 100 mL OF BOILING SOLVENT & 3 GRAMS IS SOLUBLE IN 100 mL OF COLD SOLVENT.

A) IF 130 mL OF THE FILTRATE (→ use cold) WAS COLLECTED AFTER VACUUM FILTRATION OF THE PURIFIED COMPOUND, WHAT IS THE AMOUNT OF MATERIAL LOST THROUGH SOLUBILITY?

$$130\ mL\ filtrate \times \frac{3.0\ g\ cold}{100\ mL} = 3.9\ g\ of\ material\ lost$$

B) IF 6.0g OF PURIFIED COMPOUND WAS COLLECTED, WHAT IS THE % RECOVERY?

$$\%\ RECOVERY: \frac{ASPIRIN}{ASA} \times 100 = \frac{6.0\ g}{10\ g} \times 100 = 60\%$$

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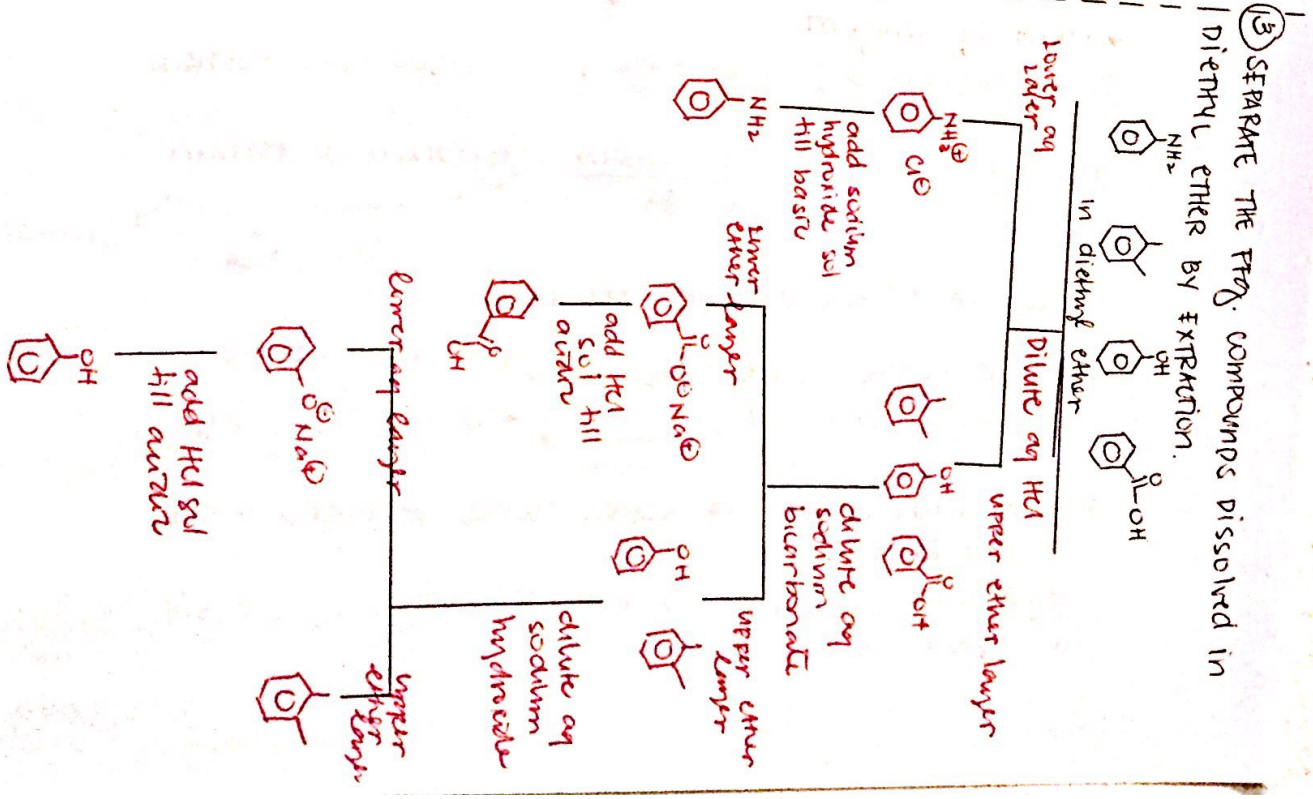
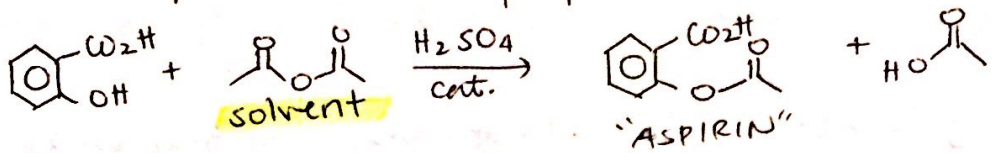
(3) SEPARATE THE FOLLOWING COMPOUNDS DISSOLVED IN DIETHYL ETHER BY EXTRACTION.



CHEM 113A QUIZ A STUDY GUIDE

① Balanced equation for the preparation of aspirin



limiting reagent

FLOW CHART

Salicylic acid + Acetic anhydride ← STEP 1

H_2SO_4 heat

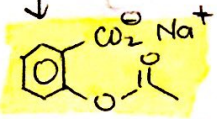
ASA, salicylic acid, acetic acid + impurities

Add H_2O, Chill, Vacuum Filter ← STEP 2

solution: $HOAc$, H_2SO_4, SA, trace ASA [DISCARD]

solid: ASA + impurities ← Perform $FeCl_3$ Test

Add aq. $NaHCO_3$ ← STEP 3, Grav. Filter

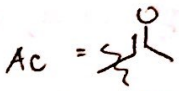
solution: 

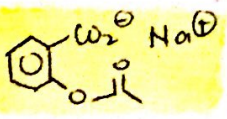
solid: impurities [DISCARD]

Acidify to pH < 2, Chill, Vac. Filter

solution: NaCl, HCl, trace ASA [DISCARD]

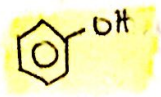
solid: ASPIRIN ← Perform $FeCl_3$ Test

Ac = 
carboxylic acid → CO_2H

②  ← soluble in H_2O b/c of its ionic character.

③ a phenol (alcohol) would test ⊕ in the $FeCl_3$ test.

↓



④ You are going to prepare aspirin (MW: 180.2) starting with 10.00 g of salicylic acid (MW = 138.1) & 1 mL of conc sulfuric acid.

A. Acetic anhydride (MW: 102.1, density: 1.08 g/mL) is used with a 3-fold excess. Calculate the vol in mL of acetic anhydride needed.

$$\rightarrow 10\,g\,SA \times \frac{1\,mol\,SA}{138.1\,g} \times \frac{3\,mol\,AA}{1\,mol\,SA} \times \frac{102.1\,g}{1\,mol\,AA} \times \frac{mL}{1.08\,g} = \boxed{20.5\,mL\ AA\ needed}$$

B. What is the theoretical yield? (in g)

$$10\,g\,SA \times \frac{1\,mol\,SA}{138.1\,g} \times \frac{1\,mol\,ASA}{1\,mol\,SA} \times \frac{180.2\,g\,ASA}{1\,mol\,ASA} = \boxed{13.05\,g\ ASA}$$

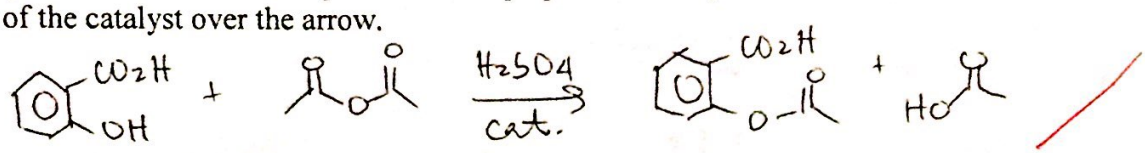
C. If your actual yield is 8.50 g, % yield?

$$\%\ yield: \frac{8.50\,g}{13.05\,g} \times 100 = \boxed{65.1\ \%}$$

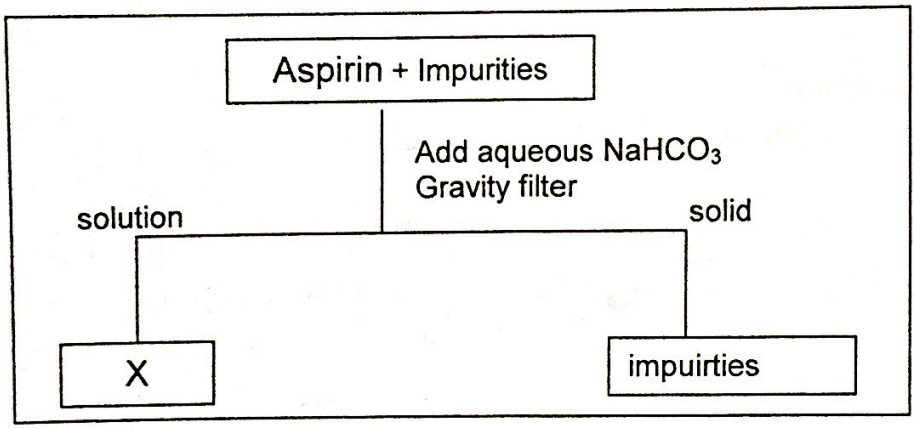
QUIZ A
Chem 113A

Name: Theresa Santos
Aspirin Synthesis
Grade 9.5 **/10 pts**

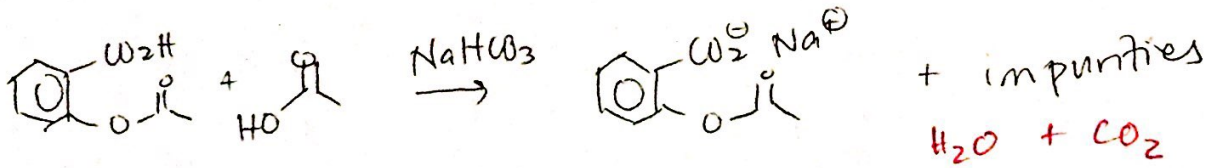
1. [1.5pt] Provide the balanced equation for the preparation of aspirin in this experiment; include the identity of the catalyst over the arrow.



2. [2pts]In the purification of crude aspirin, the flow diagram shows the step taken.



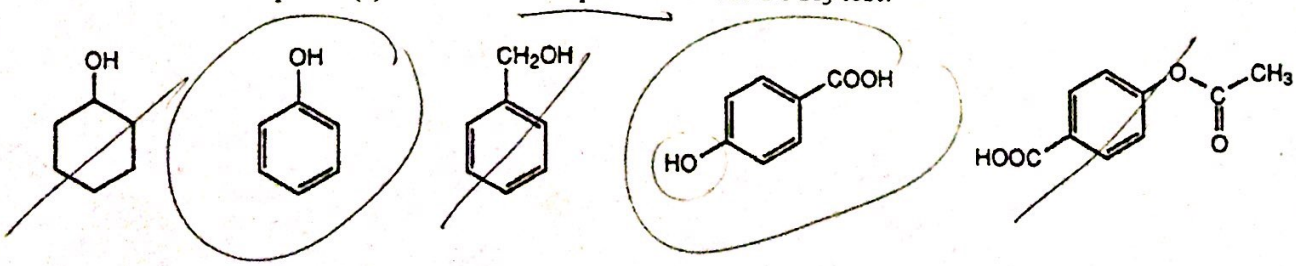
Provide the balanced equation in the formation of X and why is X soluble in the aqueous solution?



soluble because of its ionic character.

-0.5

3. [2.5pt] Circle the compound(s) that would test positive in the $FeCl_3$ test:



4. (Show work to get full credit) You are going to prepare **aspirin** (MW: 180.2) starting with 6.00 g of **salicylic acid** (MW= 138.1) and 1 mL of concentrated sulfuric acid.

A. [2 pt] Acetic anhydride (MW:102.1, density: 1.08 g/mL) is used with a 3-fold excess [*3 times the number of moles required*] . Calculate the volume in mL of acetic anhydride needed.

$$6\text{ g SA} \times \frac{1\text{ mol SA}}{138.1\text{ g}} \times \frac{3\text{ mol AA}}{1\text{ mol SA}} \times \frac{102.1\text{ g}}{1\text{ mol AA}} \times \frac{1\text{ mL}}{1.08\text{ g}} = 12.3\text{ mL}$$

acetic anhydride needed.

B. [1 pt] What is the theoretical yield (in g)?

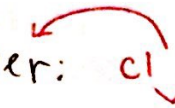
$$6\text{ g SA} \times \frac{1\text{ mol SA}}{138.1\text{ g}} \times \frac{1\text{ mol ASA}}{1\text{ mol SA}} \times \frac{180.2\text{ g ASA}}{1\text{ mol ASA}} = 7.83\text{ g ASA}$$

C. [1 pt] If your actual yield is 5.50g, what is your percent yield?

$$\frac{5.50\text{ g}^{AY}}{7.83\text{ g ASA}} \times 100 = 70.2\%$$

EXPERIMENT B REVIEW

① will NOT Have two layers: acetone + H_2O
diethyl ether + ethyl acetate

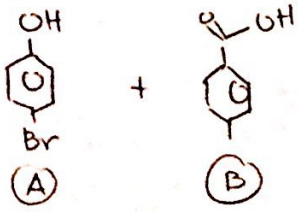
LOWER LAYER:  + H_2O

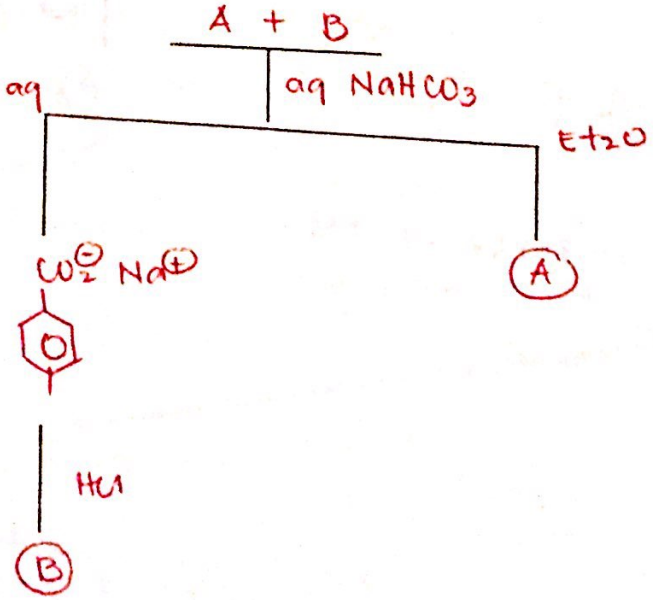
TOP LAYER:  + H_2O
ethyl acetate

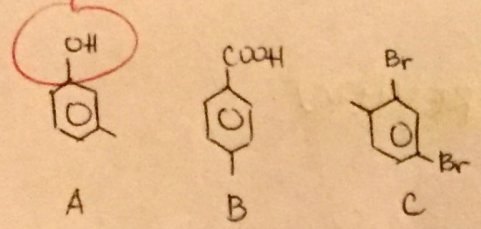
② UNSUITABLE FOR EXTRACTION: ethanol + H_2O, b/c ethanol is miscible in H_2O.

WATER AS THE UPPER LAYER: Chloroform + H_2O, because chloroform (1.50 g/mL) is denser than H_2O (1.00 g/mL)

③ FLOW DIAGRAM TO SEPARATE THE TWO COMPOUNDS.
use $NaHCO_3$



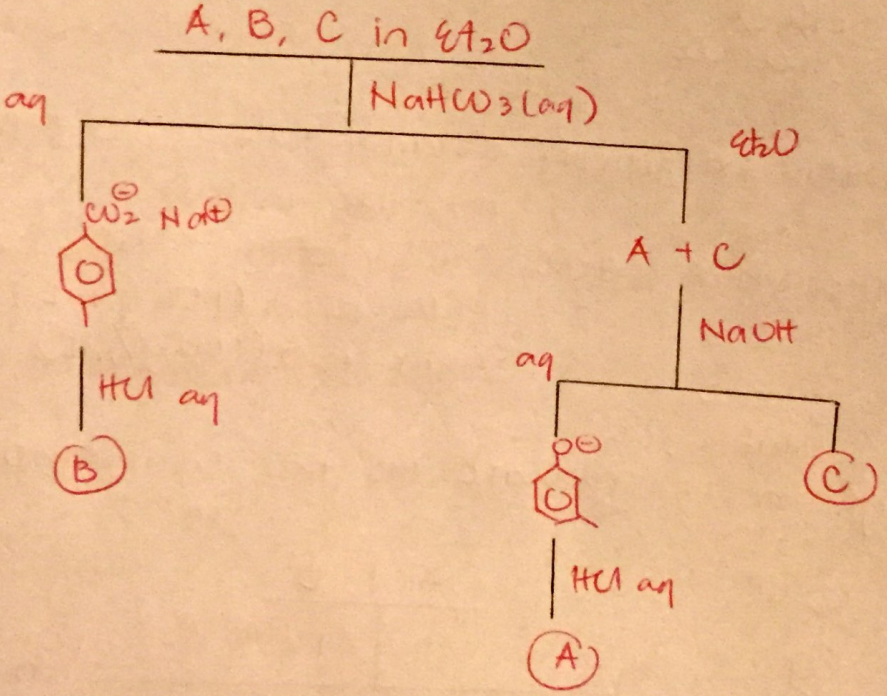


④ 

are found in diethyl ether

* extracted by aq NaOH sol: A + B
* extracted by aq $NaHCO_3$: B

* SEPARATE THE 3 COMPOUNDS:



DISTRIBUTION COEFFICIENT

(A) → $10-x$; $\frac{x}{100}$ (B)

(5) 10g A is dissolved in 100 mL H_2O. The distribution coefficient for compound A between hexanes and H_2O is 4:

$K_{A(Hexanes/H_2O)} = 4$ (C)

A) How much of A will be in the hexanes if you extract it from the water one time with 80 mL of hexanes? (D)

$$\frac{\left(\frac{A}{D}\right)}{B} = C \rightarrow \frac{\left(\frac{10-x}{80}\right)}{\frac{x}{100}} = 4 \Rightarrow \frac{10-x}{80} = 4\left(\frac{x}{100}\right)$$

$x = 2.38$

so, 10g A − 2.38 = **7.6 g A in hexane**

B) How much of A will be in the hexanes if you extract it from the water with two times sequential extractions using 40 mL hexanes each time, & then combine the hexanes extracts? (D_2)

A_2

* $\frac{10-x}{40} = 4\left(\frac{x}{100}\right)$; $x = 3.8$

1st EXTRACTION: 10g A − 3.8 = 6.15g

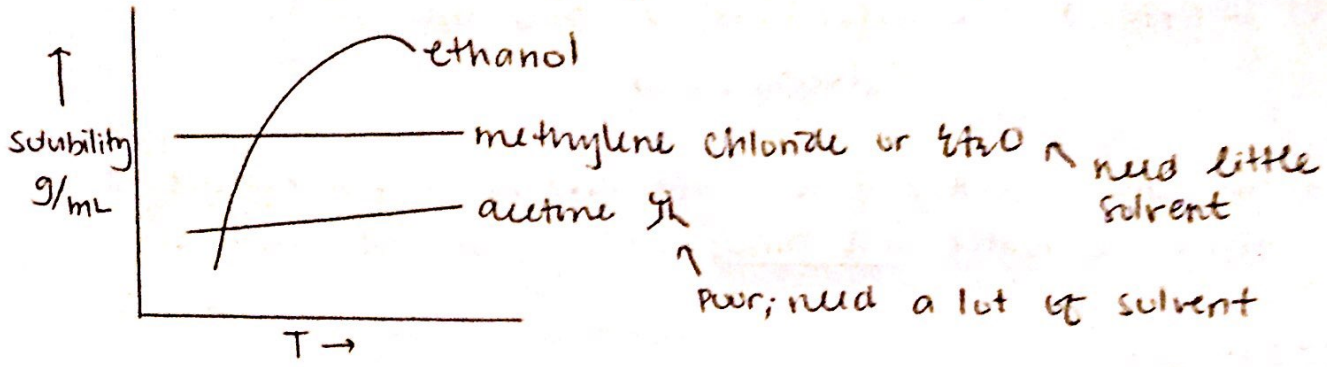
* $\frac{3.8-x}{40} = 4\left(\frac{x}{100}\right)$; $x = 1.47$

2nd EXTRACTION: 3.8 gA − 1.47 = 2.34g

6.15 + 2.34

8.5g A in hexane

① solubility vs temp graph for benzophenone:

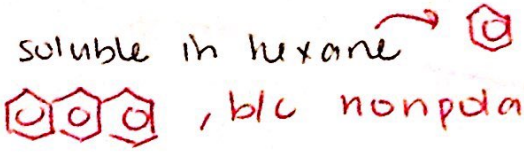


* WHICH SOLVENT IS BEST CHOICE FOR RECRYSTALLIZATION OF BENZOPHENONE?
Ethanol, b/c it is soluble at ↑T and insoluble at ↓T.

② TWO (DIFF) COMPOUNDS, A + B, HAVE THE SAME mp OF 111-112 °C. THE melting POINT OF THE MIXTURE OF A + B will be:
Lower than 111-112 °C

③ Increasing polarity


④ * soluble in ethanol → OH ~~alc~~ (polar)
glucose, b/c like dissolves like. glucose has OH can dissolve in OH

* soluble in hexane →

, b/c nonpolar dissolves in nonpolar hexane.

RECRYSTALLIZATION

COMPOUND	COLD sol. in 0°C H_2O (g/100 mL)	HOT sol. in 100°C H_2O (g/100mL)
acetanilide	0.54	5.0
Phenacetin	0.076	1.22

IF YOU HAD 1.50g OF CRUDE acetanilide TO PURIFY

(A.) TO START THE RECRYSTALLIZATION, HOW MUCH WATER IS NEEDED TO DISSOLVE THE IMPURE acetanilide?

need hot water

$1.50\,g\ crude \times \frac{100\,mL}{5.0\,g} = \boxed{30\ mL}$ hot water

(B.) How much acetanilide will be left in the filtrate [assume VOLUME FROM A.]? → use cold H_2O

$30\,mL \times \frac{0.54\,g}{100\,mL} = \boxed{0.162\,g}$ acetanilide

(C.) How much RECRYSTALLIZED acetanilide is expected?

$1.50\,g - 0.162\,g = \boxed{1.34\,g}$

(D.) IF % RECOVERY IS 60%, HOW MUCH RECRYSTALLIZED acetanilide in g DID YOU GET?

$\frac{60}{100} \times 1.34\,g = \boxed{0.804\,g}$

(E.) How much PHENACETIN can STAY IN THE FILTRATE [same assumption as in B]? IF THE 1.50 g CRUDE HAD 0.20g PHENACETIN, WOULD THE RECRYSTALLIZED BE PURE? → cold

$30\,mL \times \frac{0.076\,g\ phena}{100\,mL} = 0.023\,g\ phena$

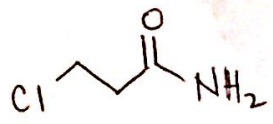
THE PRODUCT will contain $(0.20g - 0.023g) = 0.18\,g$ phena

(F.) PROVIDE A REMEDY BY showing calculations:

$0.20\,g \times \frac{100\,mL}{0.076\,g} = 290$ mL HOT H_2O needed to keep phenacetin in solution @ 0°C

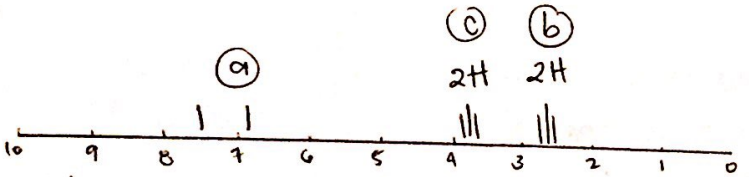
QUIZ C REVIEW

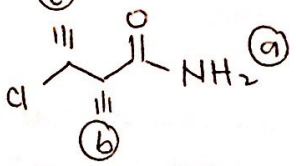
CORRECT:



IR INFO: 3350 / 3150] N-H stretch
2900 CH stretch
1660 - 1630 Amide R_{NR_2}
1450 CH_3 Bend

NMR INFO:





one side is carbon, & and the other is Cl.

↓

b/c both sides are carbon?

HDI = $C_cH_hN_nO_\emptyset X_x$

$HDI = 0.5[2c+2-h-x+n]$

then C_3H_6NOCl

$HDI = 0.5[2(3)+2-(6)-(1)+(1)]$

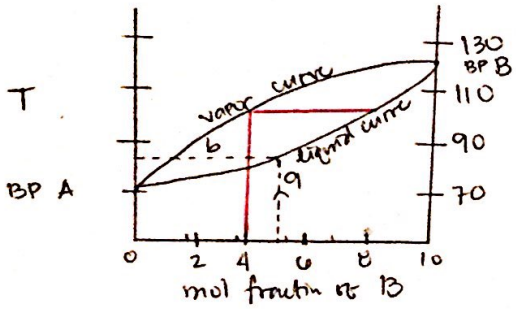
$HDI = 1$

EXPERIMENT D REVIEW

① IF A COMPOUND BOILS AT 100°C AT 760 mmHg, ITS BOILING POINT AT 725 mmHg WILL BE LOWER

② IN A DISTILLATION OF A MIXTURE OF TWO COMPONENTS, THE FIRST COMPOUND TO CONDENSE INTO THE RECEIVING FLASK WILL BE THE ONE WITH THE LOWER BOILING PT.

③ IF THE MOLE FRACTION OF B IN THE LIQUID IS 0.8, WHAT IS THE COMPOSITION OF B IN THE VAPOR ABOVE THE LIQUID? WHAT WILL BE THE READING ON THE THERMOMETER @ THIS POINT?



* THE VAPOR PHASE COMPOSITION IS ENRICHED IN THE AMOUNT OF A OVER THAT LIQ PHASE.
* THE MOL FRACTION OF B IN VAPOR PHASE IS 0.4 THE BP WILL BE ~100°C

④ IN A FRACTIONAL DISTILLATION, THE NUMBER OF THEORETICAL PLATES REFERS TO: THE EFFICIENCY OF A FRACTIONAL DISTILLATION COLUMN.

⑤ ON GAS CHROMATOGRAPHY, WHEN ANALYZING PENTANOL, HEXANOL, HEPTANOL, AND OCTANOL, THE ORDER OF INCREASING RETENTION TIME: !
PENTANOL < HEXANOL < HEPTANOL < OCTANOL
5 6 7 8