

14/8/17

Fischer Esterification

$$m(\text{PABA}) = 0.80 \text{ g}$$

$$V(\text{EtOH}) = 10 \text{ ml}$$

$$V(\text{H}_2\text{SO}_4) = 0.80 \text{ ml}$$

$$d(\text{EtOH}) = 0.789 \text{ g/ml}$$

slightly more \rightarrow instrumental error

Time	Observations
0-15	as soon as acid added, white precipitate formed immediately, liquid starts bubbling by bubbles <small>may have put less acid - slower</small>
15-25	- Dissolving \rightarrow white powder - bubbling - opaque white solution
25-39	- Still white powder / precipitate - few more drops EtOH 55 min taken off, still white **
During cooling	nothing much
upon diluting	no bubbles when enough Na_2CO_3 added crystals crashed out of solution

** Extra acid added $\sim 0.4 \text{ ml}$

\rightarrow same solution on hotplate

for an hour

- Extra Na_2CO_3 to neutralise acid $\sim 20 \text{ ml}$
- into ice water
- crystals appear

cos? ? over filter paper \rightarrow loss of product
- little bit of solid particulates in filtrate?

$$m(\text{Beaker}) = 39.12 \text{ g}$$

$$m(\text{B+Sample}) = 39.35 \text{ g}$$

$$m(\text{Sample}) = 0.23 \text{ g}$$

* not a little bit of sample left as residue



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After recryst some sample left in flask

↓ yield

↳ to increase scrape for eyes or use a liquid that won't dissolve it.

$$m(\text{vial lid}) = 2.25g$$

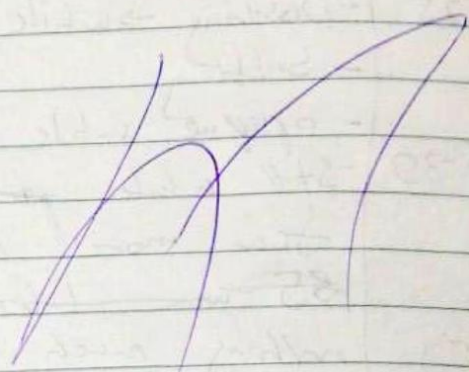
$$m(\text{Vial lid} + \text{Sample}) = 2.42g$$

$$m(\text{Sample}) = 0.17g$$

$$SF = 2.50cm$$

$$\text{TLC} \left\{ \begin{array}{l} x \text{ PABA} = 1.30cm \\ x \text{ Sample} = 2.00cm \end{array} \right.$$

$$R_F = \frac{x_{\text{thing}}}{x_{\text{Sample}}}$$



melting point analysis:

test 1 { first begin to melt: 81.4°C
fully melted (clear point): $81.4^{\circ}\text{C} - 96^{\circ}\text{C}$

test 2 { first: 82.0
clear point: 84.6

Discussion:

relate results to aim (1)

yields & alt pathways

MP analysis & TLC results, purity

} (5)

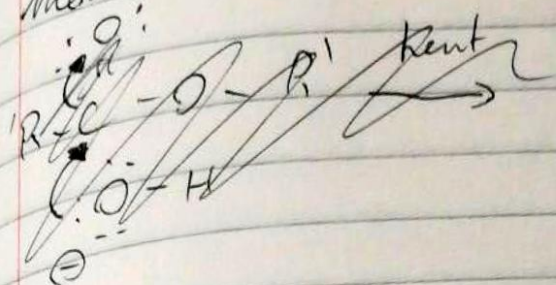
any other factors? (2)

↳ hot plate wasn't working

→ H_2SO_4 ?

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Mechanism:



look up online \rightarrow arrows, lone pairs, structure

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