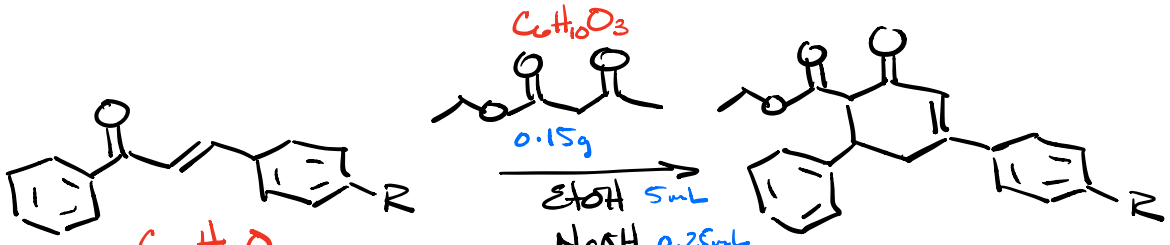
Reaction 38 for Michael-Aldol



$C_{15}H_{12}O$
Made in Rxn 37
~ 2 mmol

10mL RB flask

From 38 Instructions
0.24g of Chalcone

What mmol scale is reaction 38?

How many mmol Chalcone is 0.24g?

How many mmol Ethyl acetoacetate is 0.15g?

$C_{15}H_{12}O$ = 208.27 g/mole

$C_6H_{10}O_3$ = 130.14 g/mole

$$0.24\text{g Chalcone} \times \frac{1\text{ mole Chal}}{208.27\text{g Chal}} \times \frac{1000\text{ mmol Chal}}{1\text{ mole Chal}} = 1.2\text{ mmol Chalcone}$$

$$0.15\text{g Ethylacetoacetate} \times \frac{1\text{ mole}}{130.14\text{g}} \times \frac{1000\text{ mmol}}{1\text{ mole}} = 1.2\text{ mmol Ethyl aceto-acetate}$$

1:1 Ratio @ 1.2 mmol

$\frac{2.0\ \text{mmol}}{1.2\ \text{mmol}}$ ← Desired Scale / ← Existing Scale = Scale factor = 1.67

multiply all values by 1.67 to make a 2 mmol Reaction

Assume d ~ 1

Chalcone $0.24\,g \times 1.67 = 0.40\,g$ = 0.40 mL

Ethylacetoacetate $0.15 \times 1.67 = 0.25\,g \sim 0.25\,mL$

EtOH $5.0\,mL \times 1.67 = 8.35\,mL$

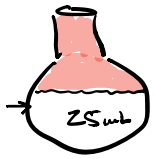
NaOH $0.25\,mL \times 1.67 = 0.42\,mL$

Total Rxn Volume = 9.42 mL

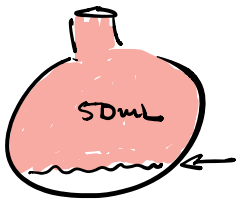
Work up + 2mL DI H_2O × 1.67 = 3.67 mL

Total Volume 13.09 mL

5 mL
10 mL
25 mL ⇐
50 mL
100 mL
250 mL



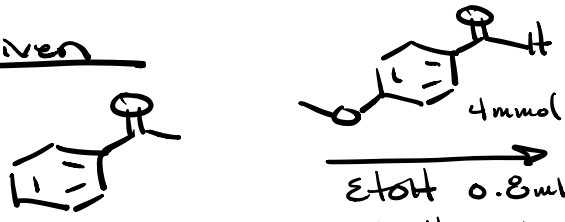
½ full ±10%



From Last Class, Calculations for Exp 37

$C_8H_8O_2$ = 136.15 g/mol

Given



0.004 mol × $\frac{136.15\ g}{1\ mol}$ = 0.545 g

~ 0.50 mL

4 mmol × 1.03

= 4 × 0.12 mL

= 0.48 mL

EtOH 0.8 mL × 4 = 3.2 mL

NaOH 0.10 mL × 4 = 0.4 mL

+ 4 × 2 mL DI workup = 8 mL

Total Volume = 0.48 + 0.5 + 3.2 + 0.4 + 8

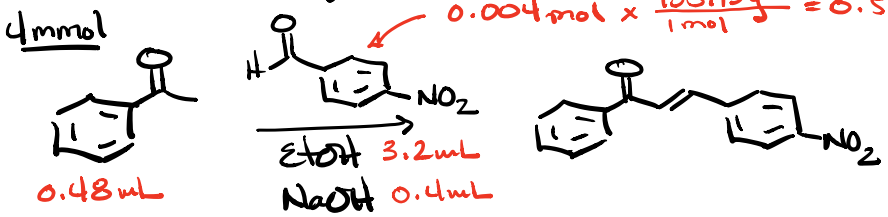
= 12.58 mL

⇒ RB flask size Required 25 mL

Example

⇒ Assigned 4-Nitro derivative

4 mmol



0.004 mol × $\frac{135.13\ g}{1\ mol}$ = 0.54 g

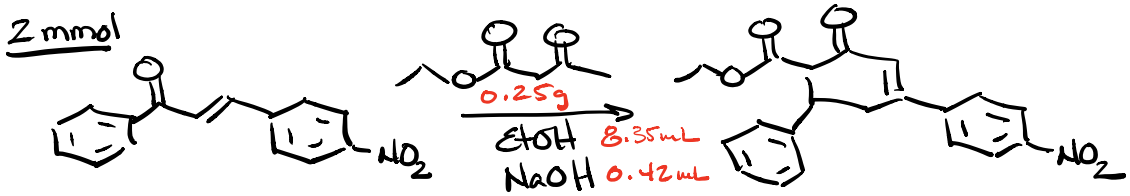
EtOH 3.2 mL

NaOH 0.4 mL

0.48 mL

0.49 g

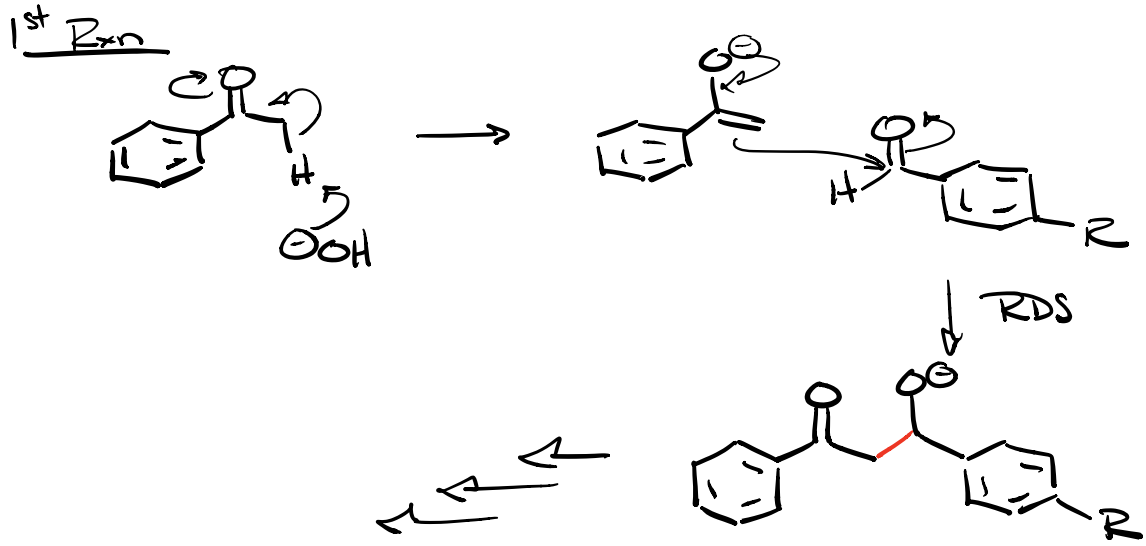
2 mmol

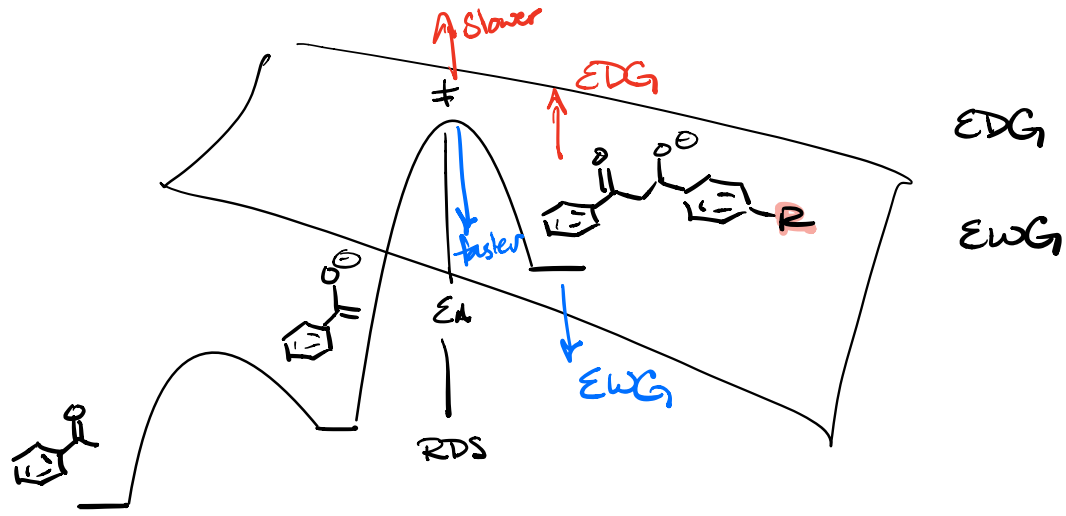


EtOH 8.35 mL

NaOH 0.42 mL

0.002 mol × $\frac{253.26\ g}{1\ mol}$ = 0.51 g





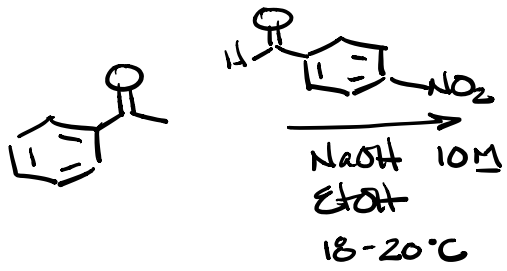
R Groups in Experiment

Evaluate by Induction

- $-CH_3$
- $-OCH_3$
- $-Cl$
- $-NO_2$

original
$-H$

Slow
$-H$
$-CH_3$ Strongest EDG
$-Cl$
$-OCH_3$
EWG
fast
$-NO_2$ Strongest EWG



Ideas to Slow Rxn down

→ Cool Rxn down ⇒ Run @ 0°C

→ Decrease M NaOH

→ Less NaOH

} 2 drops of 2.2 M

→ Possibly reduce Conc. of Reaction w/ more Solven

→ order of addition