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CHEM 345
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Solventless Aldol Reaction

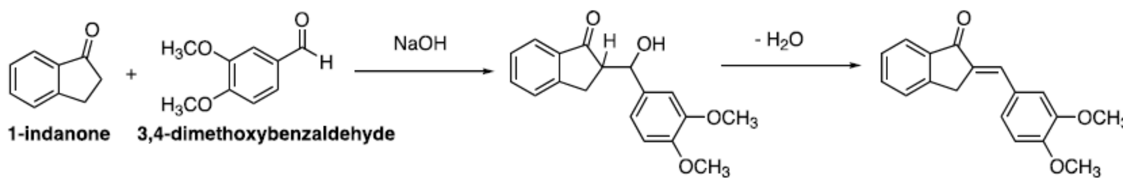
Reference: Handout; Chemistry lessons: carbonyl chemistry, aldol reaction, melting points of solids, eutectic mixtures; Green lessons: solventless reaction, atom economy, catalysis; Zubrick, Ch. 12

Purpose: To perform the base-catalyzed aldol condensation of 1-indanone and 3,4 - dimethoxybenzaldehyde

Table of Reagents:

Compounds	MW	Amount	BP (°C)	MP (°C)	Density
3,4 - dimethoxybenzaldehyde	166.176 g/mol	0.25 g	281 °C	41.5 °C	1.114 g/cm^3
1 – indanone	132.16 g/mol	0.2 g	244 °C	38.5 °C	1.103 g/cm^3
NaOH	39.997 g/mol	0.05 g	1388 °C	323 °C	2.13 g/cm^3
HCl	36.450 g/mol	2 mL	-85.05 °C	-114.2 °C	1.19 g/cm^3

Balanced Chemical Equation:



Safety:

- NaOH → caustic; Handle with care

Experimental Procedures	Data & Observations
1. Tare small beaker or test tube	*Mass of Test Tube:* 16.931 g
2. Obtain 0.25 g 3,4 – dimethoxybenzaldehyde and 0.2 g 1-indanone in test tube; Stir with spatula until brown	*Mass of 3,4 – dimethoxybenzaldehyde:* 0.251g *Mass of 1-indanone:* 0.2075 g
3. Add 0.05 g of grounded NaOH to reaction mixture until becomes solid; Ground NaOH	*Mass of NaOH:* 0.052 g
4. Stand for 15 min; Add 2 mL of HCl • Check pH; make sure it is acidic (litmus paper will turn red) • Scrape well to dislodge product from walls of beaker	

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5. Isolate solid with vacuum filtration and dry • Record the crude product mass	
6. Recrystallize product from 90% EtOH/10% water • Rinse any reaming product from test tube • Use minimum solvent	
7. Determine mass and melting point of recrystallized product	*Mass of Product:* 0.394 g *True Melting Point:* 178 – 181 °C *Product's Melting Point:* 152 °C

Post-lab Questions:

1. a) Our crude product was yellowish orangish. The true boiling point range of our product is between 178 – 181 °C, however, our product melted at 152 °C meaning that it experienced melting point depression and is not pure.

We collected 0.394 g of the product as well as the color of our product was yellowish-orangish.

$$\%\,Yield = \frac{Product\ Obtained}{Theoretical\ Yield} \times 100$$

Obtained: 0.394 g *Color and State:* Yellowish-Orangish

- First, we must obtain the theoretical value of our crude product in grams:

$$0.2075\ g\ 1indanone \times \frac{1\ mol}{132.16\ g} = 0.00157\ mol$$

Limiting Reagent

$$0.251\ g\ 3{,}4dimethoxybenzaldehyde \times \frac{1\ mol}{166.176\ g} = 0.00151\ mol$$

$$0.00151\ mol \times \frac{280.318\ g}{1\ mol} = 0.423\ g\ Product$$

- Next, we must use the equation above to get our % Yield:

$$\%\,Yield = \frac{0.394\ g}{0.423\ g} \times 100 = 93.14\%\ yield$$

- *We received a 93.14% yield for our product*

b) We were instructed not to recrystallize our product, and in regards we were only able to get our crude product's melting point, which was 152 °C.

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c) Our crude product melted at 152 °C, which concludes that it contains impurites as it has experienced melting point depression. The true melting point range of the pure product was recorded to be between 178 – 181 °C, whereas our product melted below the range. On the same note, this proves that our product's crystalline structure was disrupted by impuritues, which lowered our thought to be pure product's melting point. However, we never recrystallized our product which might have resulted in this discrepency in our melting point.

d) We never recrystallized our crude product, thus we can not provide a purified yield. However, we could say that we did receive a high yield for our crude product, which might have been a mixture of the pure product and our reagents.

2. <u>**Atom Economy**</u>

$$Atom\ Economy = \frac{MW\ of\ products}{MW\ of\ reactants} \times 100$$

$$Atom\ Economy = \frac{280.318\ g}{(132.16+166.176)} \times 100 = \ \ 93.96\%$$

<u>**E-Factor**</u>

$$E_{Factor} = \frac{Mass\ of\ Waste}{Mass\ of\ Product} = \frac{[(0.251\ g+0.2075\ g+0.052\ g)-0.394\ g]}{0.394\ g} = 0.296$$

3. The two effects impurities have on the melting point of a solid is that it <u>lowers the melting point</u> of the solid as well as it <u>broadens the melting point range</u>.

4. In order to identify the unknowns, you must first take the authentic sample of the pure solid 2 and mix the sample with any one of the unknowns. Next, you will take the melting point of the mixture, and depending on the result you will be able to determine the identity of the two samples. Going back to the mixture, if the melting point range of the mixture turns out to be between 102-104 °C, then the solid is the pure sample 2. Otherwise, if it experience melting point depression, in which the melting point is lower than 102 °C, then the unknown must have been sample 3.

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5. Aldol Condensation