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Aldol Condensation

React two equivalence of benzaldehyde with acetone in the presence of sodium hydroxide to form dibenzalacetone via aldol condensation. Isolate the product with recrystallization and vacuum filtration. Categorize the product with IR and H-NMR.

Referances

(1) Kateley, L. J., Guide for Organic Chemistry Laboratory, Seventeenth edition, Lake Forest College, 2011

(2) Sigma Aldrich. Merck 2025, 1

Reagent Table

Reagent	**FW** ($\frac{g}{mol}$)	**Density** ($\frac{g}{mL}$)	**mmol**	**Conc.** ($\frac{mg}{mL}$)	**Equiv.**	**Mass** (mg)	**Volume** (μL)
Benzaldehyde	106.12	1.043	2.01	NA	2.0	213	204
Acetone	58.08	NA	1.00	40	1.0	58.1	1452
Dibenzalacetone	234.30	NA	(1.0)	NA	(1.0)	(234)	NA

Calculations

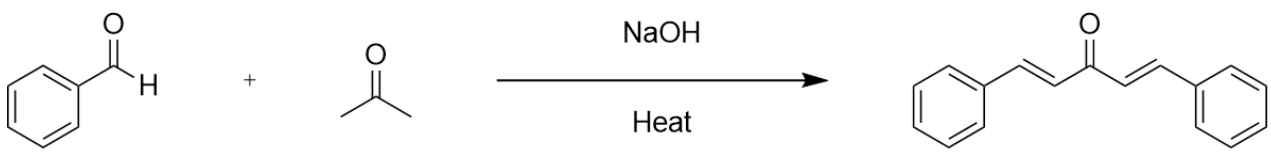
Benzaldehyde: $204\mu L(\frac{1.043mg}{1\mu L}) = 213mg(\frac{1mmol}{106.12mg}) = 2.01mmol$

Acetone: $1452\mu L(\frac{1*10^{-3}mL}{1\mu L})(\frac{40mg}{1mL}) = 58.1mg(\frac{1mmol}{58.08mg}) = 1.00mmol$

Dibenzalacetone: $1.0mmol(\frac{234.3mg}{1mmol}) = 234.3mg$

Percent Yeild: $\frac{0.221mg}{0.234.3mg} = 94.3\ \%$

Net Reaction



Experimental

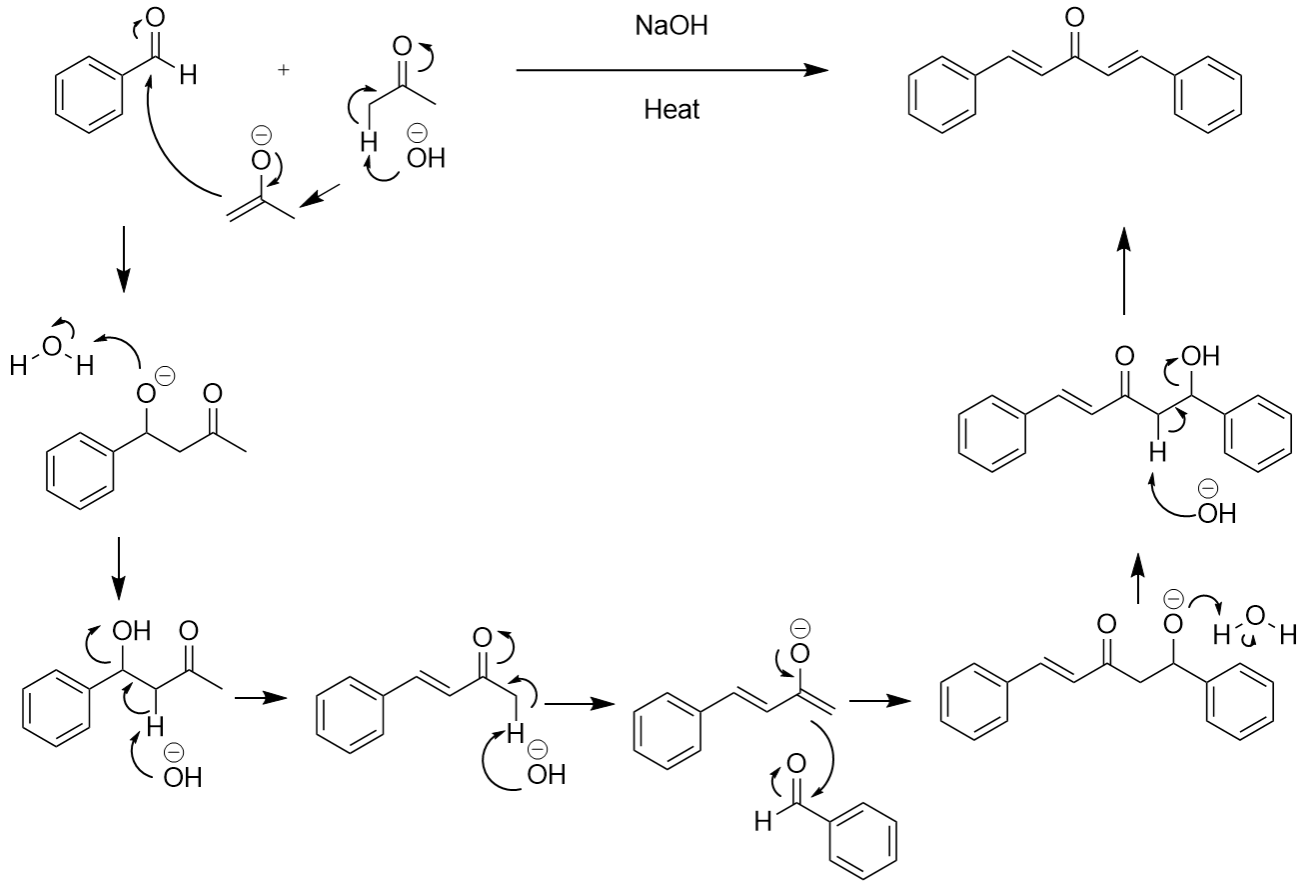
1mLmL of 6M NaOH and 1mL of water were added to a centrifuge tube. --μL (2.01mmol) of cherry smelling benzaldehyde were added. 1452 μL (1.00mmol) of acetone soln was added.

The reaction tube was capped with a rubber septum and shaken periodically for around 15min. As soln reacted, the reaction tube warmed and soln shifted from clear to cloudy orange to clouldy bright yellow.

The precipitate was isolated via vacuum filtration and washed 3 times with a total of 3mL of water, 1mL and the existing filtrate per wash. Product was trasfered to beaker. Around 4mL of boiling 95% ethanol were added to help product disolve. Ethanol rapidly cooled so beaker was stirred on hot plate. As product dissolved soln turned orange. A few drops of DI water was added to help the product crash out, forming a yellow solid.

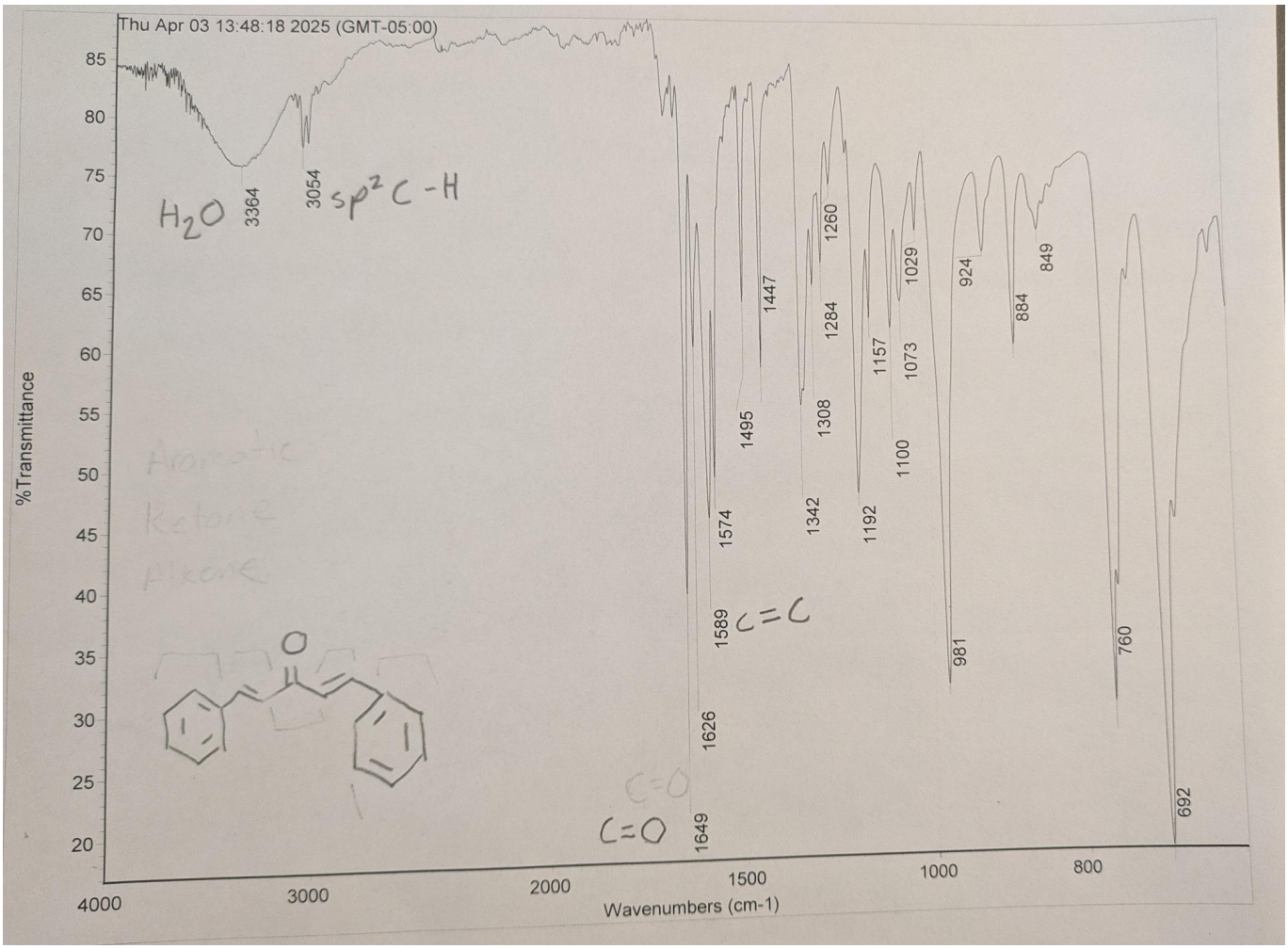
Product was isolated with vacuum filtration. The product was dried with filter paper and a % yeild of --% was calculated. Mass of container: 19.535g, Mass of container with product: 19.756g

Mechanism



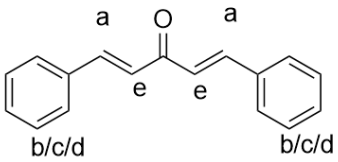
Spectra

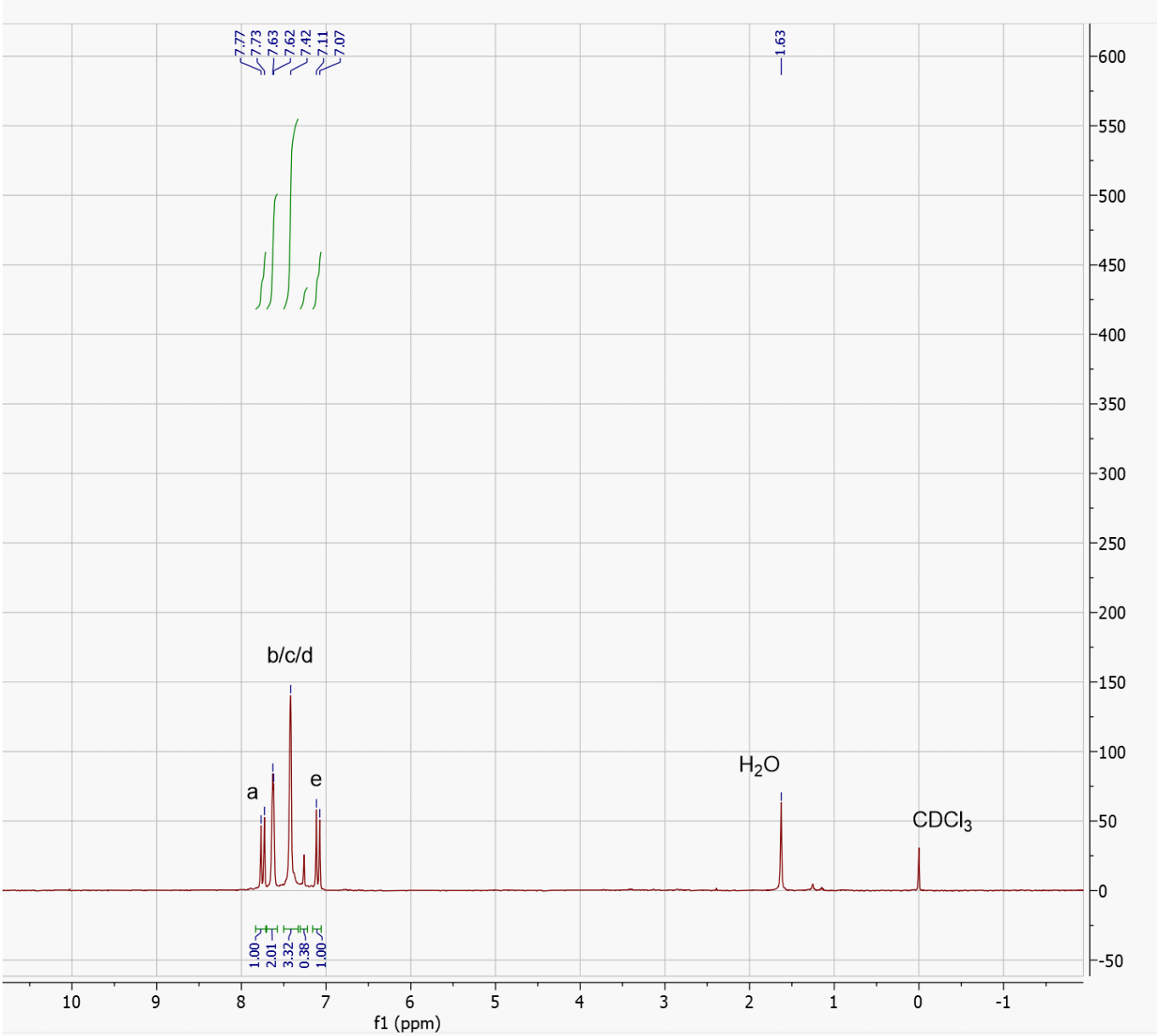
IR



NMR

H-NMR MNove File (I don't think this link actually works)

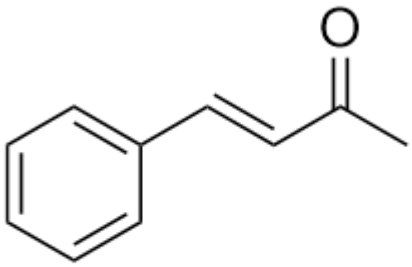




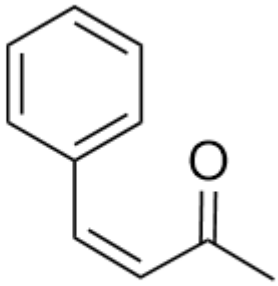
Questions

1. Draw structures with correct bond angles for cis,cis- and trans,trans-stereoisomers of dibenzalacetone and for benzalacetone.

Benzalacetone Stereoisomers:

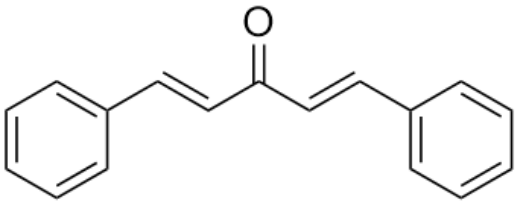


Trans

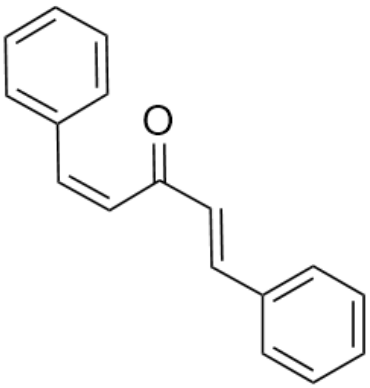


Cis

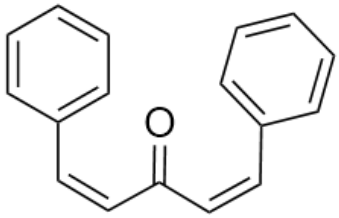
Dibenzalacetone Stereoisomers:



Trans, Trans



Cis, Trans



Cis, Cis

2. Check to see if there is any evidence for methyl protons on the carbonyl carbon on the NMR Spectra. Which of the structures in 1 would have this signal?

The coupling constant changes with each stereoisomer. The trans, trans stereoisomer has a similar methyl proton as the product.

3. Which stereoisomer of dibenzalacetone have you synthesized? Cite your evidence, which must include the vinyl proton coupling constant and calculations. If you need a refresher: See Mohrig, et. al., Section 22.9, pp. 377–382.

Coupling constant :

$(7.77 - 7.73) * 400 = 16Hz$

$(7.11 - 7.07) * 400 = 16Hz$

Instrument = 400 MHz Cis: 7-12 Trans: 12-18 - further apart

4. A typical ketone has a IR stretch around 1715 cm-1. Where is your carbonyl stretch? Explain deviations from the norm.

The carbonyl is shifted down because it is conjugated with the alkenes and the aromatic rings.

5. Why is intermediate bearing a hydroxyl group not isolated?

The condensation can happen at a lower temperature, removing the hydroxyl group, because it has extended conjugation. This lowers the activation energy since the enolate and produced alkene can resonate with the aromatic rings.

Conclusion

The final percent yeild was 94.3%. The H-NMR and IR both show evidance of water, which slightly inflated the percent yeild. Some product was left in glassware and in the Hirsch funnel after recrystallization.

Since the trans stereoisomer is thermodynamically favored, we would predict that the product is the trans, trans, dibenzalacetone. This is supported by the H-NMR. The larger coupling constant of 16Hz supports that the product is trans as the cis isomer would have a lower coupling constant.

Note: I tried a different formatting thing for this report, please let me know if it looks alright.