



Organic chemistry experiment

The synthesis of DBA

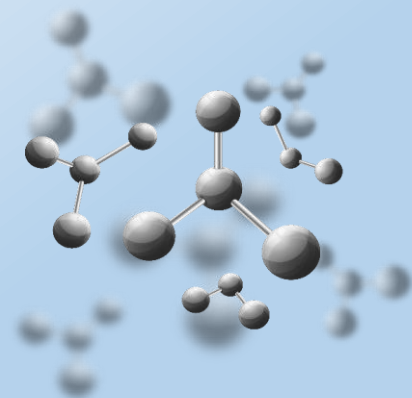
Experimental center of chemical education
Fudan University





Target

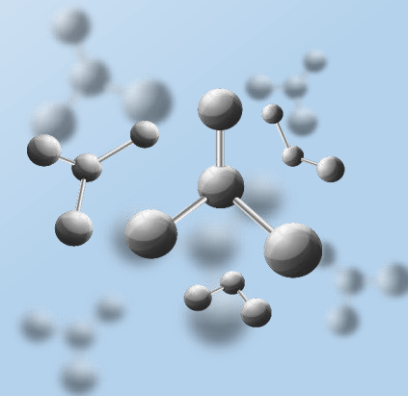
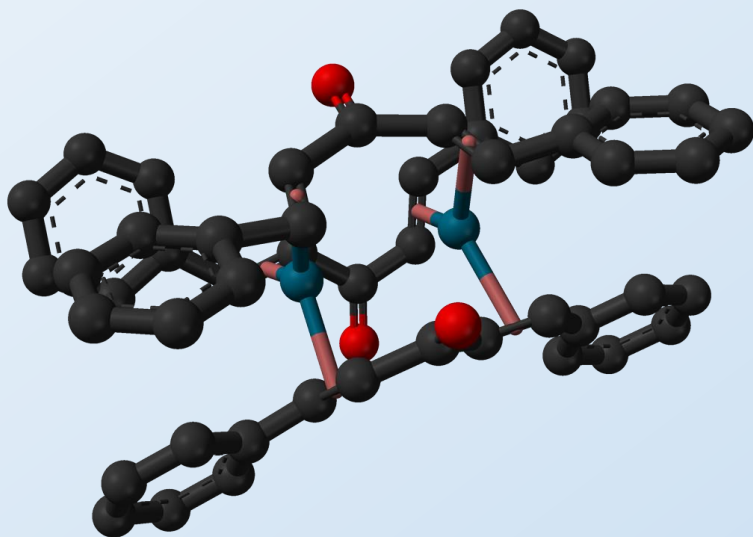
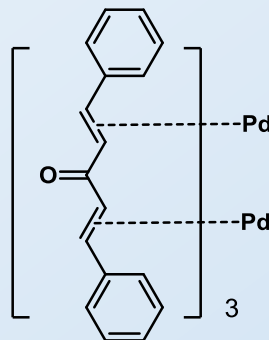
1. Master the principle of aldol reaction.
2. Obtain the product through controlling the ratio of starting materials.





Background

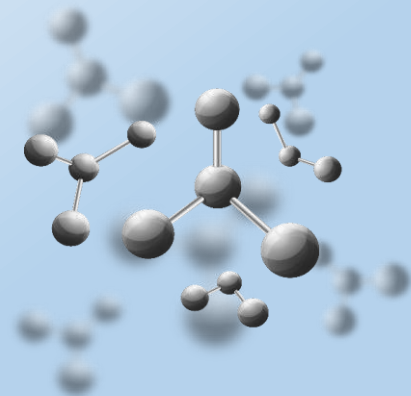
$\text{Pd}_2(\text{dba})_3$ is a good source of Pd(0) which is widely used as a catalyst in cross coupling.





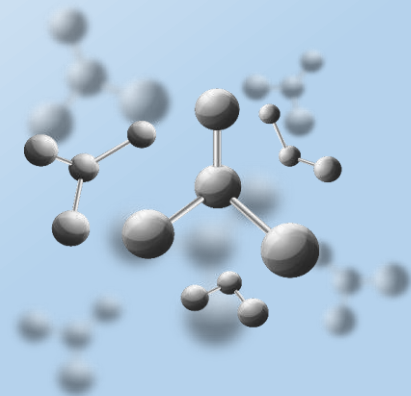
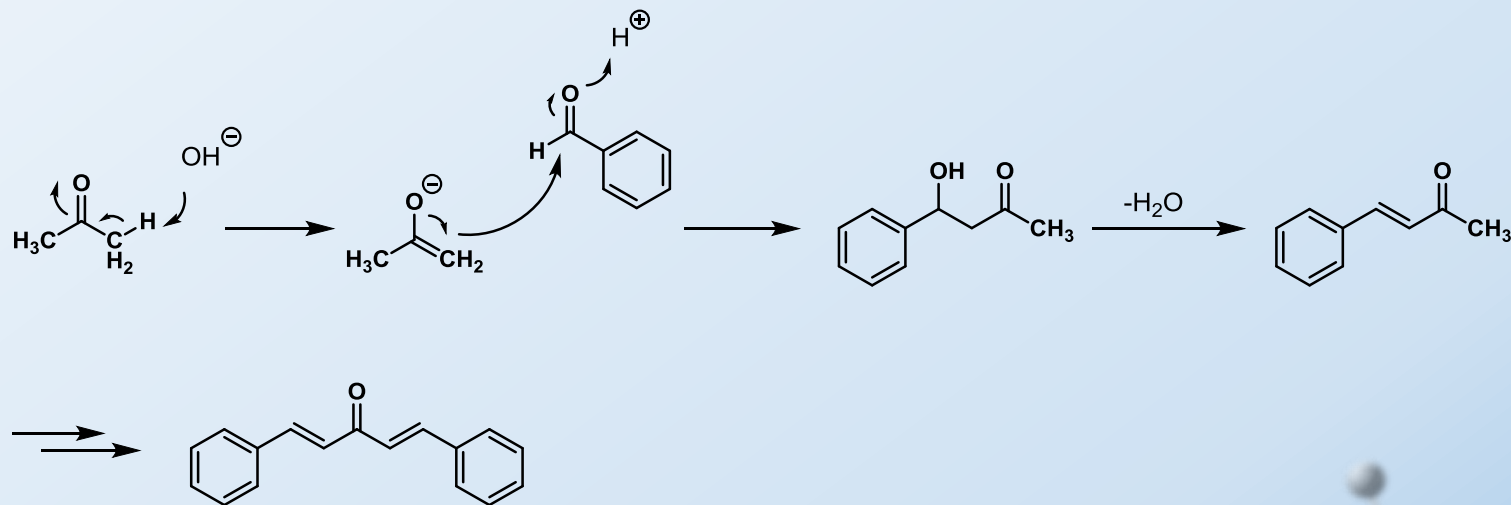
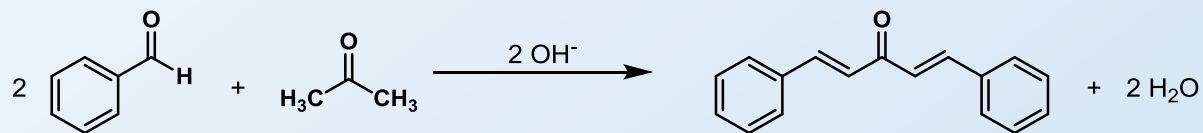
Experimental principle

1. Two molecules of carbonyl compounds with α -H, can condense to form β -hydroxy aldehyde(ketone). While raising the reaction temperature, will form further form α,β -unsaturated aldehyde (ketone).
2. A non- α -H aromatic aldehyde can condense with carbonyl compounds containing α -H to form α,β -unsaturated aldehyde (ketone), it is so-called Claisen-Schmidt reaction.





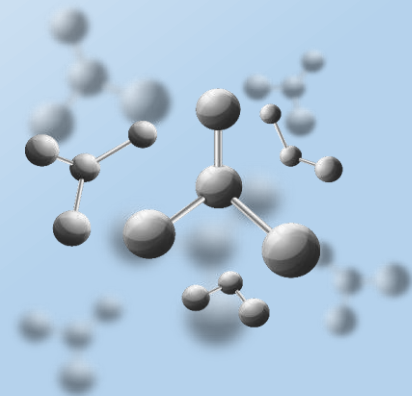
Experimental principle





Reagents

	M.w.	m.p.	b.p.	d.
Benzaldehyde	106.12	-26	179	1.041
Acetone	58.08	-95	56	1.359
EtOH	46.07	-114	78	0.789
DBA	234.3	113	130(2.7Pa)	

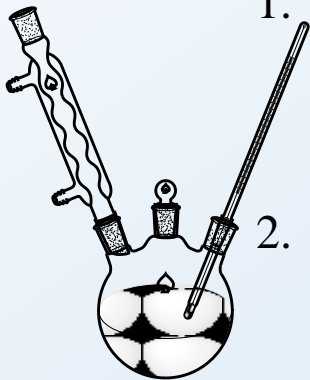




The synthesis of DBA

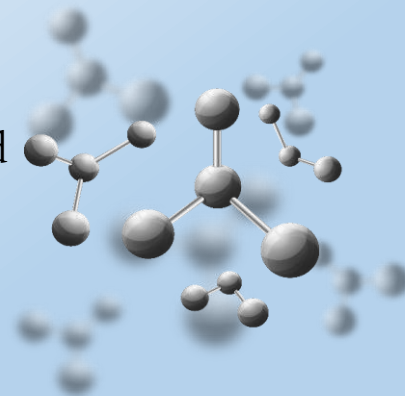
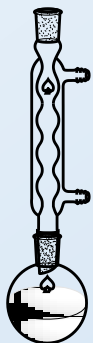
Synthesis

1. 2.0 g NaOH, 20 mL H₂O, cool down 16 mL EtOH added to 100 mL three-neck flask
water bath
2. 2.1 g PhCHO, 0.58 g Acetone mixed slowly added half to flask
control the temperature
3. 15 min added the rest, EtOH washed
4. 30 min later suction filtration, washed by H₂O, dry, weigh



Recrystallization

1. Crude product is added to 50 mL round-bottomed flask
EtOH is added (about 5 mL/g)
2. Water bath, reflux, EtOH is added slowly until totally dissolved
3. Cool down to r.t., followed by icy water bath
4. Suction filtration, washed by H₂O, dry, weigh





Attention

1. Stir should be kept during reaction to ensure sufficient reaction.
2. PhCHO and acetone should be weighed exactly.

