Reaction of A Ketone and Grignard Reagent, Ethyl Magnesium Bromide

Ilana Berlin

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Purpose:

React a ketone, 2-butanone, with a Grignard reagent, ethyl magnesium bromide, to form an alkoxide, 3-methyl-3-pentanol. Isolate product using a separatory funnel and drying agents and categorize it using H NMR and IR spectroscopy.

Reagent Table and Calculations:

Ketones/ Alcohols	MW (mg/mmol)	Density (g/mL)	Equiv.	BP (°C)	mmol	Mass (mg)	Volume (µL)
EtMgBr (3.0M in Et_2O)	n/a	n/a	1.2	n/a	4.82	n/a	1606
2-butanone	72.11	0.805	1.00	80	4.02	289	360
3-methyl-3-pentanol (theoretical)	116.20	0.8237	1.00	153	4.02	467	385

3-methyl-3-pentanol (actual)	116.20	0.8237	0.600	153	2.41	281	231

EtMgBr: 4.82mmol($1x10^{-3}$mol/1mmol)(1L/3.0mol)(1μL/$1x10^{-6}$L) = 1606μL

2-butanone: 360μL($1x10^{-6}$L/1μL)(1mL/$1x10^{-3}$L)(0.805g/1mL)(1mg/$1x10^{-3}$g) = 289mg

289mg(1mg/72.11mmol) = 4.02mmol

3-methyl-3-pentanol (theoretical): 4.02mmol(116.20mmol/1mg) = 467mg

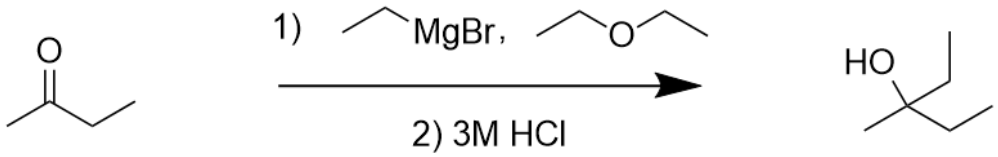
467mg($1x10^{-3}$g/1mg)(0.8237mL/1g)(1μL/$1x10^{-3}$mL) = 385μL

3-methyl-3-pentanol (actual): 281mg(1mmol/116.20mg) = 2.41mmol

281mg($1x10^{-3}$g/1mg)(0.8237mL/1g)(1μL/$1x10^{-3}$mL) = 231μL

Percent Yield: 2.41mmol/4.02mmol x 100 = 60.0%

Net Reaction Equation: (Use Chemdraw)



References:

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

Experimental: (be concise and use abbreviations h, min, and soln)

Constructed Apparatus: 10mL round bottom flask with a stir bar was connected to an upturned distillation head fitted with a rubber septa. Distillation column was attached to the top of the distillation head and a small piece of cotton was stuffed in the end.

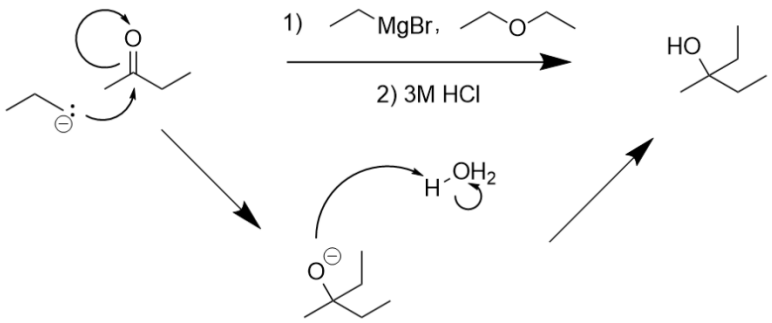
Alkoxide Synthesized: 630µL of 2-butanone and 870µL anhydrous diethyl ether added to a reaction tube that was then capped with a rubber septa. 1606µl of ethyl magnesium bromide was added to the round bottom flask. Ketone soln was slowly added via syringe over the course of 5 min. Another 500µL of diethyl ether was used to rinse reaction tube and added to the soln. Hot plate was turned on and soln was left to stir for approximately 25 min. Soln was removed from heat and placed in an ice bath.

Reaction Quenched with 3M HCl: 3mL of 6M HCl was added to the soln in the round bottom flask. Soln was transferred to 30mL separatory funnel. Round bottom flask was rinsed with 1mL of diethyl ether.

Product Isolated, Dried, and Analyzed: Water layer and ether layer were separated into 10mL flasks. 2mL of ether was added to the water layer. The water was removed and the ether was added to the other ether soln. The combined ether soln was washed with a 1.5mL 10% sodium bicarb soln. The soln was dried with anhydrous sodium bicarb for 5 min. The soln was transferred into a tared centrifuge tube and dried with a light stream of

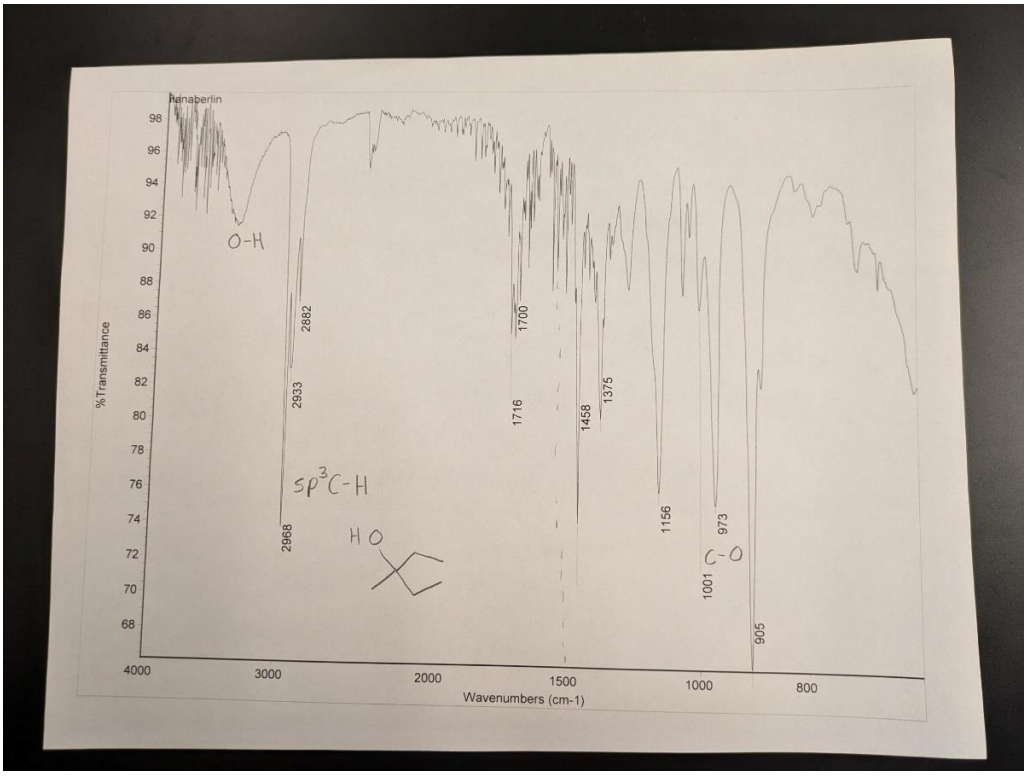
air. The dried product was weighed, percent yield was calculated, and H NMR and IR spectra were collected.

Mechanism: (Use chemdraw and net reaction equation)

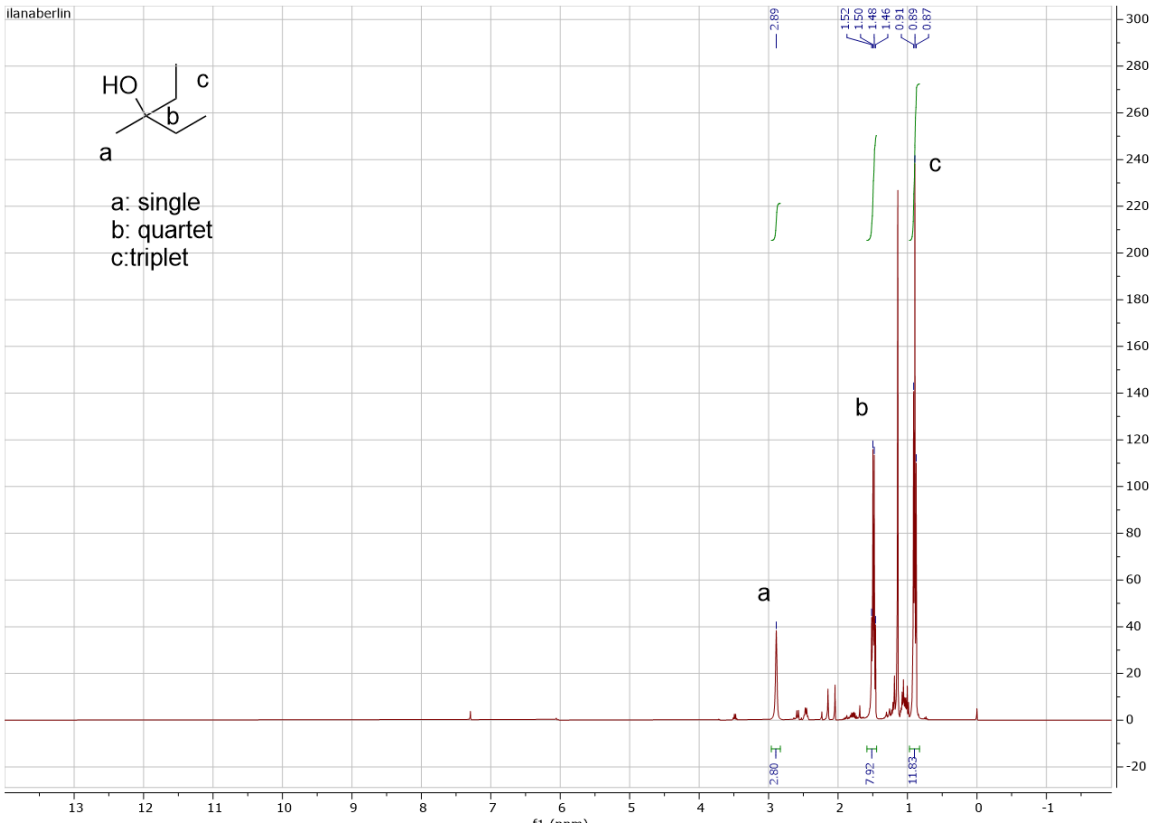


Annotated Spectra:

IR:



NMR:



Questions:

1. Why is it important to use dry glassware and anhydrous solvent in this reaction?

If water comes into contact with the Grignard reagent, it will react with them to form a salt rather than the ketone.

2. Why must an inert atmosphere be used to remove Grignard reagent from the reagent bottle?

Similar to the reason for dried glassware, if the Grignard reagent comes into contact with the air then it will reaction with the moisture in the air to form a salt.

3. Which layer was the ether in the separatory funnel (top or bottom)? Why?

The ether layer was the bottom layer of the separatory funnel as water is denser than ether.

Conslusion: (key findings, yield, and improvements):

The Grignard reagent is extremely reactive. Adding extra reagent helps improve the conditions of the experiment as some of it reacts with the water on the surface of the equipment and in the air. For this experiment to be done in better conditions the glassware would have to been heated and cooled and the reaction would take place in an inert atmosphere. For this reason, the percent yield is relatively low at 60.0%.