

Sodium Borohydride Reaction

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02/26/2025

React achiral compound, benzil, with sodium borohydride to form chiral compound, hydrobenzoin. Isolate product via vacuum filtration and classify product through IR and H-NMR. Determine isomer via melting point.

Reagent Table and Calculations:

reagents, solvent & product	FW (g/mol)	density, g/mL	mp °C	bp °C	equiv.	mmol	Mass mg
benzil	210.22	NA	94-95	NA	1.00	0.999	210
sodium borohydride	37.83	NA	> 300 (dec)	NA	1.10	1.1	42
95% ethanol (solvent)	46.07	0.789	NA	78	NA	NA	NA
hydrobenzoin	214.16	NA	146 148 137	NA	1.00	(0.999) 0.798	(214) 171

() – theoretical

Benzil: $210\text{mg} (1\text{mmol}/210.22\text{mg}) = 0.999\text{mmol}$

Sodium Borohydride: $42\text{mg} (1\text{mmol}/37.83\text{mg}) = 1.1\text{mmol}$

Hydrobenzoin: $0.999 \text{ mmol}(214.16\text{mg}/1.00\text{mmol}) = 214\text{mg}$

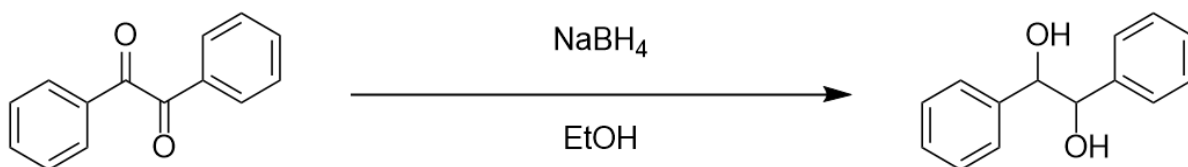
$171\text{mg} (1\text{mmol}/214.16\text{mg}) = 0.798\text{mmol}$

Empty Vial: 10.034g, Vial with Product: 10.205g

Product = $10.205\text{g} - 10.034\text{g} = 0.17\text{g} = 171\text{mg}$

Percent Yield: $171/214 * 100 = 80\%$

Net Reaction Equation: (Use Chemdraw)



References:

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

(2) Sigma Aldrich. Hydrobenzoin *Merck* 2025, 1

Apparatus:



Erlenmeyer Flask and hot plate

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

0.210g (0.999mmol) of benzil was added to an Erlenmeyer Flask. The solid yellow crystalline benzil was dissolved in 2mL of 95% ethanol. The hot plate was used to warm the soln to help solid dissolve. Soln was cooled to room temp with a warm water bath. Small amount of benzil precipitated out of the soln. Large spindle-like yellow crystals formed.

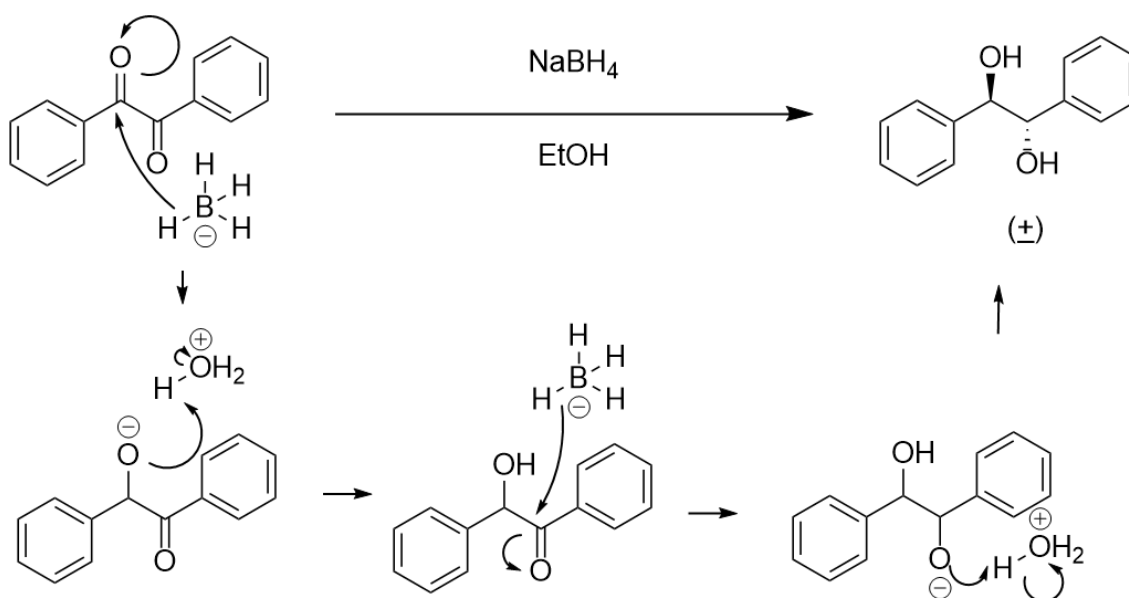
0.042g (1.1mmol) of NaBH_4 was added to the soln while the soln was gently stirred. White bubbles and color shift from yellow to clear provided evidence for a reaction. After 10min, 2.5 mL of DI water was added. The solution shifted from milky white to clear. The hot plate was turned up and the soln was boiled. Approximately 3.5 more mL of water was added but the soln did not turn cloudy. As the soln cooled to room temp product crashed out of the soln. The soln was further cooled in an ice bath until a white sludge formed, indicating that product precipitated out.

The product was isolated with vacuum filtration, cold water and filtrate were used to wash product. Product was dried with filter paper. Percent yield of 80% was calculated. IR spectra was collected. H-NMR spectra was collected. A drop of D₂O was added to NMR tube and spectra was rerun. Melting point of 130-134°C was observed, suggesting that the meso isomer was formed.

PRODUCT WAS STORED IN LABELED PETRI DISH IN DRAWER FOR USE IN NEXT LAB.

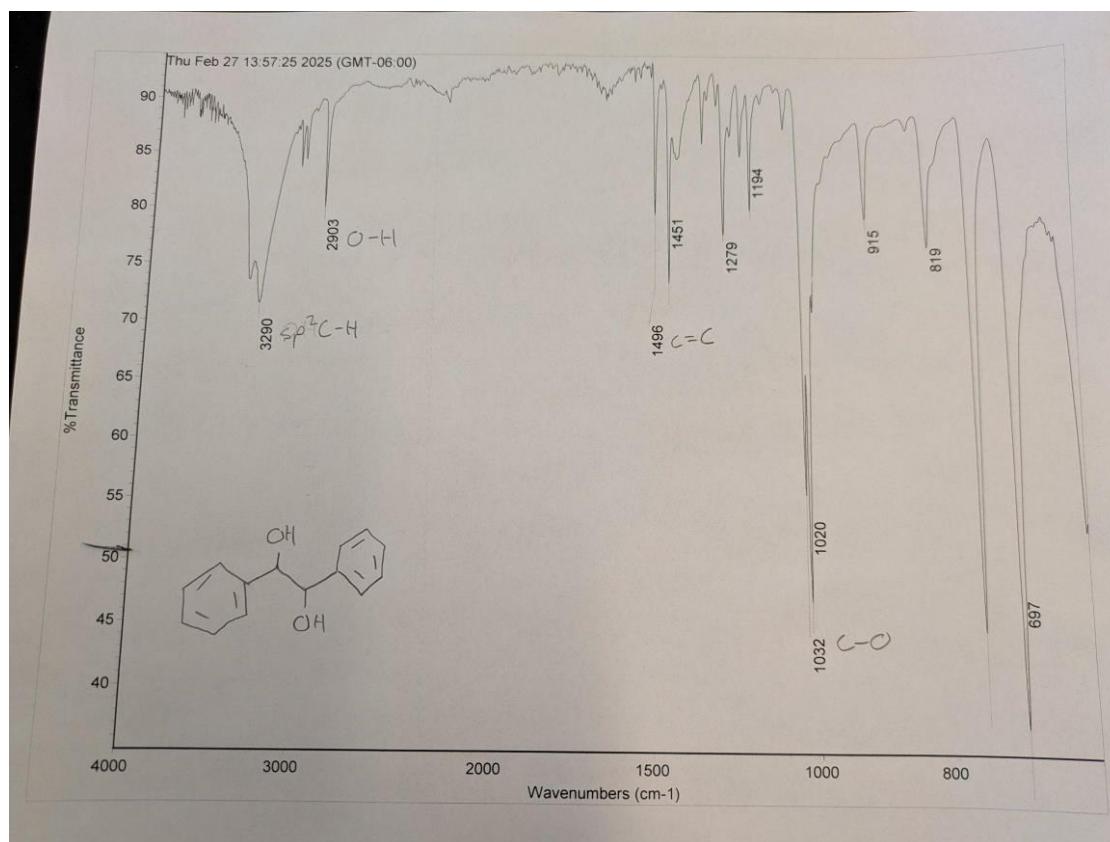
SAVE PRODUCT

Mechanism: (Use chemdraw and net reaction equation)

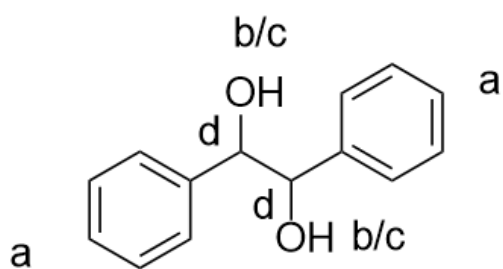
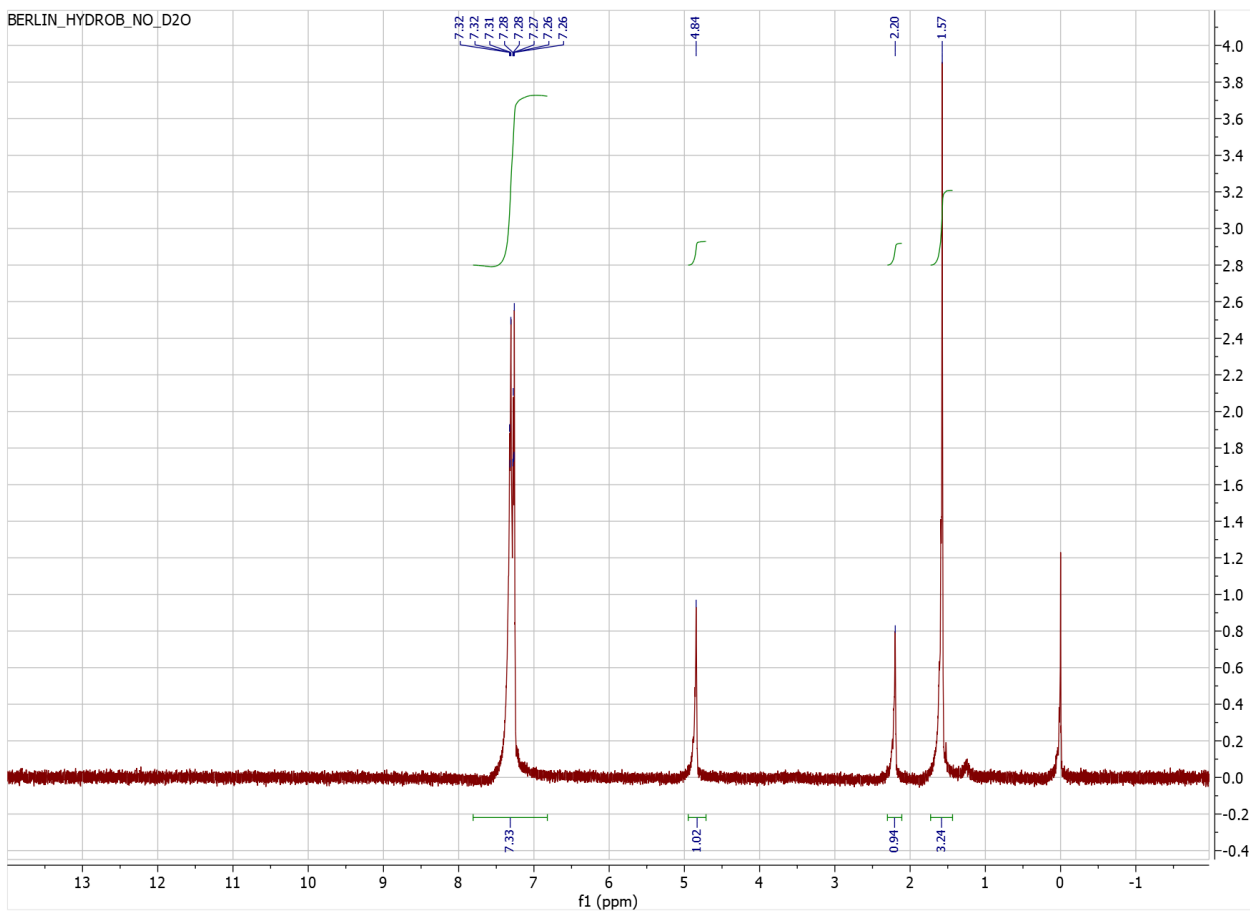


Spectra (is there starting material) :

IR:

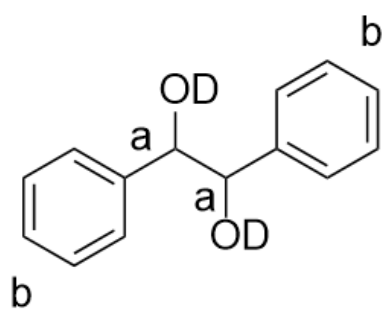
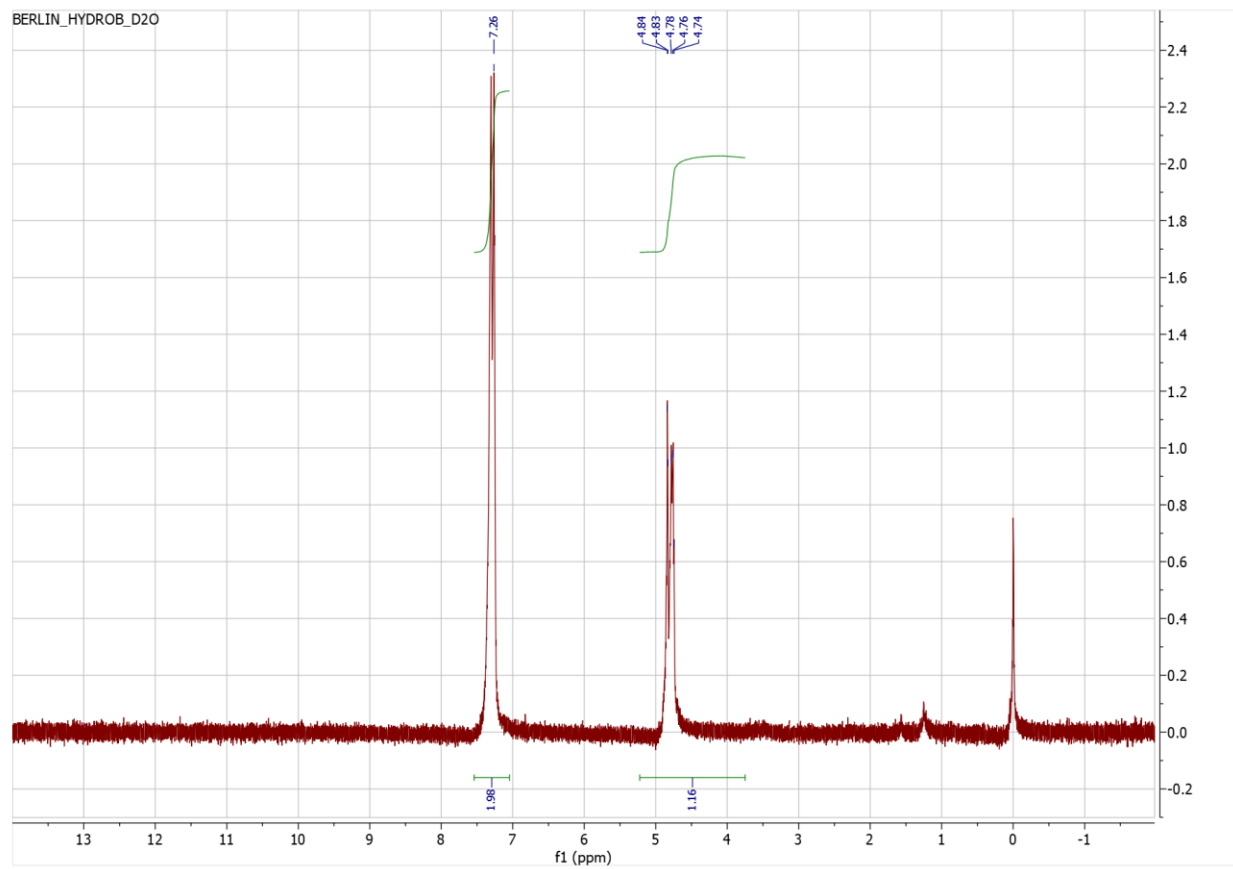


H-NMR:



a: multiplet: 10
 b/c: singlet: 2
 d: singlet: 2

H-NMR with D₂O: Missing OH singlets after D₂O addition indicated replaced.



a: singlet: 2
b: multiplet: 10

Questions:

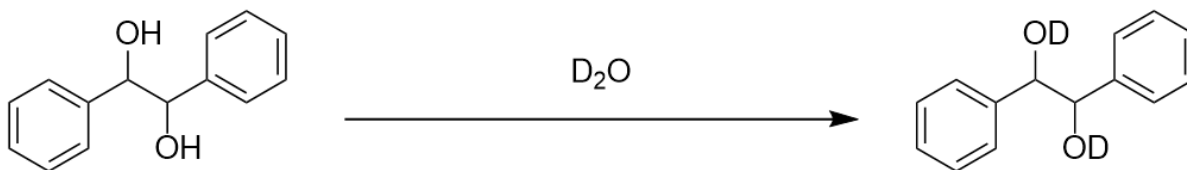
1. Which stereoisomer did you make and what evidence supports this conclusion?

The reaction product the meso isomer, this is supported by the melting point. The meso isomer has the lowest melting point, around 137°C, while the melting point of the R and S isomers are slightly higher, at 146°C and 148°C respectively. The melting point of my product was 130-134°C which is on the lower end. Melting point may have been further lowered by impurities.

2. What information did you get from the NMR spectrum from adding D₂O? a)

Draw the reaction equation for deuterium exchange.

Adding D₂O replaces the H in OH with D which removes that peak from the H-NMR. This allows us to determine which singlet is alcohol H and which is the lone H.



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

The final product is meso-hydrobenzoin, as indicated by the melting point.

The final percent yield was 80%. Possible product loss in the solution because it did not automatically crash out when hot water was added indicating that solution was not fully

saturated when water was added. Product was lost on the filtration funnel and on the filter paper when drying.

Dissolving and crashing the benzil back out help increase the surface area of the benzil by breaking apart large chunks which helps the reaction precede faster. Even so, giving a few more minutes for the reaction to go may have been beneficial. It also may have helped to let the solution boil for longer.