

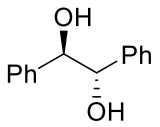
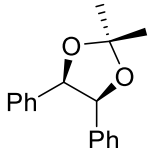
Dimethyl Acetonide Synthesis

Ilana Berlin

03/06/2024

Synthesize dimethyl acetonide from hydrobenzoin using FeCl_3 as a catalyst. Isolate product through separation and vacuum filtration. Characterize product with IR and H-NMR. Determine chirality of the starting material using characters displayed by the H-NMR of the final product.

Reagent Table and Calculations:

Reagents	FW (g/mol)	mmol	Equivalents	Mass (mg)
$\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (catalyst)	270.30	0.133 (0.119)	0.25	36 (32.2)
 Hydrobenzoin	214.26	0.476	1	102mg
 Dimethyl acetonide	254.33	0.0629 (0.476)	1 (theoretical)	16 (theoretical)

() Theoretical

$$102\text{mg}(1\text{mmol}/214.26\text{mg}) = 0.476\text{mmol}$$

$$0.476\text{mmol}(0.25\text{mmol}/1\text{mmol}) = 0.119\text{mmol}(270.30\text{mg}/1\text{mmol}) = 32.2\text{mg}$$

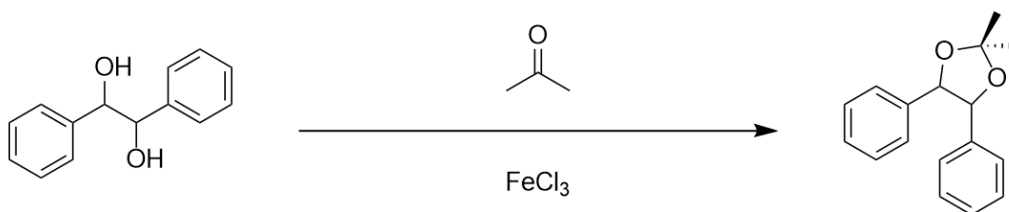
$$36\text{mg}(1\text{mmol}/270.30\text{mg}) = 0.133\text{mmol}$$

$0.476\text{mmol}(254.33\text{mg}/1\text{mmol}) = 121\text{mg}$

$8.464 - 8.448\text{g} = 0.016\text{g} = 16\text{mg}(1\text{mmol}/254.33\text{mg}) = 0.0629\text{mmol}$

$0.0629/0.476 = 13\% \text{ Yield}$

Net Reaction Equation: (Use Chemdraw)

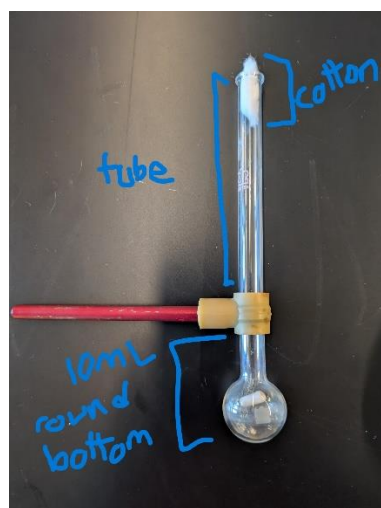


References:

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

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

Apparatus (Label parts):



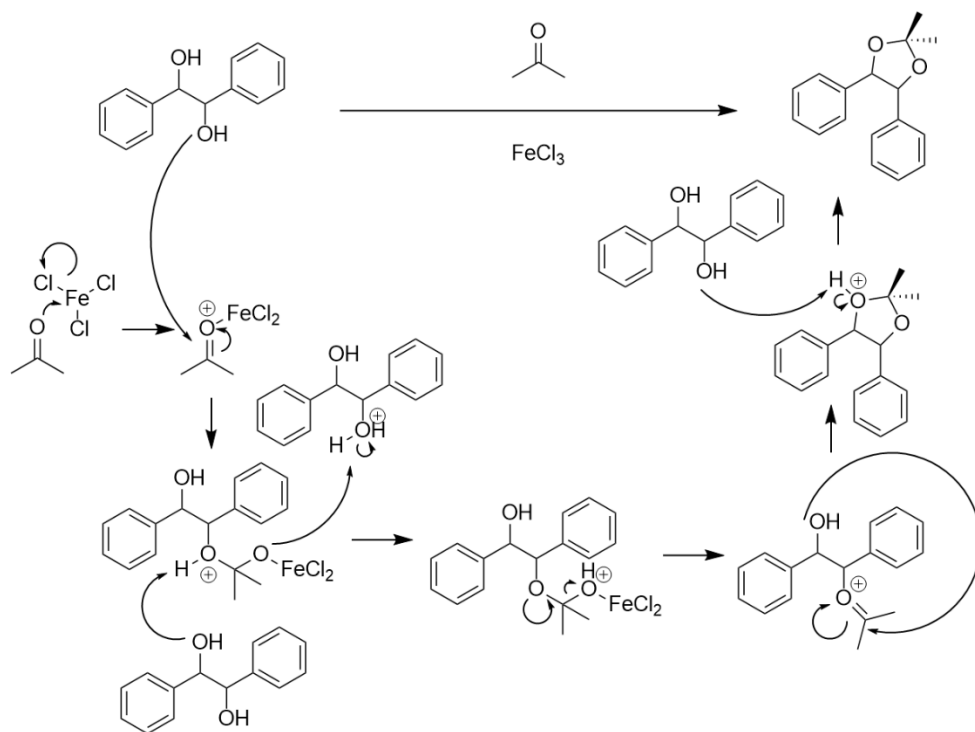
Experimental: (be concise and use abbreviations h, min, and soln):

0.102g (102mg) of hydrobenzoin was added to the round bottomed flask. 0.032g (32mg) of FeCl_3 was also added to the flask. A stir bar was added, and the flask was filled with 2.5mL of reaction grade acetone. The yellow Soln was heated above the BP of acetone (56°C) to around 70°C to reflux. Soln refluxed for approximately 20 min. As soln relaxed the color shifted to orange.

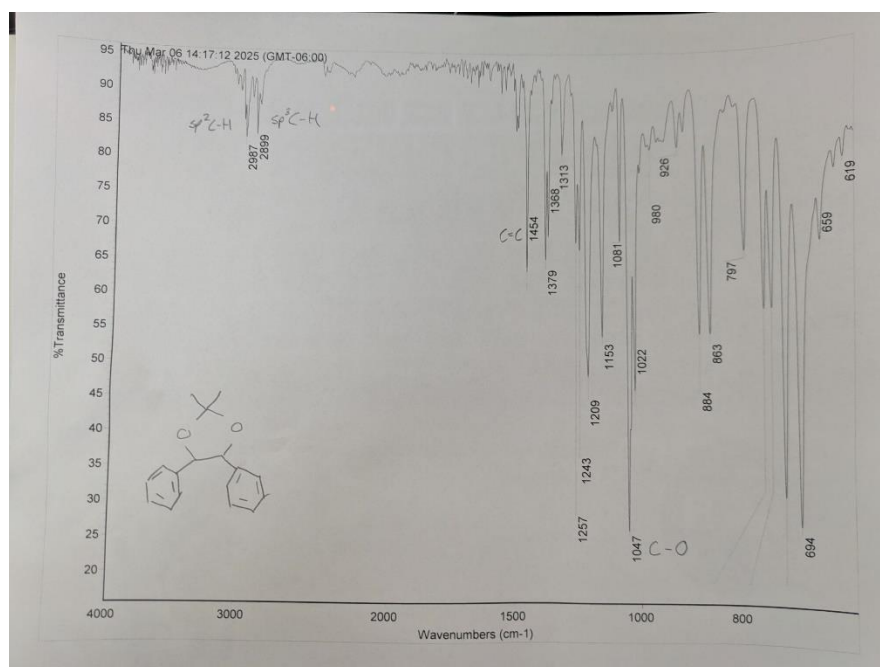
Soln was cooled to room temp and 3mL of 1M Potassium soln was used to wash. Soln was transferred to sep funnel. Bottom red aqueous layer and top yellow organic layer were separated. Aqueous layer was washed twice with 3mL of diethyl ether. Combined organic layer was washed with 3mL of saturated NaCl brine and dried with sodium sulfate (Na_2SO_4) for 15min. Soln was transferred into a centrifuge tube and dried with air and a warm water bath.

3 mL of pentane was added and resulting suspension was isolated though vacuum filtration. Tube and filter were washed with 3 more mL of pentane. Vacuum was run until all pentane had evaporated. Resulting oil was transferred into weighed (8.448g) vial. Percent yield of 13% was calculated and spectra were collected.

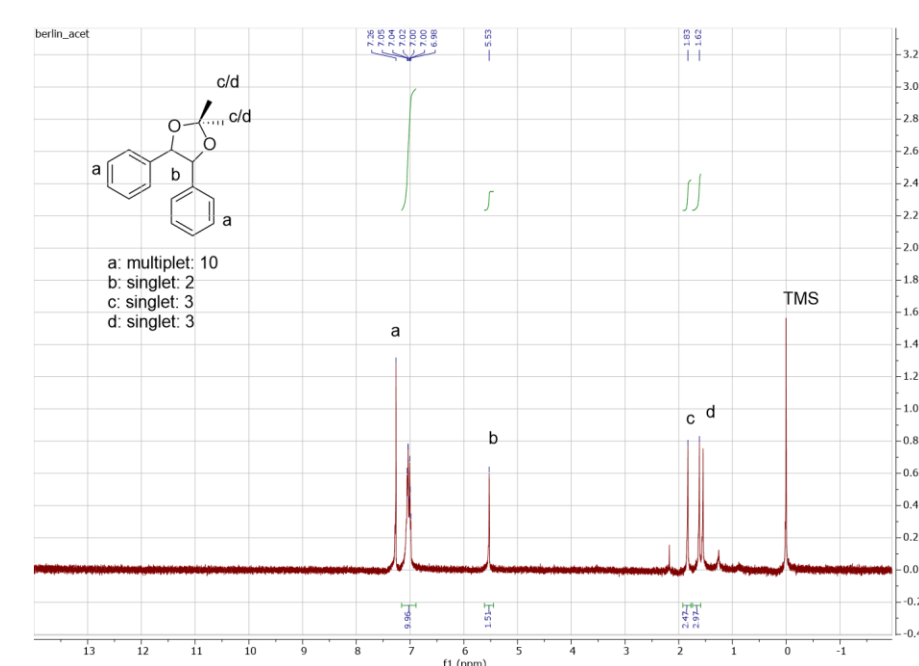
Mechanism: (Use chemdraw and net reaction equasion)



Spectra:



H-NMR:



Questions:

1. Explain the significance of the number of methyl signals observed in the proton NMR spectrum.

Since there are 2 methyl peaks it indicates that the methyl groups have slight differences in position. This shows that we started with meso-hydrobenzoin.

2. Explain the role of FeCl₃ in the reaction.

FeCl₃ acts as a catalytic acid. It accepts electrons from the carbonyl, giving the carbonyl a positive charge making it more reactive.

3. Calculate the number of equivalents of acetone relative to hydrobenzoin and explain the significance of this value for the success of this reaction

$$3\text{mL}(0.791\text{g}/1\text{mL}) = 2.37\text{g}(1\text{mol}/58.08\text{g})(1\text{mmol}/1\times 10^{-3}\text{mol}) = 40.9\text{mmol}$$

There is an approximately 85:1 ratio of acetone to hydrobenzoin in the original mixture. This helps ensure that all of the starting hydrobenzoin gets used up.

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

FeCl_3 can act as a Lewis Acid and create acidic conditions that allow for acetal formation.

In this reaction the resulting acetal can help us determine the chirality of the starting material.

The final percent yield was 13%. A lot of product was lost during separation. As the aqueous layer sat on the lab bench it separated into two distinct layers, indicating that there was still an organic layer containing product that had been previously mixed in. If there was more time I would have liked to take the remaining water layer and try to extract more product. If experiment were repeated, weigh the side arm flask so product does not have to be transferred to new container as well.