

JoVE: Science Education
Hydrogenation of Chalcone
--Manuscript Draft--

Manuscript Number:	10350
Full Title:	Hydrogenation of Chalcone
Article Type:	Manuscript
Section/Category:	Manuscript Submission
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Overview:

This experiment will demonstrate the hydrogenation of chalcone as an example of an alkene hydrogenation reaction (**Figure 1**). In this experiment, palladium on carbon (Pd/C) will be used as a heterogeneous catalyst for the process. A balloon will be used to supply the hydrogen atmosphere.

Principles:

The addition of hydrogen across a unit of unsaturation is called a hydrogenation reaction. Since its discovery in 1897 by Paul Sabatier, metal-catalyzed hydrogenation of π -bonds, such as alkenes (**Figure 2**), has evolved into an important process in chemistry. Over the years, new and more robust catalysts were developed, which expanded the scope of this process and enabled hydrogenations to be *asymmetric*. When a heterogeneous catalyst is used, the accepted mechanism (**Figure 3**) begins with adsorption of hydrogen onto the surface of the Pd/C catalyst. Next, binding of the alkene occurs followed by two sequential hydride transfers furnishes the saturated alkane. Under a homogenous catalyst, the mechanism is different. Notice that the ketone functionality can also undergo reduction. However, catalytic hydrogenation is *chemoselective* towards the alkene moiety.

Procedure:

- 1) Add 210 mg of chalcone, 12 mg of 5% Pd/C, and 8 mL of MeOH to a 25 mL round-bottom flask equipped with a magnetic stir bar.
- 2) Seal the round-bottom flask with a rubber septum and start stirring the reaction mixture.
- 3) Obtain a balloon of hydrogen from the hydrogen gas cylinder and set aside.
- 4) With a needle, apply a vacuum to the reaction mixture until bubbling is observed.
- 5) Then, stop the vacuum and insert the hydrogen balloon.
- 6) After 30 seconds, remove the hydrogen balloon.
- 7) Repeat steps 4-6 three more times.
- 8) Insert the hydrogen balloon and allow the reaction mixture to stir for 30 minutes.

- 9) Remove the hydrogen balloon and septum. Vacuum filter the reaction mixture through a pad of celite into a *tared* round-bottom flask.
- 10) Remove the solvent by rotary evaporation to obtain the product 3-phenylpropiphenone as a white solid.
- 11) Calculate the percent yield and establish its purity and identity by melting point and ^1H NMR.

Representative Results:

3-Phenylpropiphenone was obtained as a white solid (150 mg, 71% yield); m. p. 65 – 70 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.00 (d, $J = 7.2$ Hz, 2H), 7.59 (t, $J = 7.2$ Hz, 1H), 7.49 (t, $J = 7.6$ Hz, 2H), 7.37 - 7.26 (m, 5H), 3.35 (t, $J = 7.2$ Hz, 2H), 3.12 (t, $J = 7.6$ Hz, 2H)

Summary:

In this experiment, we have demonstrated a catalytic hydrogenation reaction of an alkene. Chalcone was hydrogenated to form 3-phenylpropiphenone.

Applications

Hydrogenation is an exothermic reaction (releases heat) because the product alkane is more stable than the reactant alkene. The amount of heat released from the reaction can serve as an indicator of the stability of the alkene. In the food industry, hydrogenation is used for processing vegetable oils, which are triglycerides bearing multiple alkene units. Varying the reaction conditions controls the degree of hydrogenation. Hydrogenation is used for the industrial synthesis of hydrocarbons from coal. This is known as the Bergius process and involves treating coal (elemental carbon) with high pressures of hydrogen and a metal catalyst under high temperature. Its inventor, Friedrich Bergius was awarded the Nobel Prize in Chemistry in 1931.

Legend:

Figure 1: Diagram showing the hydrogenation of chalcone to 3-phenylpropiphenone.

Figure 2: Diagram showing the general alkene hydrogenation reaction.

Figure 3: Diagram showing the mechanism for catalytic alkene hydrogenation.

Figure 1

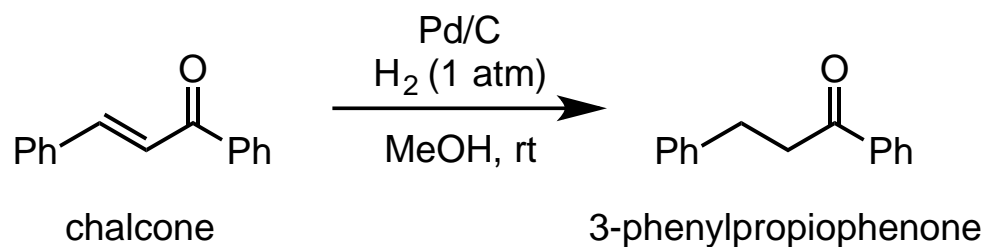


Figure 2

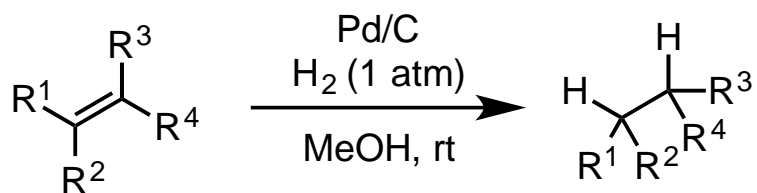


Figure 3

