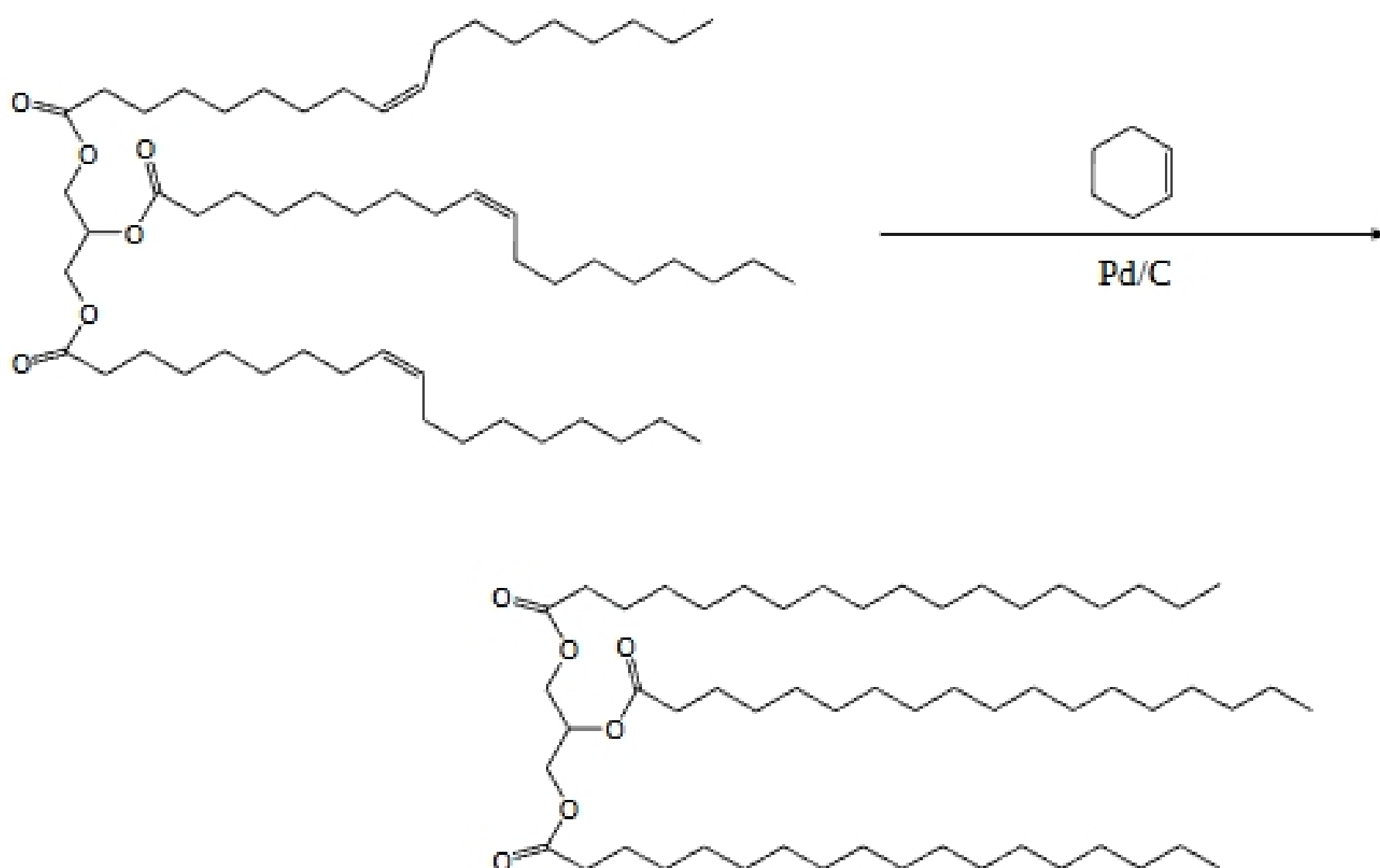


Transfer Hydrogenation of Olive Oil



Required reading:

Pavia, Lampman, and Kriz:

“Tests for Simple Multiple Bonds”, p. 466

Alkenes (and alkynes) can be hydrogenated with hydrogen gas in the presence of a special catalyst, usually palladium, platinum or nickel. Hydrogen gas is routinely used for hydrogenations in research and industrial labs, but handling gaseous reagents requires special equipment, and hydrogen gas is explosive. A convenient alternative to using hydrogen gas is to carry out a transfer hydrogenation. Certain organic molecules can act as hydrogen sources and “transfer” one or more equivalents of hydrogen to other molecules. In this experiment, you will perform a transfer hydrogenation on olive oil (an unsaturated fat) using cyclohexene as a hydrogen source to produce shortening (a saturated fat). The major fat of olive oil is triolein, which will form tristearin upon complete saturation.

Safety:

The palladium catalyst is very reactive and can cause fires on contact with certain organic liquids. Palladium is also toxic.

Procedure:

Place 400 mg of olive oil in a 5 mL conical vial. Add 1 mL of cyclohexene followed by 50 mg of 10% Pd/C. Add a boiling chip, attach a condenser and reflux the reaction mixture gently for 15 minutes. Allow the mixture to cool to room temperature.

Assemble a filter pipet by placing a small wad of glass wool in a disposable pipet. The wad should be pressed into the pipet gently with a piece of wire to secure it from falling out. Add about 1/2 inch of celite to the pipet and filter the reaction mixture through the filter pipet onto a watch glass. If black particles of the palladium catalyst get through, filter it again. Rinse the pipet with a few drops of hexane to recover the last traces of product.

Gently warm your fat solution for 10 minutes under a hood to remove the solvents and byproducts. Allow the residue to cool to room temperature; the saturated fat should solidify. If the fat does not solidify, cool it on an ice bath for 10. Determine the mass of the product and obtain an approximate melting point by placing about 50 mg (0.05 g) in a test tube and heating it slowly in a water bath, noting the temperature of the water when the fat melts.

Perform a qualitative test for unsaturation on your product by dissolving about 50 mg of it in 1 mL of dichloromethane and adding a 4% solution of bromine in CH_2Cl_2 (prepared by the instructor) until the color just persists (count the number of drops needed – a typical amount is about 25 – 30 drops from a Pasteur pipet). Perform the same test on about 50 mg of olive oil, using the same amount of 4% bromine in CH_2Cl_2 . The bromine is an orange-red color, which will dissipate if the bromine reacts with an alkene to form a colorless organic halide.

Write Up:

Be sure to include a calculation of the percent yield of your saturated fat. Discuss your melting point and compare it to the literature value. Discuss the results of your unsaturation tests.

Questions:

1. Cyclohexene can “transfer” one equivalent of molecular hydrogen to become molecule A or two equivalents to become molecule B. Draw the most likely structures of molecules A and B.
2. Ammonium formate ($\text{HCOO}^- \text{NH}_4^+$) can also be used as a hydrogen source in a transfer hydrogenation, giving up one equivalent of hydrogen. Write a balanced

equation showing the hydrogenation of 1-butene with ammonium formate in the presence of a Pd/C catalyst.