

CHEM-333: EXPERIMENT 4: STEAM DISTILLATION OF ESSENTIAL OILS; EXPERIMENTS A, C, D AND BELOW.

Reading: For this experiment read Chapter 10.

This week you will get to use steam distillation to isolate the oils from either caraway, cinnamon, clove, or cumin (see below) and prepare a derivative of the main component of that oil. You may wish to include all four derivatization equations in your notebook, or you may wait until you come to lab to find out which spice you get.

Steam distillation is not a common research laboratory procedure, but it is invaluable when it is useful, as in extraction of essential oils. I know what your thinking—didn't we just do distillation last week? Whereas the apparatus is the same, the physics is considerably different as your compound is "distilled" as a minor component of an aqueous mixture but at a much lower/ less destructive temperature than distilling the compound directly. Now you will know how to make those weird smelly extracts found in those hippie shops.

You will finish this lab next week, as well as getting a tour of the chemistry library to find out about your "Literature Search" project.

When assembling the setup as pictured in Figure 10.2, note that there is no need for a dropping funnel. The water initially in your round bottom flask will be sufficient for the distillation. Record the weight of your spice, and proceed as directed. Figure 10.2 does not include clamps, but they are obviously required. Boil the mixture vigorously, but watch for boil-overs! Collect about 125 mL into an Erlenmeyer flask; by the time you are done, the distillate should be clear and colorless (check for oily drops by catching some distillate in a small beaker).

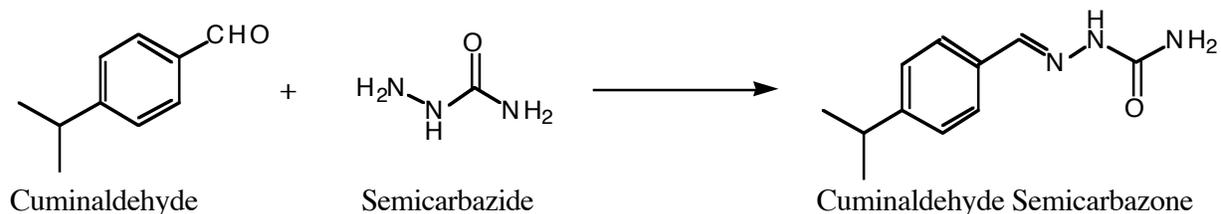
After you pour the distillate into the 250 mL separatory funnel, rinse the Erlenmeyer flask with 10 mL of methylene chloride using a pasteur pipette and add to the separatory funnel. Which layer is the methylene chloride? (Check the density in the back of your book.) Shake the funnel, let it settle, and collect the methylene chloride layer in a small Erlenmeyer flask. Repeat the extraction with another two 10 mL portions of methylene chloride. Add a sodium sulfate until it remains granular to the combined methylene chloride extracts, and filter it through a **tiny** piece

of cotton into a **weighed/tared** 50 mL round bottom flask. Then use the rotary evaporator to remove the solvent. Use the weight of the oil to calculate the percent of oil in the ground spice.

All waste solid from the steam distillation should be disposed of in the waste containers provided, **not** down the sink. You may not have time to complete the derivatization of your oil in one week, but there will be time next week. Use all of the oil that you have isolated.

Derivatization: Why derivitize? Often the compound you isolate is too volatile or unstable to characterize itself. By making a derivative that is less volatile (and/or stable) you can better characterize your compound by meltingpoint or other technique.

Cumin (Adapted from Moore/Dalrymple/Rodig)

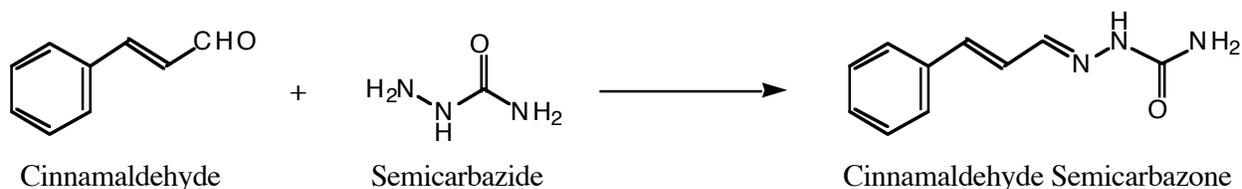


The dried fruit of cumin (*Cuminum cyminum* L.), a small shrub cultivated in eastern Europe and India, is an important seasoning in curries, goulashes, and sausages. It is also a major component

of chili powder used in Mexican food. The major volatile constituent of cumin is *p*-isopropylbenzaldehyde (cuminaldehyde). Cumin oil also contains limonene and other compounds which contribute to the aroma of this condiment, as can be observed by comparing the odor of the oil before and after isolating cuminaldehyde as the semicarbazone.

To prepare the semicarbazone, dissolve your oil in 3 mL of ethanol and transfer it to a test tube. Add to this a solution of 0.20 g of semicarbazide hydrochloride and 0.30 g of sodium acetate in 2 mL of water. Warm this mixture briefly on a steam bath, cool, and allow the derivative to crystallize. Collect the crystals in a Hirsch funnel and recrystallize from methanol: add enough so that the solid dissolves when heated on a steam bath, then cool to get crystals. Compare the melting point to the literature value (216° C); recrystallize again if necessary.

Cinnamom (Adapted from Pavia, Lampman, Kriz)



Cinnamom, widely used in cooking, is one of the best known of the spices. To prepare the semicarbazone, dissolve your oil in 3 mL of ethanol and transfer it to a test tube. Add to this a solution of 0.20 g of semicarbazide hydrochloride and 0.30 g of sodium acetate in 2 mL of water. Warm this mixture briefly on a steam bath, cool, and allow the derivative to crystallize. Collect the crystals in a Hirsch funnel and recrystallize from methanol: add enough so that the solid dissolves when heated on a steam bath, then cool to get crystals. Compare the melting point to the literature value (215 °C); recrystallize again if necessary.