

NATURAL PRODUCT ISOLATION

In this experiment you and a partner will isolate the volatile and fragrant component from a natural source using the purification technique of steam distillation. Once isolated, the 'essential oil' can then be identified by the use gas chromatography-mass spectroscopy (GC-MS). This experiment is modeled after Experiment #57 from your text. Also in this experiment you will learn to perform a 'macroscale' extraction using a separatory funnel (see technique 12, pp. 677–679 of PLKE)

You may bring your own natural product to steam distill (odiferous plant matter, fruit peelings, spices, etc.) or you can choose from the following which will be provided in lab: cloves, allspice, cumin, caraway, cinnamon, and fennel (all of these should contain an essential oil that you can isolate).

Procedure for isolation of the essential oil

To remove the essential oil from the organic/plant matter you will use the technique of 'steam distillation' (See technique 18, p. 750 of PLKE). The steam distillation apparatus will be a macro scale distillation apparatus shown on p. 714 of your text. You will use a 250 mL round bottom flask and a 250 or 500 mL heating mantle as a heat source that will be supplied by the stockroom.

Steam Distillation: Add approximately 2 g of your odoriferous natural product to a 250 mL round bottom flask with approximately 100 mL of water. If it is not already ground into a powder, obtain a mortar and pestle from the stockroom and pulverize your material. Assemble the steam distillation apparatus. For a heating source, use a 250 mL heating mantle heater*. Be sure to plug the heating mantle into a 'variac' outlet. Before you begin the distillation, have your instructor check your set-up. To distill the mixture, set the variac knob to 60. Turn the variac up if the drip rate is too slow. Distill approximately 25–30 mL of distillate into your beaker. The volatile organics should co-distill with the water while leaving heavier plant matter and salts behind (the presence of a 'milky white' distillate is a good sign that the essential oil is distilling out with the water).

*Before you set up the distillation apparatus you may want to verify that your heater is in working order by feeling for heat ~20 seconds after it is plugged into a variac set at 60.

Extraction: Place a separatory funnel (see p. 678 of PLKE) in a ring stand and add the distillate you collected from the distillation followed by 10 mL of methylene chloride. You should have 2 separate layers with the methylene chloride layer on the bottom. Shake the separatory funnel for 2-3 minutes then replace the separatory funnel to the ring stand and let the layers separate. Drain the lower methylene chloride layer into a beaker. Add another 10 mL of fresh methylene chloride and shake as before. Drain the methylene chloride layer and combine this with the previous extract. Dry the methylene chloride solution by adding 1 g of anhydrous sodium sulfate to the beaker and let it sit for 15 minutes (see **GC-MS preparation** step on next page).

Isolation of the essential oil. Once the methylene chloride solution has set for 15 minutes, decant the solution into a tared beaker and evaporate the methylene chloride by using a stream of air and heating with a warm water bath. When all the methylene chloride has been evaporated, 1-3 mL of an oily residue will remain. Re-weigh the beaker to get the weight of the oil that was recovered.

GC-MS preparation. While the methylene chloride solution is drying obtain a 'GC-MS collection vial' from your instructor. To this vial add 5 drops of the methylene chloride extracts and 1 mL of fresh methylene chloride. Cap the vial and label with the number corresponding with your name on the GC-Sign-up sheet (as we did in the biodiesel lab).

Your GC-MS sample will be analyzed within 2–3 days from when you turn it in. Tape the GC-MS output to your lab notebook.

Websites to look up compounds that were identified by GC-MS:

<http://www.devointl.com/compendium/>
<http://www.nysaes.cornell.edu/flavornet/index.html>

(Extra credit) NMR preparation Once you have weighed your oil, place 3-5 drops in your NMR tube and add 0.07 mL of CDCl_3 .

(Extra credit) IR preparation. Place one drop of your oil on a salt plate and take an IR spectra. Don't forget to clean the salt plate once done with methylene chloride.

What is due (Wednesday, February 28):

- Photocopies of the appropriate lab pages, including purpose, materials, methods, data (including GC-MS output).
- Abstract (usual format), something like “ ____ grams of _____ was steam-distilled to yield ____ grams of the essential oil. GC/MS analysis of the oil revealed that its principal component was _____, with lesser amounts of (list of chemicals). This is (consistent/inconsistent) with the chemicals found in this oil by (reference name), (then state how it is or is not consistent).”