



Procedure:

- With a partner, assemble a steam distillation apparatus, similar to that shown on p.132, Fig. 11.25 in your textbook as shown. Substitute a separatory funnel for the bent adapter, as shown in the apparatus set-up on the front bench.
- Add about 100 mL of water to the round bottom flask and add your spice through one of the openings in the flask.

Some spices will need special instructions for adding them to the flask. Your instructor will give you these specifics when you receive your spice or fruit.

- Before you begin have your apparatus approved by your instructor who will show you how to use the steam line.
- Continue the steam distillation until you have collected about 200-300 mL of distillate.
- Cool the distillate in ice and carefully divide it into two portions (some extracts will float on water, so if you pour off a portion for your partner, they may get all of the spice).

Each student will perform individually:

- Transfer it to a 250 mL separatory funnel and extract 2 x 30 mL with methylene chloride.
- Dry the combined extracts over anhydrous calcium chloride until the solution is clear
- Filter by gravity (decant if possible), into a pre-weighed round bottomed flask.
- Add boiling chips and evaporate the methylene chloride over a steam bath in the hood. Do not over evaporate to dryness!
- The residue will be your essential oil, which will range from a trace to > 1 mL, depending on which spice you chose. Obtain the mass.
- Prepare a dilute solution of your oil in methylene chloride, about 1 drop oil in 1 mL CH_2Cl_2 . Determine the retention time and relative amount of the major component(s) in the oil by gas chromatography (GC).
- Obtain an IR spectrum.

Clean up

- Be sure not to let the residue from the steam distillation set, it is best cleaned up when still slightly warm. See your instructor about the disposal of your spice.
- IR salt plates must go back into the tin when the last person is finished using them.
- All aqueous extract can go down the drain.

Possible Spices

Allspice	Lemon Peel
Anise	Nutmeg
Caraway	Parsley
Cardamom	Peppermint
Chamomile	Rosemary
Cinnamon	Sage
Clove	Sesame
Coriander	Spearmint
Cumin	Thyme
Ginger	Tumeric
Laurel berry	Valerian root

Post-Lab Questions

1. What is the normal boiling point of your spice component(s)? What advantage does steam distillation provide in this experiment?
 2. Compare and contrast the use of GC in Experiment #2 (distillation) and Experiment #3 (steam distillation).
 3. Fully analyze your IR spectrum. Based on what you have learned in lecture identify as many of the IR bands as possible. Were you able to find all of the functional groups? Obtain a literature spectrum of the pure component (links will be provided to help you). Do they match up? Note any differences.
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