

## STEAM DISTILLATION OF CLOVES

Read Tech #722 for background information, but follow procedure below -

Read the Introduction and Semi-Microscale procedure in the lab manual for background info. Also perform the 3 qualitative tests described in the "Characterizing the Product" section, Part 1. They include  $\text{Br}_2$ , Permanganate, and  $\text{FeCl}_3$  tests.

### SAFETY

Your TA will demonstrate proper and safe use of separatory funnels.

Dichloromethane has a very low boiling point. Therefore, the distillate **must** be cool (at room temperature) before adding dichloromethane and extracting.

Dichloromethane is a suspected carcinogen and must be used in a fume hood.

### PROCEDURE

Start with approximately 3g of ground cloves. Do not weigh, but estimate the amount by sight (your TA will show you what the proper amount looks like). Place cloves and 100 mL distilled water in a 250 mL round-bottom flask. Set up a simple distillation as in the distillation experiment using a 250 mL flask heater (the TA will have a demo set-up). The TA **must** check your apparatus before you start. Heat the flask with cloves slowly, usually at 4-5 on the heater dial, and only increase heat slowly. If the heating is too vigorous, the mixture will froth over into the receiver.

Collect only about 40-50 mLs distillate, which should contain the oily eugenol. Don't distill for so long that you don't have enough time for the rest of the experiment. Make sure you know how to safely use the separatory funnels, particularly regarding safe venting. Don't shake the sep funnel, but gently rock it back and forth to minimize emulsion. Extract 3 times in a 125 mL sep funnel using 10 mL portions of dichloromethane, and pool the extracts into a 150 mL beaker. Make sure you know which layer to save, and try to avoid collecting any of the aqueous layer (refer to main procedure part 2, page 53, for information on extraction).

Add about 1g anhydrous sodium sulfate to remove any remaining water, swirl to mix, and then decant into another small beaker. Rinse the sodium sulfate with another 2 mL dichloromethane. Combine the dichloromethane solutions in a roundbottom flask and *carefully* evaporate the solvent using a rotary evaporator (your TA will instruct you how to use the evaporator).

Perform 3 qualitative tests ( $\text{Br}_2$ , Permanganate, and  $\text{FeCl}_3$ ) as described in the Introduction and 'Characterizing the Product' sections. Use **extra caution handling bromine**, which can cause serious chemical burns.