

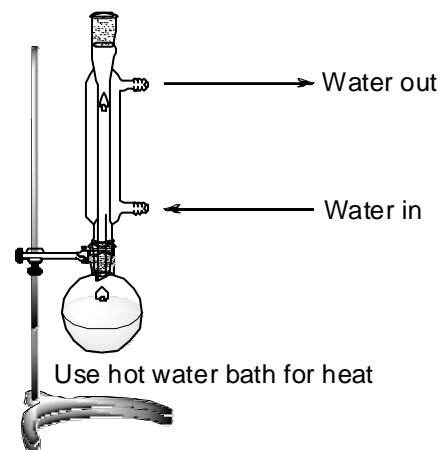
Experiment 3

CHEM 203

Experiment #3: Isolation of Trimyristin from Nutmeg

Isolation of Trimyristin:

Extraction of Trimyristin: Add 2 g of nutmeg to a 25-mL or 50-mL round-bottomed flask, followed by 10 mL of dichloromethane. Add a boiling chip and fit the round-bottom flask with a reflux condenser. Gently heat the mixture for 30 minutes. After which, cool the resulting mixture to room temperature and vacuum filter the resulting solution through a pad of Celite® using a Büchner funnel. The filtrate (solution in the filter flask) is the material that you will use for the next part of the experiment.



***Important note on making the Celite® pad:** Weigh out between 1.75-2.00 g of celite into a small beaker. When you are ready to filter your solution, add approximately 10 mL of dichloromethane to the celite and mix to make a slurry with the consistency of oatmeal. Place a piece of filter paper inside the Büchner funnel and pour the celite slurry onto the filter paper. Make sure to cover the filter paper evenly so that it cannot be observed through the celite. Then pour your sample onto the celite pad. Once the solution is on the Celite® pad, immediately turn on the vacuum. Then, wash the Celite® pad with approximately 2 mL of dichloromethane.

Purification of Trimyristin: Transfer your filtrate to a pre-weighed Erlenmeyer flask or vial. Add a boiling stick and evaporate off the dichloromethane, which should result in an oily material, which you should weigh. Then, recrystallize the residue using ethanol. For a rule of thumb with the experiment, you should use around 10 mL of 95% ethanol per gram of residue. Filter your product using vacuum filtration, dry the sample, and record its weight. Set aside a small amount to record the melting point next week. With the remainder perform the hydrolysis procedure.

Hydrolysis of Trimyristin:

Place 1 g of your purified trimyristin, 2 mL of 6M NaOH and 20 mL of absolute ethanol in a 125 mL Erlenmeyer flask and heat on a hot water bath for 15 minutes with swirling, a large amount of white solid should precipitate. After cooling to room temperature, add 20 mL of water to the flask, followed by 40 mL of 35% aqueous NaCl. Vacuum filter the white solid and wash it with 30 mL of cold water.

Dissolve the white solid in 20 mL of water and cool in an ice-water bath. Slowly acidify the solution with 6N HCl until it is acidic by litmus paper. Vacuum filter the solid precipitate and wash it with 30 mL of cold water. Allow the myristic acid to dry in your drawer until next lab period, then obtain a melting point and IR spectrum. The melting point of pure myristic acid is 54-56 °C.