

5. Solid-Liquid Extraction: Trimyristin from Nutmeg

This procedure and material has been adapted from the macroscale procedure submitted by G.D. Beal and checked by H.T. Clarke and E.R. Taylor as described in *Organic Syntheses*, Coll. Vol. 1, p. 538 (1941); Vol. 6, p. 100 (1926).

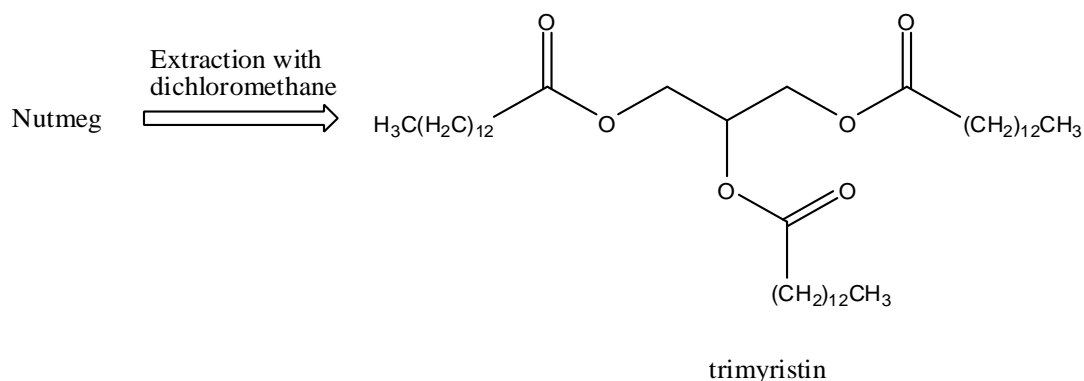
In a nutshell

In this experiment, you will be performing a solid-liquid extraction. You will use an organic solvent to extract the trimyristin from the spice, nutmeg.

Background

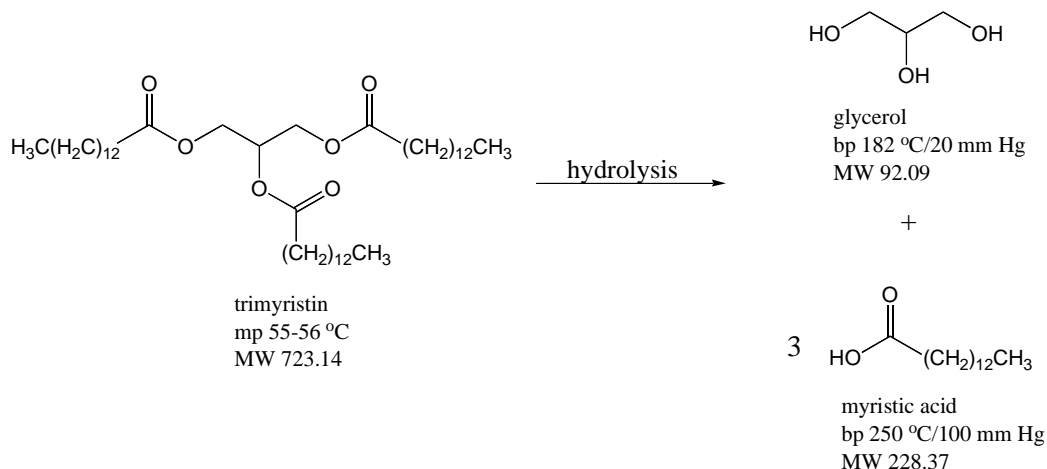
Believe it or not, a lot of you have performed this “experiment”, a solid-liquid extraction, at home anytime that you have made coffee or tea. In the first case, you are extracting the “coffee” components from the ground coffee beans using hot water. It is the same for tea but you are using a tea bag to leave behind any insoluble material.

Nutmeg, another solid, is a spice native to the Spice Islands and contains several components including cellulose, fats and essential oils. One of those fats found in nutmeg is trimyristin, which can be isolated from nutmeg through extraction with dichloromethane. This extraction and recovery of trimyristin will be the focus of today’s experiment.



In this experiment, you will be extracting a trimyristin from nutmeg using an organic solvent. The original procedure was published in 1926 and called for 1500 g of crushed nutmegs to be extracted with 500 mL of diethyl ether. In your case, you will be using 2 g of nutmeg and dichloromethane as your extraction solvent to isolate the crude oil, which will be recrystallized from ethanol.

If we wanted to continue on, trimyristin could be hydrolyzed to a fatty acid, myristic acid, and a triol, the trialcohol glycerol. Hydrolysis is a chemical reaction in which a chemical compound reacts with water to form two molecular fragments. In our case, trimyristin, a triester, hydrolyzes to glycerol and myristic acid. The hydrolysis is typically carried out by refluxing the ester in an aqueous base, such as KOH or NaOH, so the resulting myristic acid is produced in its salt form, a myristate. This process is known as saponification (making soap). After saponification is complete, the salt can be acidified to release the free carboxylic acid as a solid. The overall reaction is:



After completing this experiment, you should understand and/or perform the following.

- 1) write an extraction scheme for this experiment
- 2) understand the different extraction processes

Notebook notes.

You **MUST** write out a flowchart of the experimental procedure in your notebook so that you can follow where you are in the extraction. Also, record any physical change in the reaction (i.e., color) as well as the measurements of the reagents, the crude material and product.

Experiment

In this experiment, make sure to record your weights of reagents, crude material, and final product. Also, record any physical observations you make, such as color changes, during this extraction.

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. Record the melting point of your final product.