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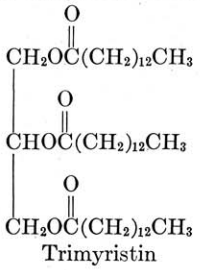
Trimyristin from Nutmeg

The isolation and reaction of natural products has a greater appeal to students than working from laboratory books with compounds whose greatest virtue is that the reaction is known to give good results. Some recent organic laboratory books take advantage of this natural product appeal using, for example, the isolation and reactions of caffeine to introduce students to many laboratory techniques. We propose the isolation and saponification of trimyristin from nutmeg as an excellent experiment to introduce general chemistry students to laboratory techniques or for use with the organic chemistry course in which case more techniques can be introduced. At Illinois Wesleyan, this experiment has been used in the second semester of general chemistry to introduce the techniques of extraction, recrystallization, refluxing, and melting points in two 3-hr laboratory periods with excellent results and high student interest. In the organic chemistry laboratory, the above techniques plus thin layer chromatography and infrared analysis were used. The equipment necessary can be as simple as a beaker, a test tube, and a gravity funnel which we use when the experiment is used for general chemistry.

Trimyristin (see structure below) is the triglyceride of myristic acid and is somewhat unusual in that high concentrations of it are found in nutmegs without large amounts of closely related esters. Thus, this isolation is not representative of most natural product isolations which are quite tedious and complex. At least one current organic laboratory manual details the isolation of trimyristin; however, it goes no further.[1]



Trimyristin

We use the whole nutmeg[2] and grind it, then extract with ether at room temperature in a beaker. Possible variations here include use of commercial ground nutmeg, refluxing with a condenser or use of a Soxhlet extractor. Recrystallization from acetone is fast enough to permit development of good technique within a short time (several attempts can be made within a half hour). An advantage in the low melting point of trimyristin (56°C) is that a water bath can be used as a melting point bath.

Saponification of trimyristin requires ethanol to be present for solubility to occur and hence reaction. This is a place students have trouble if not enough alcohol is present at all times, and the importance of solubility for reaction to occur is well illustrated.[3] An interesting feature of this saponification is that starting material (trimyristin) and product (myristic acid) have almost identical melting points (56° and 54°C respectively) and therefore melting points alone are no indication of product identity; other methods must be used. Thin layer chromatography may be used to identify the compounds and their mixtures. Mixed melting points can be used at this point. If the saponification is incomplete, the mixture is refluxed longer with base. This is a good place to introduce infrared spectra either by having available spectra of the pure triglyceride and acid and having the student compare his with these, or by having known spectra nonavailable and using the 0.1 micron difference between the carbonyl stretching absorption bands of the glyceride (5.75 micron) and acid (5.85 micron).

Another variation is use of the saponification to determine the neutralization equivalent followed by acidification of the reaction solution to recover myristic acid.

For use in general chemistry we have used isolation of trimyristin and determination of its melting point followed by saponification. The following experimental procedure for isolation and saponification is that used in general chemistry and can be varied according to equipment available.

Experimental

Extraction. A nutmeg seed is crushed and ground in a mortar. Ether (50 ml) is stirred with the solid for about 10 min and the resulting mixture is gravity filtered. The ether is evaporated from the filtrate by placing the beaker containing the solution into a larger beaker of warm water in the hood. The resulting yellow oil is dissolved in a small amount of warm acetone, and the solution is cooled to get a white solid, mp 55–56°C.

Saponification. To 0.30 g of trimyristin is added 10 ml of 6*N* sodium hydroxide and 10 ml of ethanol. The solution is gently boiled for 1-hr and poured into 100

[1] Helmkamp, G. K., and Johnson, Jr., H. W., "Selected Experiments in Organic Chemistry," 2nd ed., W. H. Freeman and Co., San Francisco, 1968, p. 58.

[2] We obtain our whole nutmegs from a food store in cans containing nine nutmegs each.

[3] Without ethanol no saponification occurred after several days of refluxing.

ml of water. Addition of 20 ml of concentrated hydrochloric acid results in formation of a white solid which is washed with 5 ml of water and dried, mp 53–54°C.

Thin-Layer Chromatography. Eastman Chromagram Sheet 6060 silica gel was used with 90% benzene, 10% absolute ethanol (V/V) as developer, and an iodine chamber for detection. R_f values were 0.57 for trimyristin and 0.78 for myristic acid.