Fig. 34-1

Experiment 34

A FATTY ACID FROM NUTMEG

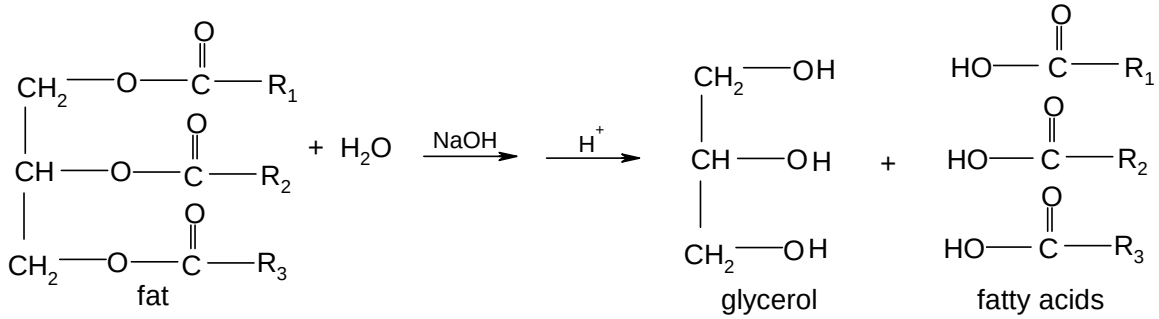
Sir Derek H. R. Barton (1918 -1998).
Received Nobel Prize in 1969 for work in natural product chemistry.
http://nobelprize.org/nobel_prizes/chemistry/laureates/1969/barton-bio.html

Test Topics and Comments

Tiglycerides, fatty acids, soap. This experiment will probably require two laboratory periods.

Discussion

Many of the products that we use (including the medical field) are extracted from natural products. A few examples include penicillin, quinine, taxol (an antitumor drug) and cyclosporine (inhibits rejection of transplanted organs). In today's experiment, techniques from several previous experiments will be combined to obtain and identify a chemical from nutmeg. As described in ***Experiment 5***, triglycerides can be saponified to fatty acids. In this experiment, a triglyceride will be extracted from nutmeg. After saponification, the fatty acid obtained should be identifiable from a determination of its melting range.



saponification followed by acidification

Commonly, saponification of triglycerides derived from natural products yields a mixture of fatty acids. Fatty acids derived from plants are usually richer in unsaturated fatty acids than those derived from animals. The triglyceride derived from nutmeg is unusual as it contains only one fatty acid. The fatty acid obtained in this case is saturated. Since saturated fatty acids are solids and often can be distinguished using melting range measurements, nutmeg is a very convenient starting material for this experiment. On the negative side, saturated fatty acids contribute to health problems. Seek more information on the health issues in the library or on the Internet.

The salts that are obtained as a result of saponification before the acidification step were discovered more than 5000 years ago and are still used today as soaps. Refer to your organic chemistry textbook for a discussion of the properties of soaps and the mechanism of their cleansing action.

Procedure

Set up a reflux apparatus with a 50 ml round bottom boiling flask equipped with boiling chips or a magnetic stirrer. Heat can be supplied with a heated water bath, a steam bath or a heating mantle. Transfer 5 g of ground nutmeg to the round bottom and add 15 mL of diethyl ether. Reflux for about 30 minutes. Decant off the ether layer and properly dispose of the solid remaining in the flask. Remove the ether by rotary evaporation or simple distillation. A solid residue should result.

Recrystallize the solid from acetone (about 5 mL). Transfer the solid to a 25 mL round bottom, add 0.1 g of sodium hydroxide and 5 mL of 95% ethanol and reflux for 15 minutes. Allow the mixture to cool to room temperature and add 5 mL of water to the mixture. Pour the reaction mixture slowly with vigorous stirring into a beaker containing 5 mL of 3 M HCl and about 5 g of ice. After precipitation is complete, add another 10 mL of water and filter using a Hirsch funnel. Wash the collected solid on the filter with ice-cold water. After the sample has dried, determine the yield and the melting range of the solid. If the sample is impure, recrystallize by dissolving the solid in a small amount of boiling methanol and then adding water dropwise (while maintaining boiling) until there is the first hint of cloudiness. Cool and collect the recrystallized product using vacuum filtration. Based on the melting range, determine if the product is lauric acid, myristic acid, palmitic acid, stearic acid or some other acid.

Reference

de Mattos, M. C. S.; Nicodem, D. E. *J. Chem. Educ.*, **2002**, *79*, 94.

Prelaboratory Preparation - *Experiment 34*

First, be sure to list all the goals of the experiment. Describe the properties that enable a compound to be a candidate for a cleaning agent. Also compare the functional group of soaps to the functional group of detergents. What is the problem with consumption of saturated fatty acids? Why do the fatty acids listed as possible products all contain an even number of carbons? Prepare a table for insertion of useful and observed data such as molecular mass, mass, moles, melting points and percent yields and recoveries and look up and record the melting ranges of common fatty acids. Find saponificaton of an ester to a carboxylic acid in the *Reaction-Map of Organic Chemistry* in ***Appendix C*** and include the number of the reaction in your report.

Observations

Report all relevant observations including, masses and melting ranges.

Conclusions

This section should include the following:

1. Were the goals of the experiment achieved? Explain your answer.
2. What was the identity of your product? Explain your answer.
3. Based on the melting range of your product, comment of the purity of the product.
4. Assuming that there were no mass losses during the procedure, calculate the mass percent of the fatty acid in nutmeg. What does this result mean to you?
5. How could the percent recovery have been improved?
6. Would nutmeg be a good starting material for preparation of soap? Explain your answer.