M. S. Ntamila
and A. Hassanali
University of Dar es Salaam
P.O. Box 35061
Tanzania

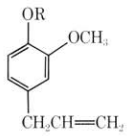
Isolation of Oil of Clove and Separation of Eugenol and Acetyl Eugenol

An instructive experiment for beginning chemistry undergraduates

Although the study of naturally occurring compounds has been an important preoccupation of organic chemists, few exercises involving the isolation, separation, purification, and physical and chemical study of natural products suitable for beginning students have been worked out.[1] The present experiment which we have developed at Dar es Salaam,[2] has the advantages that it is simple to perform, requires only small quantities of readily available material, and may be completed within a normal practical period.

Cloves are the dried unopened buds of the clove tree, *Eugenia caryophyllata* (also known as *Eugenia aromatica*),[3] and are among the oldest spices and drugs in history.[4] Today cloves are used mainly in three ways: as a spice, as a stimulant by chewing and smoking, and by distillation of clove oil which is widely used in perfumery, dentistry, and medicine.[3,5]

Clove oil obtained from Zanzibar cloves consists principally of eugenol (I), 85–90%, with large amounts of acetyl eugenol (II), 9–10%, and trace quantities of furfural, methyl-*n*-amyl-ketone, caryophylline, methyl salicylate, and others.[3]



(I) R = H
(II) R = $COCH_3$

The experiment consists of steam distillation of crushed cloves followed by extraction of the distillate with an organic solvent. The oil of clove obtained this way may be treated simply as a mixture of eugenol (I) and acetyl eugenol (II), the trace components being virtually undetected. Separation of the two is readily achieved since one is phenolic. Essentially pure specimens of each are obtained. Use of thin-layer chromatography (Silica Gel coated 20 × 4-cm plates or microscope slides) helps the student confirm that separation has indeed been achieved.

Chemical study of the two components involves the performance of a number of tests including bromine, ferric chloride, and hydroxamic ester tests, and the preparation of the benzoyl derivative of eugenol.

Finally, ir spectra of the two components are examined, and the information obtained compared with that from chemical tests.

Practical Details

Crush about 30–35 g of cloves with pestle and mortar and place in the distillation flask (500 ml) containing about 100 ml water; steam-distil for 1½ hr heating both the boiler and the distillation flask.

The receiver contains oil of clove, some separated and some dispersed in water. Extract the mixture with 3 portions of 25 ml chloroform. Examine the chloroform extract using the tlc plates (Silica Gel coated plates and running with chloroform/ligroin: ¾). Develop the plate by exposing to iodine vapor or preferably spraying with concentrated sulfuric acid in ethanol and then heating at 120°C. Retain a small sample (0.5 ml) of the chloroform solution.

For the separation of the two major components, extract the chloroform solution with 3 portions of 25 ml 5% sodium hydroxide solution. The chloroform layer which now contains mainly acetyl eugenol is dried over anhydrous sodium sulfate, filtered, and the chloroform removed by distillation. Yield of acetyl eugenol: 0.2–0.3 g.

The combined alkali extracts are made acid to litmus by the addition of hydrochloric acid and extracted with chloroform. The chloroform extract is similarly dried over anhydrous sodium sulfate, filtered, and evaporated to dryness. Yield of eugenol: 2–3 g.

Examine solutions of eugenol and acetyl eugenol against that of the original extract by thin-layer chromatography. The original oil of clove will be seen to have been clearly separated into its two major components.

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[1] The isolation of caffeine from tea is one of the oldest experiments available. See "Experimental Organic Chemistry," Baldwin, J., McGraw-Hill Co., New York, **1970.**

[2] See also Unilever Laboratory Experiment No. 5 on "The isolation of oil of clove followed by extraction of eugenol."

[3] Guenther, E., (*Editor*), "The Essential Oils," Vol. IV, Van Nostrand Co. Inc., New York, **1950,** pp. 396–436.

[4] Tidbury, G. M., "The Clove Tree," Crosby Lockwood and Son, Ltd., London, **1949.**

[5] "The Merck Index," 8th. Ed., Merck and Co., Inc., U.S.A., **1968,** p. 758.