

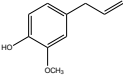
CHEM 304 Experiment Prelab Coversheet

Name: Justin Arthur Student Date: 08/27/2014 Exp. #: JAS-11

Title: Isolation of Eugenol from the Steam Distillation of Cloves

Purpose: To isolate eugenol from cloves by steam distillation and analyze it using IR and $^1\text{H-NMR}$ spectroscopy

Chemical & Safety Data:

Substance	Structure	MW (g/mol)	m.p. (°C)	b.p. (°C)	Density (g/mL)	Role
Name: Dichloromethane	N/A	84.93	-97	39 to 40	1.32	Solvent
Formula: CH_2Cl_2		Safety: Irritant, possible cancer hazard, inhalation may cause CNS effects				
Name: Sodium sulfate	N/A	142.04	884	N/A	N/A	Drying Agent
Formula: Na_2SO_4		Safety: Possible irritant				
Name: Eugenol		164.20	-12 to -10	254	1.06	Analyte
Formula: $\text{C}_{10}\text{H}_{12}\text{O}_2$		Safety: Irritant, potential allergen				
Name: Chloroform-d	N/A	120.38	-64	61	1.5	Solvent
Formula: CDCl_3		Safety: Irritant, possible cancer hazard, inhalation may cause CNS effects				

Safety Notes:

- When heating a reaction apparatus, be sure that it is open to the air so that pressure build up and rupture of the apparatus does not occur.
- When heating liquids, make sure the liquid is stirred (or a boiling chip is added) to prevent "bumping".
- When performing an extraction, make sure to vent the separatory funnel often to prevent pressure build-up

Prelab: _____ (25 points)

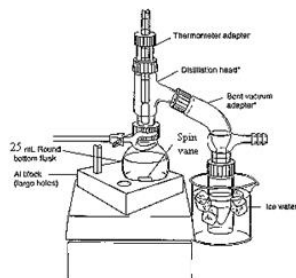
Inlab: _____ (15 points)

Postlab: _____ (60 points)

Total: _____ (100 points)

DATE	EXP. NUMBER	EXPERIMENT		
8/28/2014	JAS-11	Isolation of Eugenol from Cloves		
NAME			LAB PARTNER	WITNESS
Justin Arthur Student				

1. The apparatus sketched below was assembled and charged with 1.032 g of ground cloves and 17 mL distilled water. The cloves were soaked for about 15 min until thoroughly wetted.



2. The mixture was heated to boiling. Initial hot plate setting = 3. After 20 min, mixture still not boiling so setting increased to 7. Distillate collected at rate of about 1 drop/2-3 sec. ~~10 mL~~ 6.5 mL of distillate collected, then distillation was discontinued.
3. The distillate was transferred to a separatory funnel, extracted with 2 mL dichloromethane, then again with (2 x 1 mL) dichloromethane
4. The combined organic extracts were dried over sodium sulfate, and transferred to a tared ~~beaker~~ conical vial (Tare wt = 18.643 g).
5. The dichloromethane was evaporated by heating on a hot plate (UNDER HOOD!) to yield a pale yellow oil.

$$\text{Vial + eugenol} = \underline{18.720 \text{ g}}$$

$$\text{Wt eugenol} = \underline{0.077 \text{ g}} \quad (\% \text{ Recovery} = (0.077 \text{ g}/1.032 \text{ g}) \times 100 = 7.46\%)$$

6. An IR spectrum was obtained by the thin film method.

Comment [JMH1]: Note that if a mistake is made in the notebook, it should be crossed out with ONE LINE ONLY. The entry should be legible even after crossing it out.

Isolation of Eugenol from Cloves by Steam Distillation and its Identification by Infrared Spectroscopy

Justin Arthur Student
CHEM 304
September 8, 2014

INTRODUCTION

“Essential oils” are the volatile components associated with the aromas of many plants.¹ In this experiment, the essential oil eugenol (the main component of oil of cloves) will be isolated from ground cloves by steam distillation and identified by infrared spectroscopy.

The principle of steam distillation is based on the fact that two immiscible liquids will boil at a lower temperature than the boiling points of either pure component, because the total vapor pressure of the heterogeneous mixture is simply the sum of the vapor pressures of the individual components (i. e. $P_T = P_A^o + P_B^o$, where P^o is the vapor pressure of the pure liquids). This leads to a higher vapor pressure for the mixture than would be predicted for a solution using Raoult's Law (that is $P_T = P_A^o N_A + P_B^o N_B$, where N is the mole fraction of the component in the mixture). The higher total vapor pressure leads to a lower boiling point for the mixture than for either single component.² During the isolation of a liquid natural product by steam distillation, water is one of the components, and the liquid natural product being isolated (which is immiscible with water) is the other component. The product can be steam distilled from the natural source at a relatively low temperature (always less than 100 °C), thus avoiding decomposition of the product.²

Steam distillation can be carried out in two ways: the direct method and the live steam method.³ In the direct method, steam is generated by boiling a mixture of the source of the compound of interest and water. The live steam method is carried out by passing steam from an external source into the distillation flask. The direct method of steam distillation will be used in this experiment.

EXPERIMENTAL

The apparatus shown in Figure 1 below was assembled and the 25-mL round bottom flask was charged with 1.032 g of ground cloves and 15 mL of distilled water. The cloves were allowed to soak in the water until thoroughly wetted (about 15 min), then the mixture was distilled, the distillate being collected at the rate of about one drop every 2 – 3 seconds. After about 6 mL of distillate were collected, the distillate was extracted with 2.0 mL of CH_2Cl_2 (aka DCM), then again with (2 x 1.0 mL) of DCM. The DCM extracts were combined, dried over Na_2SO_4 , and evaporated to give the product eugenol as a pale yellow oil.

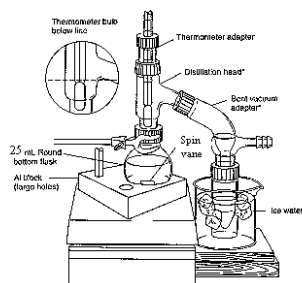


Figure 1⁴

Product mass: 0.077 g (7.46% recovery)

FTIR (film, NaCl plates): 3560 (OH), 3080 – 3000 (sp^2 CH), 2980 – 2940 (sp^3 CH), 1640 (alkene C=C), 1600, 1514 (aromatic C=C) cm^{-1} .

Comment [h2]: TITLE. Note that the title summarizes in one sentence the purpose of the experiment

Comment [h3]: Lab Reports should be written using passive voice. The experimenter is ideally an objective observer, and writes his or her reports as such.

Comment [JM4]: This example report assumes that the student has discussed the principles of IR spectroscopy in a previous lab report. If this is a technique new to the student, the principles of that technique must be discussed in this section.

Comment [JM5]: PURPOSE of the experiment

Comment [JM6]: PRINCIPLE of the MAJOR technique(s) introduced in the Technique lab. Note that the source of the information used in these introductory paragraphs has been cited. And that this information is presented in such a manner as to indicate the familiarity of the writer with the technique.

Comment [h7]: The experimental description here assumes the reader is schooled in the techniques of organic chemistry. Note that actual amounts are used, not necessarily the amounts listed experiment handout.

Comment [jm8]: Include all results here.

DISCUSSION

Steam distillation of cloves produced 0.0770 g of an oil which contained in its IR spectrum the functional groups O-H (at 3560 cm^{-1}), $\text{sp}^2\text{ C-H}$ ($3080 - 3000\text{ cm}^{-1}$), aliphatic C-H ($2980 - 2940\text{ cm}^{-1}$), and both alkene C=C (at 1640 cm^{-1}) and aromatic C=C (at 1600 and 1514 cm^{-1}). The IR spectrum is attached to this report. These data are consistent with the structure of eugenol, shown in Figure 2 below:

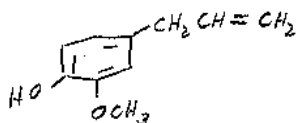


Figure 2: Eugenol

In addition, the IR of the product from the steam distillation of cloves closely corresponds with that of an authentic sample of eugenol shown in the Spectral Database for Organic Compounds.⁵ Therefore, it can be concluded that the oil which was isolated from cloves is in fact, eugenol.

0.0770 g of eugenol was recovered from 1.032 g of cloves. This corresponds to a percent recovery of 7.46%:

$$\% \text{ Recovery} = \frac{\text{Amt. Eugenol isolated}}{\text{Amt. Cloves used}} \times 100 = \frac{0.0770\text{ g}}{1.032\text{ g}} \times 100 = 7.46\%$$

Although the % recovery (7.46%) seems slightly low relative to the expected 10%⁵, the experiment proceeded as planned. There were no spills or other abnormal physical losses. It is possible that the ratio of the size of the glassware to the theoretical amount of eugenol which can be obtained from cloves in this experiment is large, leading to adherence of a large percentage of the product on the sides of the glass apparatus. If this is so, then steam distillation of a larger sample of cloves should give an improved recovery. Otherwise, it can be concluded that the specific sample of cloves used contains approximately 7.5% eugenol.

CONCLUSION

In this experiment, it was shown that about 7.5% of an oil could be recovered from cloves by steam distillation. This oil was identified as eugenol by comparison of its infrared spectrum with an authentic sample.

REFERENCES

1. Pavia, D. L.; Lampman, G. M.; Kriz, G. S.; Engel, R. G. *Introduction to Organic Laboratory Techniques, A Microscale Approach*; 3rd ed.; Brooks/Cole: Pacific Grove, CA, 1999; p. 139.
2. Pavia, D. L.; Lampman, G. M.; Kriz, G. S.; Engel, R. G. *Introduction to Organic Laboratory Techniques, A Microscale Approach*; 3rd ed.; Brooks/Cole: Pacific Grove, CA, 1999; p. 663.
3. Pavia, D. L.; Lampman, G. M.; Kriz, G. S.; Engel, R. G. *Introduction to Organic Laboratory Techniques, A Microscale Approach*; 3rd ed.; Brooks/Cole: Pacific Grove, CA, 1999; p. 665.
4. Wenqiang, G.; Shufen, L.; Ruixiang, Y.; Shaokun, T.; Can, Q. *Food Chemistry*, **2007**, *101*, 1558.
5. Spectral Database for Organic Compounds (SDBS). http://riodb01.ibase.aist.go.jp/sdbs/cgi-bin/cre_index.cgi?lang=eng (accessed January, 2012)

Comment [JM9]: Structures must be drawn BY HAND. This will give the student more practice and also help prevent errors.

Comment [h10]: THOROUGHLY INTERPRET YOUR DATA, provide the meaning of each piece of data, and state what can be concluded from these data.

Comment [JM11]: PROVIDE CALCULATIONS. Make sure to show all of your calculations

Comment [h12]: More conclusions that can be made from the data.

Comment [h13]: COMMENT ON SOURCES OF ERROR and how they could be avoided or minimized.

Comment [h14]: References must be cited using numbered endnotes in the order in which they first appear in the text.

Reference formats are taken from *The ACS Style Guide*; 2nd ed.; Dodd, J. S., Ed.; American Chemical Society: Washington, D. C., 1997 (the latest edition is available in Dacus Library).

ANSWERS TO QUESTIONS

1. Why is eugenol steam-distilled rather than purified by simple distillation?

Eugenol has a high boiling point (254 °C), and many organic compounds decompose at such high temperatures. Steam distillation allows eugenol to be distilled at a much lower boiling point (< 100 °C), thus minimizing the potential for decomposition.

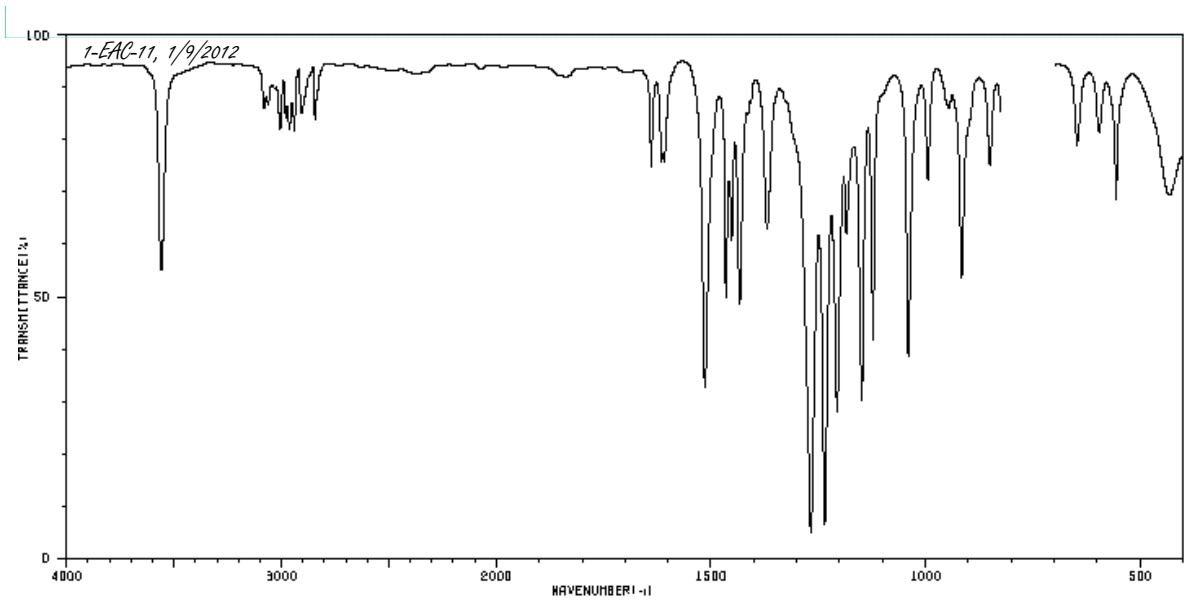
2. In a steam distillation, the amount of water actually distilled is usually greater than the amount calculated, assuming that both water and organic substance exert the same vapor pressure when they are mixed that they exert when each is pure. Why does one recover more water in the steam distillation than was calculated? (Hint: Are the organic compound and water truly immiscible?)

In most cases, organic substances have some solubility in water. If this is true, then the amount of water which is required to steam distill the substance in its entirety is the calculated amount plus an amount needed to distill over the amount of substance dissolved in water.

3. Steam distillation is one way to isolate an essential oil from a plant or fruit. Describe two other methods.

Expression (or cold-pressing) is the process of mechanically squeezing the oils out of the source, and is usually used for isolating citrus fruit essential oils. Solvent extraction is the process of treating the source with an organic solvent, such as hexane or supercritical carbon dioxide. The oils dissolve in the organic solvent, and then are isolated by evaporating the organic solvent.

Comment [JMH15]: Answer the post-lab questions here.



Comment [JMH16]: Attach any spectra, chromatograms, etc. at the end of the report. Include experiment number and date on the attachment.