Experiment 4: Steam Distillation of a Volatile Oil from Cloves

Heather Graehl

Tuesday, March 06, 2007
10:08 PM

Title: Experiment 4: Steam Distillation of a Volatile Oil from Cloves

Purpose: To purify eugenol from clove leaves, analyze purity with GC, and obtain a percent yield.

Pre-lab:

Compound	Boiling point	Hazards
Hexane	69 C	Highly Flammable, Harmful, Dangerous for environment
Eugenol	254 C	Harmful

1. What is the theory behind steam distillation (i.e. how does it work and how can it be used to purify and impure organic compound)?

    Steam distillation is used for separating volatile organic compounds from nonvolatile inorganic salts or from leaves and seeds of plants. Steam distillation allows separating substances at lower temperatures which is useful since many organic compounds tend to decompose at high sustained temperatures which regular distillation would require. By adding water or steam to the distillation apparatus, the boiling points of the compounds are depressed, allowing them to evaporate at lower temperatures.

2. What is the theory behind gas chromatography (GC), (i.e. how does it work and how can it be used to determine the identity and purify of an organic compound)?

    A GC has a column which different chemicals pass through at different rates depending on their interaction with the column filing. As the chemicals exit the end of the column, they are detected and identified electronically by absorption. By comparing the GC readout of the compound extracted in laboratory to a commercial grade product, the purity can be determined. If a single compound is supposed to be in the product, it should be represented by a single peak in the readout.

3. What kind of mixtures may be better separated by steam distillation than by simple distillation? Why?

    Mixtures with organic compounds that are sensitive to high heat should be extracted by steam distillation. Steam distillation uses water or steam to depress the boiling points and thus allow separation at lower temperatures. Simple distillation is generally used in two situations: (1) when the last step in the purification of a liquid compound involves a simple distillation to obtain the pure product and determine its boiling point (2) when simple distillation is used to remove a low-boiling solvent from a dissolved organic compound with a high boiling point. In this experiment there is a big difference between the boiling point of the two compounds so simple distillation seems like it could be used by situation 2; however, the low boiling point of hexane make steam distillation a better approach.

4. Consider the possibility that the distillate contained acetyleugenol (a neutral compound) in addition to eugenol (a weak acid). Briefly describe a series of steps that could be used to isolate pure eugenol. You may want to use a flow chart.

    By making the solution acidic the H^+ ions would convert acetyleugenol to eugenol. A strong acid such as HCl could be added to the water prior to steam distillation, but HCl solution has a low boiling point so Cl^- ions would likely get into the product. Instead acetic acid with a

118C boiling point could be added to the solution; however this has a lower Ka value and will not give as many H^+ ions as a strong acid.

5. How do know when you have added enough sodium sulfate to dry hexane?

   When the sodium sulfate appears free flowing and is not clumping.

Procedure:

1. Start sand bath
2. Set up apparatus
3. Weigh 4g ground cloves into small beaker and transfer cloves into 100mL round bottom flask by washing with 50mL of water in small portions.
4. Add 2 boiling stones and mark level of mixture in flask
5. Collect distillate with 25mL round bottom flask
6. Cool collection flask with ice bath
7. Wrap flask and Claisen adapter with aluminum foil but leave opening for marked level of liquid
8. Put heat on high for 15-20 minutes until the level of liquid drops below mark
9. Add hot water through separatory funnel
10. Record temperature during distillation
11. Collect distillate in 25mL flask
12. Stop distillation after 40mL of distillate have been collected and clove mixture is foamed up into Claisen adapter
13. Transfer distillate to separatory funnel
14. Extract distillate twice with 10mL hexane.
15. Combine hexane top layers in Erlemeyer flask. Label it
16. Dry hexane layer using sodium sulfate
17. Get weight of filter flask and record
18. Gravity filter mixture into filter flask
19. Evaporate solvent using Slurpie
20. Weigh filter flask with product
21. Perform GC analysis of isolated eugenol.

Data/Observations:

Day 1

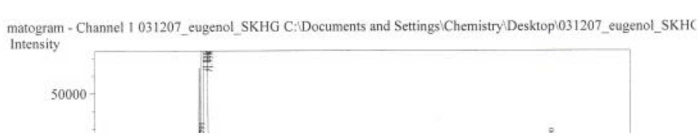
Weight of cloves	4.012g
Reflux time	53 minutes

Figure 1: GC results of synthesized product

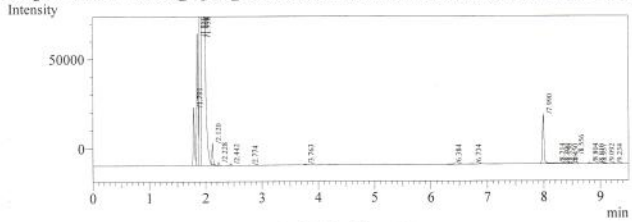
Day 2

Weight of Erlemeyer flask	99.185g
Weight of Erlemeyer flask with product	99.221g
Weight of product	0.0360g

Figure 2: GC results of pure eugenol (supplied for comparison)



matogram - Channel 1 031207_eugenol_SKHG C:\Documents and Settings\Chemistry\Desktop\031207_eugenol_SKHC

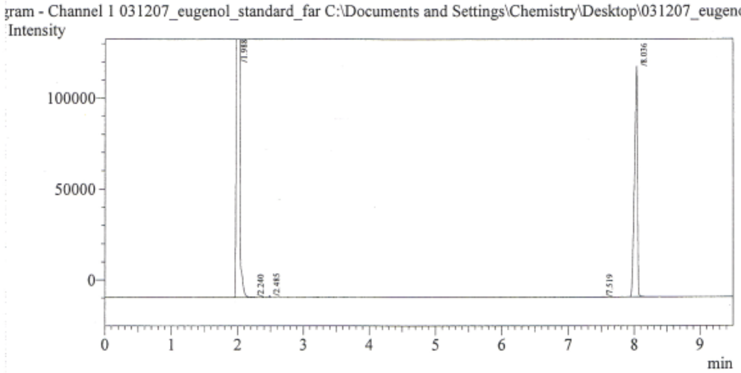


Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Conc.	Unit	Mark	Cmpd Name
1	1.791	52466	31646	0.000			
2	1.856	112864	71792	0.000		V	
3	1.908	1369871	1048664	0.000		VE	
4	1.938	2544806	1057707	0.000		SVE	
5	2.120	15905	11564	0.000		T	
6	2.228	2414	1783	0.000		T	
7	2.442	1428	1022	0.000			
8	2.774	137	66	0.000			
9	3.763	878	627	0.000			
10	6.384	1140	171	0.000			
11	6.734	779	137	0.000			
12	7.990	55711	27596	0.000			
13	8.214	3003	452	0.000		V	
14	8.300	942	321	0.000		V	
15	8.350	1265	294	0.000		V	
16	8.450	1226	218	0.000		V	
17	8.556	13260	5235	0.000		V	
18	8.804	1196	493	0.000			
19	8.910	110	48	0.000			
20	8.957	122	76	0.000		V	
21	9.092	1073	373	0.000			
22	9.234	339	118	0.000		V	
Total		4180935	2260403				

Method - Analytical Line 1

gram - Channel 1 031207_eugenol_standard_far C:\Documents and Settings\Chemistry\Desktop\031207_eugenol_standar



Peak Table - Channel 1

Peak#	Ret.Time	Area	Height	Conc.	Unit	Mark	Cmpd Name
1	1.988	2352261	1059435	0.000		S E	
2	2.240	257	119	0.000		T	
3	2.485	1214	828	0.000			
4	7.519	253	90	0.000			
5	8.036	399886	125621	0.000			
Total		2753871	1186093				

Figure 1: GC results of synthesized product: The major peak at approximately two minutes represents the solvent. The peak at eight minutes represents the eugenol product. There are other very tiny peaks suggest impurities in the product, but their magnitude is infinitesimal in comparison to the two and eight minute peaks.

Figure 2: GC results of pure eugenol (supplied for comparison): There are two distinct peaks at 2 and 8 minutes. Like in Figure 1, two minutes represents the solvent and eight minutes represents eugenol. There are far fewer tiny magnitude peaks as in Figure 1.

Post-Lab Questions:

1. Based on GC analysis of the crude distillate, how does the purity of your eugenol compare to eugenol isolated by dichloromethane extraction of solid cloves. Was steam distillation effective on separating eugenol from other organic components. An GC chromatogram for commercial eugenol will be posted.

    In answering this question, it would have been useful to have a GC of the solution prior to steam distillation as a control. Based on the GC results of my product and supplied GC of pure eugenol, I believe the steam distillation was successful due to the similarities in their GC printouts. I do not believe we had a large amount of product because our distillation was cut short at 53 minutes (90 minutes described in procedure) and the eugenol peak was significantly lower in Figure 1 than Figure 2. There were also impurities in Figure 1, which suggests the distillation was not successful, but the impurities are represented by very tiny peaks. Without comparing to a GC of the solution prior to steam distillation then I cannot be 100% sure, although I predict that the numerous tiny peaks in Figure 1 would have greater magnitude in such a test.

2. Calculate the mass percent yield of eugenol (mass eugenol/mass cloves) that you obtained. How does your value compare with the mass percent of clove oil that has been reported to be present in cloves (14-20%)?

   Mass eugenol/Mass of cloves = .0360g/4.012g = 0.907% yield.

   This percent yield is no where close to the reported percent yield of 14-20%. The main reason our yield was so low is because our distillation was cut short at 53 minutes. If the steam distillation continued longer, more product would be obtained, and a larger percent yield would be calculated.

Conclusion:

When purifying eugenol from clove leaves using steam distillation, I obtained a small 0.907% yield. Clove leaves are reported to contain 14-20% of eugenol while my percent yield is 15 times smaller than this. The most obvious reason for this discrepancy is that the distillation ran for only 53 minutes, because we ran out of time. We had a lot of trouble setting up the apparatus because we had to share a single hood with 5 people within 2 groups. We tried having 3-4 people under the hood simultaneously which significantly slowed us down. If we had more time to run the distillation, our percent yield would have been higher.

Despite the issue of not having time to synthesize sufficient product, the GC showed decent results. There were small impurities in the product, but a clear eugenol peak at eight minutes was observed (Figure 1) despite the small product amount.