

Isolation of the major component of clove oil essay sample



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Pre-Lab Questions:

1.) Briefly explain the concept of steam distillation. What is the difference between a simple distillation and a steam distillation? When a mixture of two immiscible liquids are distilled it is referred to as codistillation. This process is referred to as steam distillation when one of the liquids is water. This distillation is used to separate organic liquids from natural products and reaction mixtures in which the final product results in high boiling residues such as tars, inorganic salts, and other relatively involatile components. It is useful in isolating volatile oils from various parts of plants and not useful in the final purification of a liquid because it cannot separate components that have similar boiling points. The difference between simple distillation and steam distillation is that more water may be added during the distillation during steam distillation. Simple distillation allows a separation between two compounds of 60-70 degrees C or greater, or can be used when separating a liquid from non-volatile solids. Steam distillation is used to distill organic compounds that would decompose during simple distillation. 2.) The major component of clove oil is a compound call Eugenol. One of the minor components is called Acetyleneugenol. Draw the structures of both of these compounds (Wikipedia may be helpful). What would be a good estimate for the pKa of Eugenol?

Eugenol, pKa= 10. 19 at 25 degrees C.

3.) The recovered clove oil will be analyzed by Infrared Spectroscopy. What are the basic principles of Infrared Spectroscopy (can be included in the introduction if you prefer). How will we be able to distinguish between Eugenol and Acetyleneugenol by IR sprectroscopy? Molecule atoms act like <https://assignbuster.com/isolation-of-the-major-component-of-clove-oil-essay-sample/>

they are connected by flexible springs instead of rigid bonds which resemble a ball and stick model. The components of the molecule can oscillate in different vibrational modes. As IR radiation is passed through a sample of pure compound the molecules absorb radiation from the energy and frequency needed to bring about transitions between vibrational ground and vibrational

excited states. The IR spectrum then graphs the amount of IR radiation the compound absorbs in wavelengths. IR spectrometry is mostly used to detect the presence of specific functional groups and other structural features from positions and intensities. We can distinguish these compounds by using the C=O stretching which is obtained at 1700-1735 cm^{-1} given by the acetylenol. Purpose:

The purpose of this experiment is to isolate and identify the major constituent of clove oil. This will be done using steam distillation and infrared spectrometry. Theory: In this experiment my partner and I should be able to isolate an essential oil from ground cloves using steam distillation and extraction. After the distillation and extraction processes an infrared spectrometry sample will be taken to identify the isolated components. Having assembled two apparatus' already for lab this semester this should not be a problem. We will use an apparatus indicated in our lab manual that is commonly used for steam distillation which is similar to the apparatus of simple distillation. Using five grams of ground cloves and fifty milliliters of water we will steam distillate the crushed cloves until we collect a few drops on a watch glass and they appear clear like water and are odorless. Our lab manual indicates that we may have to distill about 150ml of liquids before

this happens. After we have obtained a sufficient amount of clear fluid we will extract the solution twice with dichloromethane then twice again with sodium hydroxide. This solution obtained will be acidified with 10ml of 3M hydrochloric acid. The acidified solution will be mixed one last time with dichloromethane and the final organic Eugenol solution will be obtained. This sample will then be put into an IR spectrometer. Procedures/Data and Observations:

1.) Use a mortar and pestle to grind enough cloves to provide about 5g of the ground spice. Weigh them in a tared weighing dish. My partner ground the cloves the amount of cloves we had was 5.03 grams. 2.) Assemble an apparatus for steam distillation using a large (250-500ml) boiling flask and a steam trap, and have your instructor check your apparatus. Apparatus was successfully assembled. 3.) Combine the ground cloves with 50ml of water in the boiling flask then; steam distill the mixture to obtain the clove oil.

Continue the distillation until a drop or two of the emerging distillate, collected on a watch glass, is odorless and water-clear; with no oily droplets. 150ml might need to be distilled before it becomes clear. Vent the steam line or raise the steam inlet tube above the liquid level in the boiling flask before you turn off the steam. Took about 20-30 minutes for emerging distillate to drop on watchglass and first few drops seemed clear. But we went ahead and placed a flask and started collecting liquid because the process of distilling 150ml of fluid was taking quite some time. A sufficient amount of fluid was collected and checked by Professor. Extraction of Clove Oil After Distillation

1.) Transfer the distillate to separatory funnel. Fluid didn't seem very clear but sufficient to finish our lab on time. 2.) Add 20ml of dichloromethane, gently shake to extract, be sure to vent by opening the stop-cock. First extraction successful. 3.) Let stand of the ring stand, remove the bottom layer to a flask, which is DCM layer and mark as " Org 1". 63. 407g initial flask. Org1: 63. 429g 4.) Add another 20ml of DCM to the sep. funnel containing distillate and do the same as step 2. Step accomplished successfully 5.) Drain the bottom layer to the flask marked as " org 1". Now you have about 40ml of organic layer/DCM in flask " org 1". 63. 558g 6.) Drain the aqueous layer to a beaker and mark as aqueous waste. First waste not to be confused with aqueous 1 that will soon be used. 7.) Transfer the content in " Org 1" DCM solution to the sep. funnel. 8.) Add 15ml of 1M NaOH, gently shake to extract, be sure to vent by opening the stop-cock. Attempt successful 9.) Drain the bottom DCM layer to the same flask mark as " org 1".

10.) Drain the top aqueous layer to another flask and mark as " Aqueous 1". Aqueous 1 to be used 11.) Transfer the DCM layer in " org 1" back into the sep. funnel. 12.) Add fresh 15ml of NaOH and shake to extract while venting. 13.) Drain the bottom layer to the flask marked " org 1". 14.) Drain the top aq. Layer to flask marked " Aqueous 1. Now you have about 30ml of aq. solution. 63. 445g 15.) Acidify the aq. solution in " Aqueous 1" to blue litmus with 10ml or more of 3M HCl acid. Acidification successful 16.) Transfer this content in " Aqueous 1" to sep. funnel and add 15ml of fresh DCM. Extract while venting. Done properly 17.) Drain the bottom layer to a clean flask and label " org 2". 18.) Add another 15ml of DCM to the sep. funnel containing

aq. solution and extract with another 15ml of DCM. 19.) Drain the bottom DCM layer to flask marked " org 2". You have about 30ml of organic/DCM layer now in " org 2". 63. 530g 20.) Add drying agent MgSO₄ to Org 2, filter and decant to remove MgSO₄. 63. 551g 21.) Obtain an infrared spectrum (OP-39) of your clove oil. Turn in your IR spectrum along with the product. Sample was successfully taken and graph produced with help from professor. Results and Conclusions:

Our experiment was executed successfully. We were able to assemble the apparatus and collect enough sample for the IR spectroscopy. We managed to collect enough fluid in a flask to carry out the extraction. The fluid didn't seem as clear as it should have been but obtaining 150ml of fluid was difficult. We were assigned after sometime to just collect as much fluid as we were able to finish the first part of the lab. The extraction process was carried out successfully. All samples were taken properly and mixed accordingly. We did not have any trouble confusing the Organic samples from the aqueous samples. The correct Aqueous 1 sample was used to obtain our final extraction of Eugenol. We obtained a sufficient amount of this sample to use for the IR spectroscopy reading. This part of the experiment was carried out successfully as well with help of our Professor. A graph was successfully produced showing functional groups that of aromatic, alkene, ether, and alcohol.

I feel that I learned to successfully steam distill an organic substance.

Assembling an apparatus for steam distillation was easy because of earlier experiments using simple distillation and fractional distillation. I am not quite sure that I completely understand the IR spectrum produced and how to read <https://assignbuster.com/isolation-of-the-major-component-of-clove-oil-essay-sample/>

it. But I do feel confident that if another sample was needed to use for IR spectroscopy that using the computer and machine to produce it would not be a problem. I am not quite sure that percent yield was calculated correctly.

Percentage calculated was extremely low. Post-Lab Questions:

- 1.) Calculate the percent recovery of clove oil relative to the mass of cloves you started with.
- 2.) Draw a flow chart showing the separation of Eugenol and Acetyleneugenol. Why is aqueous base used to separate the components?
- 3.) Assign the major peaks in the IR spectra of the two components of clove oil.