

Abstract

The purpose of this lab was to determine if there was an efficient way to synthesize vanillin from purified eugenol which was extracted from cloves. Based on our data, we can determine that we were successful in extracting eugenol using various methods. Our GC/MS data was affected by a user error that caused the larger ions to not be shown. However, based on similarity search, we were able to determine that our first peak on the GC/MS was the eugenol we were attempting to extract.

Background

Eugenol (Fig. 1) is an aromatic compound that is found within the oil of cloves. It is used to flavor some teas as well as being used as a home remedy for treating tooth aches. Around 50 years ago, the pure extract of cloves, eugenol, was very expensive. People started buying vanillin instead which was cheaper. Vanillin (Fig. 2) structurally is very similar to eugenol and has similar effects. Different compounds can be oxidized or reduced and can undergo electrophilic aromatic substitution to create vanillin, leading to multiple means of synthesizing it. Extraction of eugenol using diethyl ether is possible because eugenol is non-polar and is soluble in organic solvents.

Discussion

Using both methods 1 and 2 we successfully synthesized Eugenol which we determined from our GC/MS data (Fig. 4 & 5) We found that method 1 worked best for getting a pure solution while method 2 was less pure but yielded a significantly higher amount. While performing method 1, we ran into several issues. In our initial run, the steam bath used to purify our eugenol did not work, our product became solid and crusted on the bottom of the flask. Unable to solubilize it with diethyl ether we were unsuccessful. Thus we had to redo the steam bath with unused distillate. This may have been due to possible contamination or simply overheating. Methods 3 and 4 do work but are time sensitive and potentially dangerous due to the nature of the chemicals used.

Extraction of Eugenol from Cloves

Gunner Williams, Nicole Sachman, Kyle Reitmeier

Data/Results

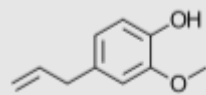


Fig. 1 - Eugenol Structure

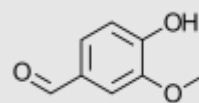


Fig. 2 - Vanillin structure



Fig. 4 – GC/MS data of distilled product (method 1)



Fig. 3 - Steam distillation set up

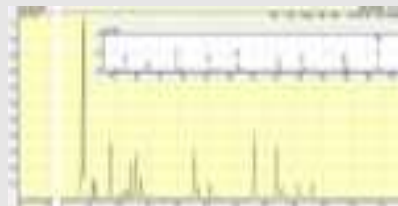


Fig. 5 – GC/MS data of direct extraction product (method 2)

Future Directions

Synthesizing vanillin from eugenol is a lengthy process involving a variety of intermediates, the steps of which are listed in the procedure. Because this is such a lengthy synthesis to perform, an alternate course could be to find a more time efficient and less complex way to undergo this process, perhaps exploring other possible compounds that share similar effects with eugenol, or have a more efficient conversion process.

References

Koenig, A., Anderson, K., Frink, B., & Schuessler, A. (2019). Complete Synthesis of Vanillin from the Eugenol in Cloves: A Project Oriented Guided Inquiry Laboratory Experience.

Procedure

Method 1:

1. Grind up cloves to create powder.
2. Distill clove powder solution and water until about 25 mL of distillate is obtained.(Fig. 3)
3. Transfer the distillate to a separation funnel and extract diethyl ether.
4. Put top layer in an Erlenmeyer flask and add small portions of magnesium sulfate until water is removed from the solution and filter.
5. Put filtered liquid into a beaker and perform a steam bath until there is only 1-2 mL of solution left.

Method 2:

1. Grind up cloves in a large test tube.
2. Add diethyl ether to the flask, and shake vigorously for 30 seconds; extract the diethyl ether using a pipette and collect in a beaker
3. Concentrate the eugenol solution by steam evaporation.

Newer Methods for Synthesizing Vanillin:

Method 3:

1. Sand bathe rhodium(III) chloride, pure ethanol, and eugenol at a sustained 143 degrees Celsius.
2. Filter out spent rhodium catalyst. Transfer diethyl ether-isoeugenol solution into beaker for steam evaporation.

Method 4:

1. Wash the isoeugenol acetate solution with water, saving the organic layer and adding THF to it afterwards.
2. Add a potassium permanganate and water solution to THF and isoeugenol acetate solution gradually over 1.5 hours and filter out brown precipitate.
3. Steam bath the organic layer.
4. Add hydrochloric acid and sodium bisulfite solution to organic layer and heat for 30 minutes.
5. Extract mixture with diethyl ether, then extract vanillin from this mixture by adding sodium bisulfite.