Extraction of Essential Oils from Caraway, Cinnamon, Cloves, Cumin, Fennel, or Star Anise by Steam Distillation

Reading   Experiment 54, 54A, and 54B in Pavia (5th edition). Read all of the Required Reading for Experiment for 54A, especially Techniques 12, 18, 22 and 28.

General

In this experiment you will isolate the essential oils from a spice using the technique known as steam distillation. In the laboratory, a selection of spices will available for you to extract, however you are welcome (even encouraged) to find your own spice. Only extract spices that are dry (not wet or moist). Generally, whole and then gently crushed spices are better than ground. If you choose your own spice, then find the major essential oils that it contains (Merck Index is a good source of this information).

Prelab

In your notebook include **Name**, **Title**, **Date**, **Purpose**, and an **Outline**, plus draw a picture (schematic) of the apparatus that you will use for the steam distillation – this will be shown during lab. See note below about the apparatus.

Procedure

Follow the procedure in the text, but we will use the Hickmann still instead of a regular distillation apparatus. You will periodically collect the distillate in the 5-mL conical vial. Your instructor will demonstrate how to evaporate the methylene chloride in your final dried extract.

To Complete the Experiment – Partial Report

Determine the weight of your product and note its appearance [color, viscosity (thick or thin), solid?] and odor (sweet, acrid, pungent, aromatic, etc.). Calculate the weight percent (*wt%*) of your essential oil:

$$wt\% = \frac{mass\ of\ essential\ oil\,(g)}{mass\ of\ dried\ spice\ (g)}\ x\,(100)$$

Obtain an IR spectrum of your product as described in the text. Analyze and identify the major peaks in your IR spectrum to determine the identity of the major constituents. Perform GC/MS analysis to find the identity of as many constituents as possible. Prepare a table that lists the structures of identified components of your spice in the first column and the IR absorptions and assignments in the second column and in the third column the molecular ion and the base peak from your MS data.