Experiment 6

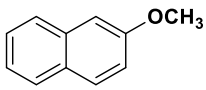
Perfume : The synthesis of Nerolin:

Synthesis of Nerolin

Certain aromatic ethers live up to their name by exuding, pleasant, distinctive odors, making them useful *aroma chemicals* for the formulation of perfumes.

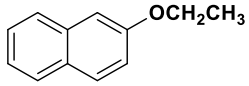
In the seventeenth century, Anna Maria de la Tremoille, introduced a perfume containing oil distilled from orange blossoms.

A major constituent of her *Nerolin oil is* the aromatic ether Nerolin (2-methoxynaphthalene)



Synthesis of Nerolin

Nerolin (new nerolin) : An aromatic ether known as Nerolin II (2-ethoxynaphthalene) is synthetic perfume fixative



Fixative: binds the other ingredients, diminishes the rate of evaporation of the more volatile components.

The **synthesis** of **unsymmetrical** ethers is a common challenge in organic chemistry. In this experiment, **ethyl iodide** and the alkoxide of **β-naphthol** were utilized in an **S_N2** reaction to form ethyl β-naphthyl ether (nerolin) in good yield.

The product could be purified by **recrystallization** from methanol/water and the melting point to be compared to the known value. The results indicate that this approach, a **Williamson Ether synthesis**, is a reasonable method for the synthesis of nerolin.

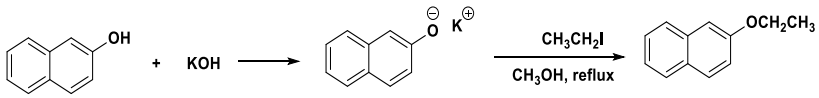
Synthesis of Nerolin

The Williamson ether synthesis is a well-known process for the manufacture of a wide range of ethers.

In our method, β-naphthol will be **deprotonated** by KOH in methanol to form the **nucleophilic species**.

This step is necessary to create a **strong nucleophile** to react with the ethyl iodide.

The enhanced acidity of the aromatic –OH, due to **resonance stability** of the conjugate base, allowed us to perform the reaction in methanol. The overall reaction is shown in the following equation:



Procedure

Ethyl β-naphthyl ether (nerolin).
1- Place 40 ml of methanol, 5.8 g (0.04 mole) of β-Naphthol and 3.3 g (0.05 mol) of potassium hydroxide into a 100 ml round bottomed flask.

2.Shake the contents of the flask for few minutes, then add 3.6 ml (0.045 mol) of ethyl iodide, a couple of boiling chips and attach a reflux condenser. Have your instructor approve your setup.
Boil the mixture for two hours.

3. Cool the reaction mixture to room temperature. Pour it into a 250 ml beaker and add 100 ml of ice cold water. Cool the mixture in an ice bath to effect crystallization of Nerolin.

4. Collect the crystallized Nerolin by suction filtration and recrystallize the product using decolorizing carbon from methanol and water(mixed solvent technique).

5. Collect the dry crystals and determine the weight and the melting point. Calculate the percentage yield of the final product.

Mixed solvent technique

Place the solid sample in a 100 ml beaker and add enough ***hot methanol*** to just dissolve the solid (remember the solution needs to be kept hot also, why?).

To encourage crystallization add a small amount of ***hot*** water until the solution turns turbid.

Add a few drops of ***hot*** methanol to clear the solution.

Allow the solution to cool undisturbed to room temperature, then place the solution in an ice-bath.

Collect the crystals by suction filtration, allowing air to be drawn through the sample by the vacuum to aid in drying of the sample.

Weigh the dry sample; determine the percent yield and melting point of the material.

(If the values are not satisfactory, the sample may be recrystallized again, or decolorizing carbon may be used followed by recrystallization to further purify the compound)

Reflux technique

- This technique involves placing a liquid reaction mixture in a round bottomed flask connected to a condenser.
- The **condenser is open** from the top. Any vapors given off are cooled back to liquid, and fall back into the reaction flask. The round bottomed flask is heated vigorously for the course of the reaction.
- The purpose is to **thermally accelerate** the reaction by conducting it at an elevated temperature (the **solvent`s boiling point**).
- The **advantage** of this technique is that it can be left for a **long period of time without** the need to add **more solvent** or fear of the reaction vessel boiling dry as any vapor is immediately condensed in the condenser.
- In addition, as a given solvent will always boil at a certain temperature, one can be sure that the reaction will proceed at a **constant temperature**. The constant boiling action also serves to continuously mix the solution. This technique is useful for **performing chemical reactions** under controlled conditions that require **substantial time for completion**.

Reflux: The setup