

**Organic –Chem. 221 Lab**

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**Experiment No: (**5)

**Experiment title:** Perfumes: The synthesis of Nerolin.

**Submission date:** 7-5-2021

**Abstract: (including objectives, chemical reactions, methods used and main results)**

Main Objectives:

1. Prepare nerolin through William’s ether reaction.

2. Using reflux distillation method.

3. Calculating the percentage yield of collected nerolin.

Methods used: distillation methods.

**Chemicals:**

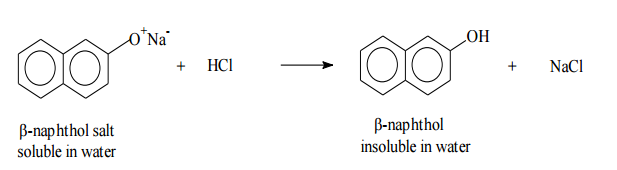
Methanol, β-naphthol, Potassium hydroxide, Ethyl iodide, Ice cold water, Boiling chips, Grease.

**Glassware:**

Round bottom flask, Condenser, Beaker, Glass rod, Buchner funnel, Erlenmeyer flask, Graduated cylinder, Bunsen burner, Funnel, Filter paper, Thermometer, Capillary tube.

**Mechanisms or Reaction:**

* reactions of aspirin:
* reactions of β-naphthol:



**Data:**

Unknown #117

Weigh of original mixture = 6.48g

Mass of filter paper = 0.33g

Table 1: properties of chemicals used

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Name** | **Structure** | **FW (g/mol)** | **Theoretical melting point (ºC)** | **Density (g/cm3)** | **Grams** | **Equivalents** |
| **Aspirin** | Image result for aspirin structure | 180.158 | 135 | 1.4 | 1.4 | 0.0078mol |
| **Β-naphthol** | Image result for Î-naphthol structure | 144.17 | 121-123 | 1.28 | 1.84 | 0.0128mol |
| **Naphthalene** | Image result for Naphthalene structure | 128.1705 | 80.26 | 1.14 | 1.73 | 0.0135mol |
| **Diethyl ether** | Image result for diethyl ether structure |  |  |  |  |  |
| **Water** | Image result for h2o structural formula |  |  |  |  |  |
| **NaOH** |  |  |  |  |  |  |
| **NaHCO3** |  |  |  |  |  |  |

Table 2: results

|  |  |
| --- | --- |
| **Product** | **Mass** |
| **Aspirin** | 2.13 g |
| **Β-naphthol** | 6.14 g |
| **Naphthalene** | 1.78 g |

**Calculation and results:**

* percentage yield of naphthalene:

- Weight of naphthalene= (weight of beaker & naphthalene) - weight of empty beaker

= 99.54- 97.76g = 1.78g

- Percentage yield of naphthalene = ×100%

= 1.73/6.48 \*100% = 94.75%

* percentage yield of aspirin:

- Weight of aspirin = (weight of paper& solid) - weight of empty paper = 2.13g

- Percentage yield of aspirin = ×100%

= 2.13/ 6.48 \*100% = 32.87%

* percentage yield of β-naphthol:

- Weight of β-naphthol = (weight of paper& solid) - weight of empty paper = 6.14 g

- Percentage of β-naphthol =

= 6.14/ 6.48\*100% = 27.47%

* Total percentage yield = 94.75% + 32.87% + 27.47% = 155.09%

**Discussion & Comments:**

As it was mentioned previously, extraction is a process that is used for the separation of two or more substances by using certain solvents. Frequently, the solute is not completely transferred from one solvent to another. In these cases, repeated extractions would be required to transfer the solute from one solvent to the other.

Based on the solvent properties that were mentioned previously, ethyl ether was used as an extracting solvent because it has high solvent power, relative inertness, and low boiling point. Ether has some disadvantages such as its high flammability. Another minor problem is that ether dissolves some water, so the ether solution must be treated with a drying agent which is calcium dichloride (CaCl2) to remove traces of water. A good drying agent should have a high efficiency and a rapid rate of drying.

After obtaining and drying the solid aspirin and β-naphthol, their melting points were measured. Their melting points turned out to be very close to the theoretical melting points as it is mentioned in table #2, which indicates that the samples obtained were almost pure. In addition to that, the total percentage yield that was obtained in the experiment was relatively high (83.4%), and it is close to 100% yield meaning that only a small amount of the three components was lost. The lost amount may be because some of the solid aspirin and β-naphthol adhered to the paper, or because multiple extractions were needed.

Questions:

**Q1.**

Example:10

FA (5 ml ,3 extraction) = (10 / (0.5 \*5 +10))3 = 0.512

FA (15 ml, signal extraction) = 10 / (0.5 \*15 +10)= 0.8

5-mL portions of a solvent give better recovery than a single extraction with 15 mL of solvent.

**Q3.**

because it increases the surface area between the solid drying agent and the liquid solution. The more surface area the two components have in contact with one another, the more they react and precipitate out.

**Q5.**

Naphthalene is actually a solid substance which tends to undergo sublimation due to its high vapor pressure. But when it is kept under vacuum of under an oven then actually, we can store naphthalene for a long time.

This is because air present in vacuum or under the oven does not contain any vapors. Hence, naphthalene does not get consumed.

Thus, we can conclude that if you place naphthalene under vacuum or in an oven for several hours to dry it then we can store it for longer period of time.