Oil of Wintergreen

Purpose: To make methyl salicylate from the esterification of salicylic acid.

- 1. In a long fat test tube (20x180 mm), place 1 gram of salicylic acid and 3 ml of methanol. Mix the reagents by swirling. **Carefully** add 1 ml of concentrated sulfuric acid mixing by swirling after each drop. Take note of any temperature changes. A white precipitate may form. Attach the condenser if you have not already done so.
- 2. Add a boiling stone to the flask and reflux for 1 hour or until you see the mixture get cloudy as a second layer forms. My reaction seemed to take 1.25 hours.
- 3. Allow the mixture to cool to room temperature. (IMPORTANT!) Extract the methanol mixture twice with methylene chloride. (CH_2Cl_2 is not miscible with methanol.)
- 4. Combine the CH_2Cl_2 extractions in either a very large test tube or a small Erlenmeyer flask. Carefully wash the extracts with 3 ml of a 5% NaHCO₃ solution. THERE WILL BE SOME H_2SO_4 in the CH_2Cl_2 .
- 5. Wash the product containing solution with saturated NaCl (or at least 1M NaCl).
- 6. Dry product over Na₂SO₄. Filter. Remove solvent under reduced pressure.
- 7. Check purity using TLC and refractive index. Find % yield.

Postlab Questions.

- 1. This lab involves 2 extractions and three washes.
 - Make a flow chart showing where your product is at each step.
 - Why do we do each of these extractions/washes?
- 2. What is the organic chemistry definition of extraction and wash?
- 3. What is the difference between the two?
- 4. Please write the mechanism for the reaction

