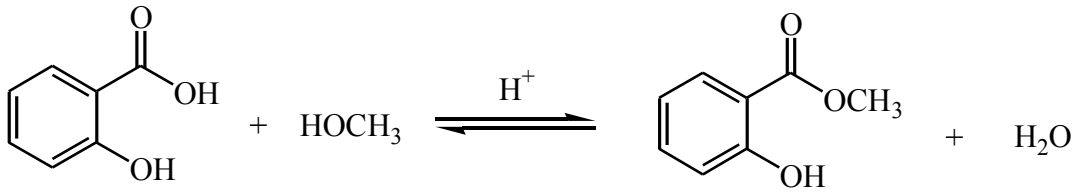
Methyl Salicylate (Oil of Wintergreen)

Reading Experiment 43 in Pavia (5th edition), however use the scaled-up procedure that is provided from the earlier version of Pavia. See the Required Reading for the experiment in the 5th edition. Review the techniques of using a separatory funnel, extraction and vacuum distillation. See the discussion of esterification in Klein, Section 21.10 (particulary) Mechanism 21.6. Also see **Separation Schemes – Tips and Notes**

General

Esterification is a common method of preparing esters. You will use this method to prepare methyl salicylate from salicylic acid. You will use the technique of vacuum distillation to purify the product.



salicylic acid methyl salicylate

Prelab

Include **Name**, **Date**, **Title** of the Experiment, **Purpose**, **Chemical Equation**, a **Reagent Table** (with theoretical yield) and an **Outline** of the procedure.

Prelab Exercise

Prepare a **Separation Scheme** in your notebook that shows how you will isolate the crude methyl salicylate product from the reaction mixture. The scheme should start at the end of the reaction (which is the end of the reflux period) – consider all components of the reaction mixture and how each step of the extraction and drying procedures are designed to remove certain materials and leave others (like your product).

Procedure

Work in pairs. Follow the procedure from the handout. Use caution (gloves would be a good idea) as one of the ingredients is concentrated sulfuric acid (H_2SO_4), which is extremely corrosive. For the *extraction* step, add 10 mL of water to the separatory funnel prior to adding the reaction mixture. You will combine your crude product with other students for the vacuum distillation.

To Complete the Experiment – Partial Report

Record the conditions of your vacuum distillation (temperature range, pressure). Calculate your percent yield based on the number samples that were combined for the distillation. You will perform an IR and NMR analysis of your product – the spectroscopic interpretation of your product needs to be reported in your notebook and a copy of all spectra included with your final report. Also, answer Questions 2, 3, 4 and 5 in Pavia.