

Chemistry II - Laboratory #8 Synthesis of Esters

Goal

The goal of this lab is to synthesize two esters.

Topics to Include in Research

organic chemistry, basic organic nomenclature, esters, esterification, reflux, condenser, separatory funnel, distillation, solvent extraction

Structures to Include in Research

acetic acid, ethyl alcohol, methyl alcohol, ethyl acetate, salicylic acid, methyl salicylate

Background/Introduction

The reaction between an organic acid and an alcohol in the presence of an acid catalyst is called esterification. In this lab, we will perform two esterifications, one between acetic acid and ethyl alcohol, and the other between salicylic acid and methyl alcohol.

To synthesize the first ester, we will combine known amounts of acetic acid, ethyl alcohol, and sulfuric acid. We will then heat them under reflux. Once the reaction is complete and the flask is cool, we will isolate and collect the ester by the technique of solvent extraction. A solution of saturated sodium carbonate will be added to a separatory funnel along with the products of our esterification. Since the saturated sodium carbonate solution is strongly basic (with a pH of around 10), the basic solution will convert any undissociated acetic acid into its salt. By vigorously mixing the basic solution with the reaction products, two layers will form in the separatory funnel: a less dense layer containing the ester, and a more dense layer containing water, sodium carbonate, and other water-soluble substances left over from the reaction. The ester layer is transferred to a flask and purified by distillation. The purified ester is identified by its boiling point and odor. The condensed vapor (distillate) can be massed to calculate the percent yield of the synthesis.

To synthesize the second ester, methyl alcohol, salicylic acid, and sulfuric acid will be combined in a test tube in a water bath and heated. After cooling, distilled water will be added. A distinct odor should result.

NOTE: This is a two-day lab.

Guidelines - Part I (Synthesis of Ethyl Acetate)

1. Use 10 mL of ethyl alcohol, 12 mL of glacial acetic acid, 15 drops of concentrated sulfuric acid, and a boiling stone to a 250 mL round-bottom flask.
2. Heat the flask under reflux (using a water-cooled condenser) in a water bath on a hot plate. The mixture should gently boil for 15 minutes.
3. A saturated solution of sodium carbonate should be prepared by combining 4.5 g of sodium carbonate with 15 mL of distilled water. This mixture should be shaken well.
4. The sodium carbonate solution (not any undissolved solid) should be added to a separatory funnel **IN THE FUME HOOD** along with the cooled contents of the round-bottom flask. The separatory funnel should then be inverted 15-20 times with occasional venting of the stopcock.
5. Once the two layers have separated, the bottom layer should be drained into the

- waste, and the top layer should be transferred into a clean, dry Erlenmeyer flask.
6. The top layer should then be distilled to purify it and record its boiling point.
 7. The results of the distillation will be massed to determine percent yield.

Guidelines - Part II (Synthesis of Methyl Salicylate)

1. Create a hot water bath (water should be about 70 °C) on a hot plate.
2. Use 2 mL of methyl alcohol, 0.70 g of salicylic acid, and 1 mL of 1.0 M sulfuric acid.
3. Use a 13 x 100 mm test tube.
4. Heat the contents of the test tube for about 10 minutes.
5. Once the test tube has been cooled, add 5 mL of distilled water.
6. Use proper chemical smelling technique to carefully note the odor of the product.

Lab handout based on the experiments “Synthesis, Isolation, and Purification of an Ester” in Laboratory Experiments for Advanced Placement Chemistry (Second Edition) by S.A. Vonderbrink (Flinn Scientific, 2006), and “Preparation of Esters” in Heath Chemistry Laboratory Experiments by Ted Hall et al. (D.C. Heath and Co., 1993)