Acetylation of Benzoin with Acetic Anhydride

CHEM HELP *ASAP*

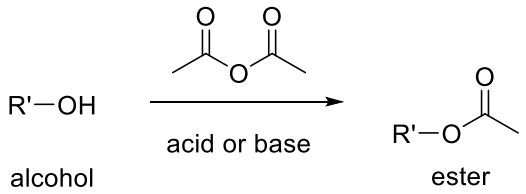
experiment video: https://youtu.be/RUaYQnBgb6c

Purpose

The purpose of this experiment is to demonstrate the acylation of an alcohol with acetic anhydride. The crude product will be isolated by simple filtration and is pure enough to be characterized in its crude form.

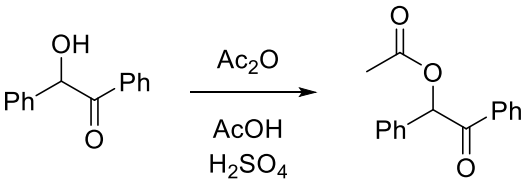
Background

The conversion of an alcohol to an ester is a standard reaction in organic chemistry (Scheme 1). A very common acylation reagent is acetic anhydride. Alcohol acylations to form an ester can be performed under basic conditions, in which the alcohol may be deprotonated to make a stronger nucleophile, or under acidic conditions, in which the electrophilicity of the acylation agent is increased through protonation. Either way, the reaction generally provides esters in high yields.



Scheme 1. Formation of an imine

Benzoin is a molecule that includes an alcohol (Scheme 2). Benzoin readily reacts with acetic anhydride in the presence of acid to give a high yield of the corresponding ester.



Scheme 2. Today's reaction – acetylation of benzoin with acetic anhydride

Procedure – benzoin acetate

(adapted from Corson, B. B.; Saliani, N. A. *Org. Synth.* **1932,** *12,* 1)

Weigh out 5 mmol of benzoin in a 20-mL scintillation vial equipped with a stir bar. Add 1.0 mL of acetic acid to the vial followed by 1.0 mL of acetic anhydride by a pipetter. Add approximately 5 drops of concentrated H_2SO_4, and stir the reaction on a hot plate heated to approximately 100 °C for 20 minutes. Check by TLC for consumption of starting material (mobile phase: 20% EtOAc/80% hexane). If complete, slowly add the reaction mixture via pipet to 17 mL of water in a beaker with rapid stirring. Once the product has separated and solidified, use a spatula to finely divided any larger clumps of solid. Filter the mixture with a Buchner funnel and rinse with water. Spread the product to air dry on a watchglass. Determine the mass of the dry product. Calculate the percent yield. Determine the melting point (melting range – both upper and lower limits) of the product. Take a TLC of the product. Once you finish your TLC, dispose your mobile phase in a waste jug. Interpret the provided NMR spectrum. Record all your observations in your notebook