Bromination of Acetanilide with NBS

CHEM HELP ASAP

experiment video: https://youtu.be/P0rrXP5IPJg

Purpose

The purpose of this experiment is to brominate an aromatic ring. The crude product will be isolated by simple filtration and is pure enough to be characterized in its crude form.

Background

Electrophilic aromatic substitutions require generation of a strong enough electrophile to overcome the stability of an aromatic ring. The classic example of a bromination involves activation of Br₂ with a Lewis acid such as FeBr₃ (Scheme 1).



Scheme 1. Traditional aromatic bromination conditions

Molecular bromine, Br_2 , is difficult to handle and generates irritating vapors. A more convenient, although less reactive, brominating agent is *N*-bromosuccinimide (NBS) (**1**). NBS is able to brominate activated rings in the presence of an acid. The acid protonates NBS and makes the bromine more electrophilic for attack by an aromatic ring (Scheme 2). Acetanilide (**2**) is a relatively reactive ring because the ring bears an electrondonating amide nitrogen.



Scheme 2. Today's reaction – aromatic bromination with NBS

Procedure – 4'-bromoacetanilide

In a 20 mL scintillation vial mix *N*-bromosuccinimide (5.0 mmol) in 10 mL acetonitrile and then add acetanilide (5.0 mmol). Once all materials have dissolved or nearly completely dissolved, add 1 drop of concentrated HCl. Allow the mixture to stir for 20 min. Pour the reaction mixture in a 100-mL beaker with 40 mL water. Rinse the vial to ensure all the solid has been transferred. Filter the mixture through a Buchner funnel with a 125-mL side-arm flask. Seat the filter paper with water. Use water to rinse the filter cake. Spread the collected solid on a tared watchglass, and let the solid air dry. Once the product is dry, determine the mass of the product. Determine your percent yield. Determine the melting point (melting range – both upper and lower limits) of the product. Take a TLC of the product using 50% ethyl acetate/50% hexane as the mobile phase. 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.