Formation of an Imine from an Aldehyde and Amine

CHEM HELP ASAP

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

Purpose

The purpose of this experiment is to synthesize benzalaniline through condensation of aniline with benzaldehyde. The crude product will be isolated by simple filtration and is pure enough to be characterized in its crude form.

Background

Imines are formed through reaction of a primary amine with either an aldehyde or a ketone (Scheme 1). The reaction involves condensation of the nitrogen with the carbonyl followed by loss of water. The overall reaction is reversible with neither side being strongly favored over the other. If an imine is the desired product, then techniques are used to remove the water and prevent the reverse reaction.

$$R^1 = H$$
, aldehyde
 $R^1 \neq H$, ketone
 $R^1 \neq H$, ketone
 $R^1 = H_2 N - R^2 \longrightarrow N^2 H_2 O$

Scheme 1. Formation of an imine

Aniline and benzaldehyde readily react together to afford a high yield of the corresponding imine. The reaction is favorable even if water is not removed because the imine stabilizes the product through providing conjugation of both the aromatic rings.



Scheme 2. Today's reaction – imine formation from aniline and benzaldehyde

Procedure – benzalaniline

(adapted from Bigelow, L. A.; Eatough, H. Org. Synth. 1928, 8, 22)

In a 100 mL beaker weigh 100 mmol of aniline. In a 20-mL scintillation vial weigh 100 mmol of benzaldehyde. Add the aldehyde to aniline with stirring at room temperature. After 15 min add 16-17 mL of 95% ethanol, and stir the reaction for an additional 15 min. Cool the mixture on ice until the entire contents have solidified with precipitate. Cool 50 mL of 95% ethanol on ice. Filter the solid through a Buchner funnel with a 125-mL side-arm flask. Seat the filter paper with ethanol. Use the additional cold ethanol to rinse the beaker as well as the solid in the funnel. Spread the collected solid on a tared watch glass to air dry. 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 using 10% ethyl acetate/90% 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.