

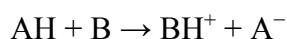
Synthesis and Characterization of a Lewis Acid-Base Adduct

Abstract

Acids and bases, as defined by Lewis in 1923, are electron pair acceptors (acids) and donors (bases). The Lewis definition is more general than previous definitions, removing restrictions on the presence of protons or solvents, such as water. In this lab, a Lewis acid-base adduct was prepared and characterized over the course of two laboratory sessions. An intermediate product was prepared during the first week. In the second week, this product was reacted to form an acid-base adduct. The intermediate and product were both characterized by melting point and IR spectroscopy. The product of the first week's reaction had a higher melting point than could be measured by the melting point apparatus. The final product, after the second week, melted at a much lower temperature, which could be measured. The infrared spectra of both compounds show characteristic peaks for N-H and B-H stretches, as well as other peaks for bending modes in the molecules.

Introduction

Throughout history, there have been several proposed definitions of what makes a chemical an acid or a base. In 1884, Svante Arrhenius proposed a definition of acids and bases which required an aqueous solution. According to the Arrhenius definition, acids dissociate to form hydrogen ions while Arrhenius bases form hydroxide ions in water. In 1923, Johannes Brønsted and Martin Lowry independently defined acids and bases as proton donors (acids) and proton acceptors (bases). The Brønsted–Lowry definition can be generalized as a reaction:



where A⁻ is an acid, B is a base, and H⁺ is a proton.

Gilbert N. Lewis proposed his theory of acids and bases in 1923 in an effort to find more encompassing, generalized definitions. In the Lewis theory of acids and bases, one is concerned only with the donation of electron pairs (bases) or the acceptance of electron pairs (acids). These definitions eliminate the need for proton donors or acceptors (typical of the Brønsted-Lowry definition) or even the presence of solvent (such as water, in the Arrhenius definition). In order to utilize the Brønsted-Lowry theory some sort of protic solvent, often water, is required to be present. The Arrhenius definition of acids and bases rigorously requires the presence of water.

Lewis's definition allows for a ready description of the formation of adducts between two stable compounds in terms of acid-base pairs. In general, one need only place a compound containing a free lone pair of electrons in the presence of a compound that is electron deficient and has an open site in its coordination sphere to produce an adduct.

In this experiment, a Lewis acid-base adduct was prepared by reacting *tert*-butylamine hydrochloride and sodium tetaborohydride (Figure 1). During the first week, the compound *tert*-butylamine was reacted with HCl to prepare the intermediate *tert*-butylamine hydrochloride. During the second week, this *tert*-butylamine hydrochloride was reacted with sodium tetaborohydride to form the final Lewis acid-base product. This compound was characterized by melting point and IR spectroscopy and compared and contrasted to the *tert*-butylamine hydrochloride starting material.

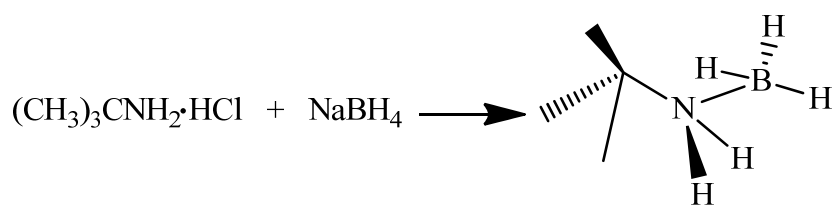


Figure 1. The Reaction to Produce *tert*-Butylamine: Borane Adduct.

Methods

Measure out 10 mL of ethanol in a graduated cylinder. Add this to a 50 mL beaker and insert a stirbar. To this beaker, add 1 mL *tert*-Butylamine. Then, add 12 M HCl to the beaker over a period of 2 m. After 5 m, remove the stir bar. The mixture should be filtered. Wet a filter paper and insert it into a Buchner funnel. Attach the funnel to a side-arm flask and attach the vacuum hose to this flask. Turn on the vacuum and check that the seal between the funnel and flask is tight. Transfer the mixture from the beaker into the funnel and evaporate to dryness using vacuum filtration. Collect the solid (**1**) for analysis and use in the following synthesis. Obtain an IR spectrum using the ATR attachment. First, collect a background spectrum with no sample on the window. Place a small amount of your sample onto the window. Move the arm into position above your sample and lower it. Clean off the plate with acetone and a Kimwipe.

Use the product **1** from the first part to prepare another compound, a Lewis acid-base adduct. Add 0.7 g of the product, 5 mL THF, and a stirbar to a round-bottom flask. Stir this mixture. While it is stirring, add 0.28 g NaBH₄ in small portions over a period of 5 m. Cap the flask loosely with a rubber septum and continue stirring it for 2 h. Then, filter the mixture using vacuum filtration, as before. Make sure that the solid product is evaporated to dryness. Add 10 mL of hexanes to the colorless residue in the beaker and filter the mixture. Wash the residue with 5 mL of hexanes and filter it again; repeat this process (washing it with 10 mL of hexanes total). Leave the product (**2**) out to allow it to dry in the air.

Results

The Lewis acid-base adduct **2** was prepared from NaBH₄ and **1**, which itself was prepared the previous week. The melting points of **1** and **2** were found to be >200°C and 73°C, respectively. The infrared spectra of **1** and **2** are shown in Figures 2 and 3.

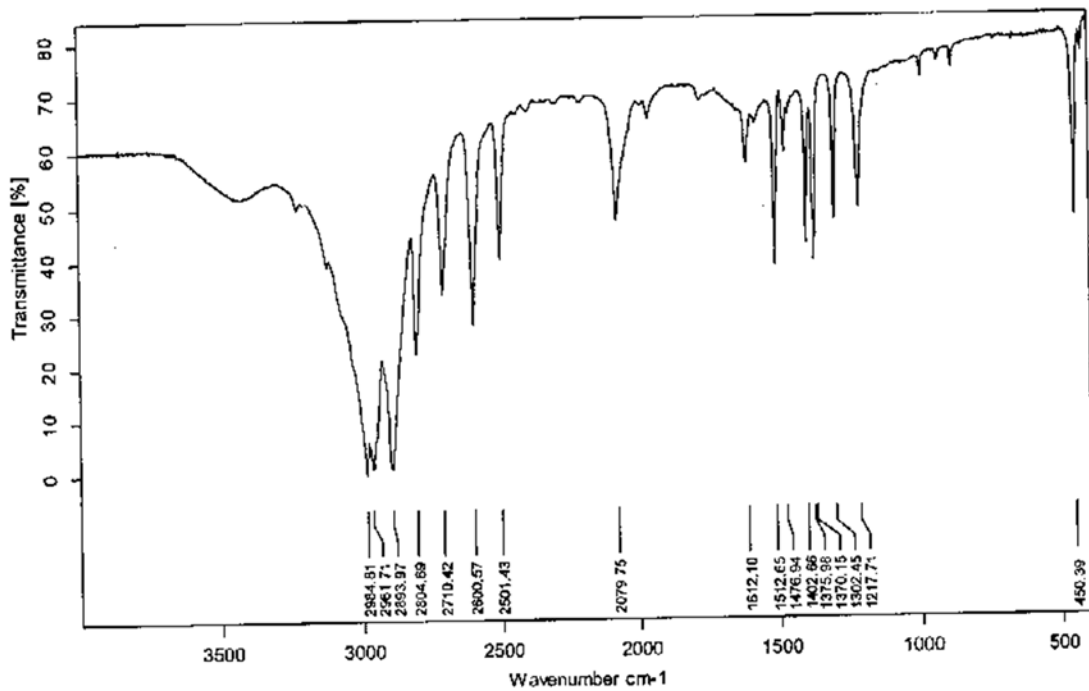


Figure 2.

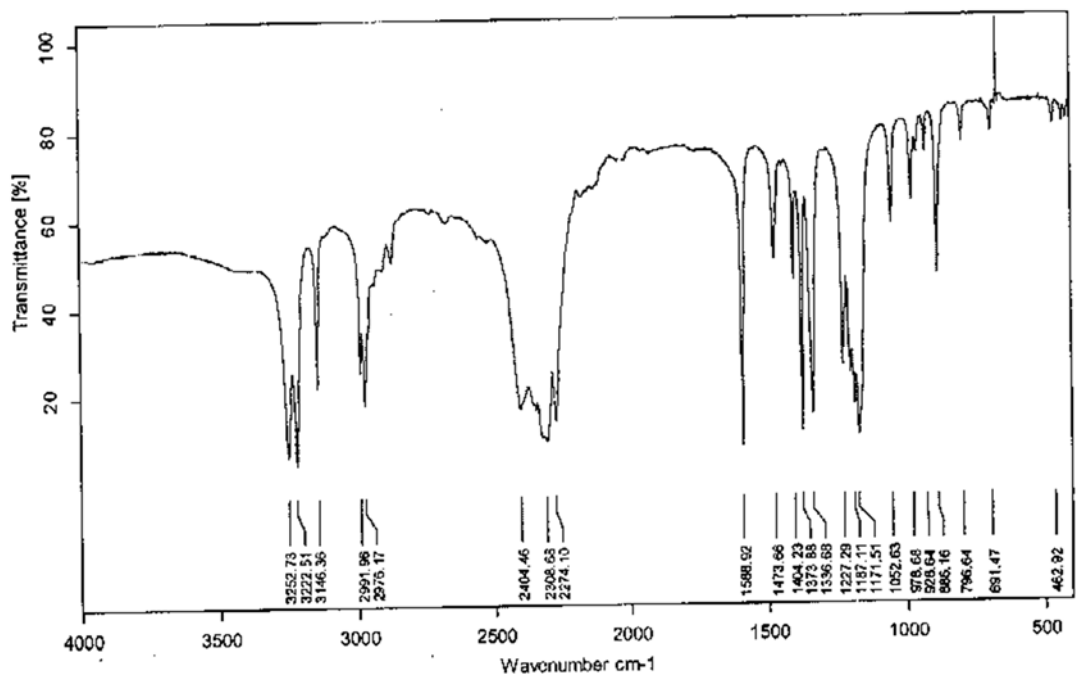


Figure 3.

Discussion

The melting point of **1** is much higher than can be measured by the melting point apparatus, while the melting point of **2** is much lower. The IR spectrum of compound **1** had the following peaks: 2985 (s), 2962 (s), 2894 (s), 2805 (m), 2710 (m), 2601 (m), 2501 (m), 2080 (w), 1612 (w), 1513 (m), 1477 (w), 1403 (m), 1376 (m), 1370 (m), 1302 (w), 1218 (w), 450 (w). For compound **2**, the peaks were: 3253 (s), 3223 (s), 3146 (m), 2992 (m), 2976 (s), 2404 (s), 2309 (s), 2274 (s), 1589 (s), 1474 (w), 1404 (w), 1374 (s), 1337 (s), 1227 (m), 1187 (s), 1172 (s), 1053 (w), 979 (w), 929 (w). N-H stretches are observed in the peaks from approximately 2500-3250. B-H stretches can be observed at a lower energy, about 2250-2400. Other peaks correspond to assorted bends.

Conclusion

A Lewis acid-base adduct was successfully prepared and characterized by IR spectroscopy and melting point analysis.