

Due Thursday: Electrolytic Synthesis Lab Report

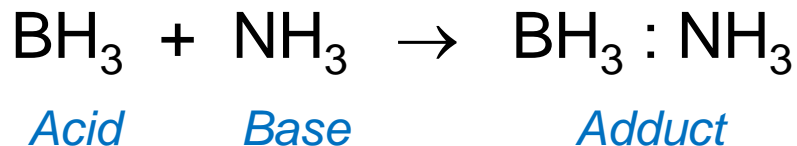
Exp. 3: Borane—Amine Adduct $\text{BH}_3:\text{NH}_2\text{C}(\text{CH}_3)_3$ (Text #4)

Lewis Acids and Bases

Lewis acid: *an electron acceptor*

*Much broader definition
than Brønsted-Lowry*

Lewis base: *an electron donor*



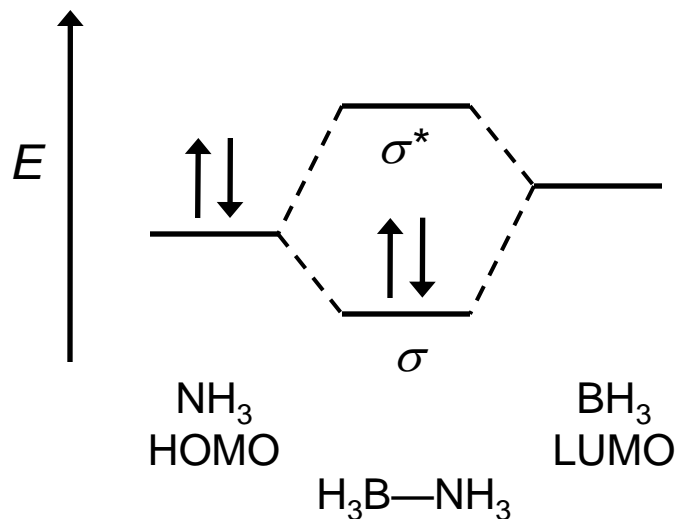
Adduct Formation: Frontier Molecular Orbitals

The key interaction in Lewis acid-base adduct formation may be simply viewed as involving overlap of frontier molecular orbitals on the reactants. (You will model this interaction next week for your reaction.)

Which HOMO and which LUMO must interact?

*The base **donates electrons**. Its HOMO must be involved.*

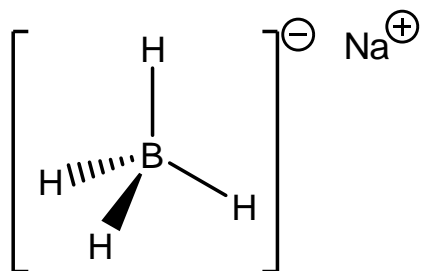
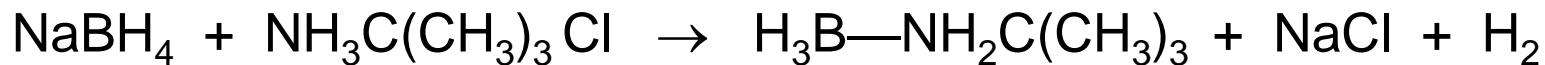
*The acid **accepts electrons** into its lowest lying empty orbital (LUMO).*



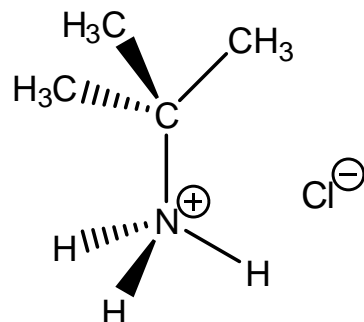
*When two orbitals interact, two new orbitals are formed, the **bonding** and **antibonding** MOs.*

Electrons are placed into MOs according to the same rules for atomic orbitals.

Our Reaction of Interest



metal hydride
("M⁺H⁻")

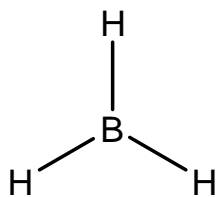


Why use NaBH₄ as a source of BH₃?

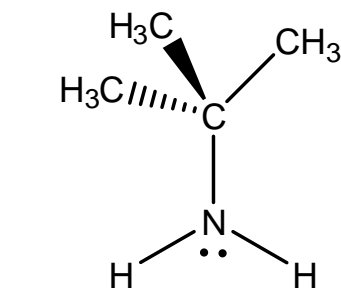
More convenient, less dangerous than B₂H₆

How does this base compare to NH₃?

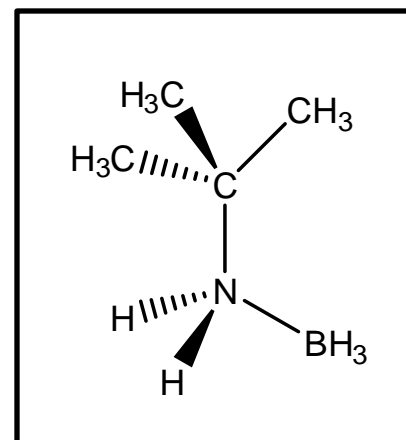
Better e- donor; sterics could be hindering for some reactions.



H₂



NaCl



H₂

NaCl

Product Characterization (to be completed 2/28)

1) Yield and Percent Yield

2) Melting Point

How can you use melting point to qualitatively determine purity?

For a pure substance, complete melting occurs over a narrow range of T.

What if your product does not melt completely below 200° C?

Contamination by NaCl – ionic compounds melt at much higher temps.

3) IR Spectroscopy

What functional groups do you expect?

B-H, N-H, N-C, C-C, C-H, B-N

Which functional group is most diagnostic of product formation?

(You will predict its characteristic frequency via modeling.)

B-N – diagnostic of new boron-nitrogen bond not found in reactants

Procedural Notes

1. We will work at **1/3 SCALE** to minimize waste. You may add slightly more THF if required to dissolve starting materials.
2. $\text{NH}_3\text{C}(\text{CH}_3)_3\text{Cl}$ is commercially available, so we will **not** synthesize it. The synthetic procedure to make the adduct begins on p. 51.
3. **Apparatus:** We will use 100-mL round-bottom flasks with magnetic stir bars. (The mechanical stirrer and drying tube are not required.) Set up **under benchtop hood** (H_2 evolved.)
4. React as long as possible (1.5-2 hrs?), allowing ~30 min. for filtration, etc.
5. **Product recovery:** Your product is **in solution in THF!!** This means that you need to **save the filtrate**. Collected solids may be discarded.
 - a) Make sure your filter flask is clean.
 - b) Use the fritted glass filter. Crystals may pass through filter paper.
 - c) **Do not** allow water to be pulled into filter flask.

Procedural Notes, continued

6. **Product Recovery**, continued: We will use **rotary evaporation** to collect your borane—amine adduct.

Rotate the flask under reduced pressure (often with heating) to evaporate solvent, leaving solid product.

How does reduced pressure help?

Boiling occurs when solvent's $P_{\text{vapor}} = P_{\text{atmosphere}}$. By reducing "atmospheric" pressure, a lower vapor pressure is required, which can be achieved at a lower temperature.

7. **Characterization** – yield, melting point, IR – will be completed next week.

a) Note: For easier yield determination, it will help if you obtain the **mass of your clean, dry round-bottom flask** prior to beginning work this week.

Formal Report Due Thurs., 3/7

You will write a formal report for this experiment, following the general format described previously.

In addition to the borane—amine synthesis, your report will also cover the **related modeling exercises** to be completed next week.