**Reduction of Ketones**

**Introduction**

The main purpose of this lab, is that NaBH4 is being used to reduce benzophenone to benzhydrol (basically reduction of ketones). The process of performing a Fourier Infrared Spectroscopy and reducing carbonyls will be understood and learned.

**Reaction Mechanism**





**Theoretical Yield**

* 1:1 molar ratio, thus 0.0025 moles of benzyhydrol \* 184.23g/mol
  + 0.461g of crude product

**Physical Data**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Compound | Molar mass  (g/mol) | Melting point (degrees C) | Boiling point (degrees C) | Density (g/cm3) | Solubility  (g/L) | Hazards |
| Benzophenone | 182.22 | 48.5 | 305.4 | 1.11 | Insoluble | Toxic, irritant |
| Hydrochloric acid | 36.46 | -114.22 | -85.05 | 1.490 | Soluble | Corrosive |
| Hexanes | 86.18 | -94 | 69 | 0.65 | 0.01 | Flammable |
| Sodium borohydride | 37.83 | 400 | 500 | 37.83 | Reacts with water | Toxic, corrosive, flammable |
| Methanol | 32.04 | -97.6 | 64.7 | 0.79 | Soluble | Toxic, flammable |
| Methylene Chloride | 84.93 | -96.7 | 39.6 | 1.33 | 13 | Toxic, flammable |
| Benzhydrol | 184.23 | 69 | 298 | 1.10 | .05 | Irritant |

*Sources: Handbook for Organic Chemistry,* ***CRC Handbook of Chemistry and Physics*** *(especially Section C: "Physical Constants of Organic Compounds" ), available at the information desk in the Science Library (in Norlin) and in the Organic Chemistry Stockroom.*

***Safety Precautions***

*Sodium borohyride is extremely corrosive and toxic if ingested. If it comes contact with skin, rinse it effectively with water. Methanol, hexanes, and sodium borohydride are flammable. Benzophenone and benzhydrol are irritants, thus avoid skin contact.*

***Wastes***

*Aqueous Waste: The filtrates of the reaction mixture and water washes of the crude product*

*Organic Waste: Hexanes used for recrystallization*

*Solid Chemical Waste: The product, used pipets, filter paper, melting point capillaries and coverslips.*

**Procedure**

1. Weigh out 456 mg of benzophenone and dissolve it in 5mL of methanol in an Erlenmeyer flask.
2. Add the stir bar
   1. Place it over a stir motor and star the stirring.
3. After it is completely dissolved, place it in an ice bath under the flask.
4. Add 142 g of sodium borohydride in two/three portions.
   1. Since its an exothermic reaction, add it slowly in order to keep the mixture from getting too hot
5. When all of sodium borohydride is added, remove it from the ice bath and stir it for another 10-15 minutes.
6. Place it again in an ice bath and slowly add 15mL of 1M HCl.
7. Perform a vacuum filtration, collect the crystals
   1. Rinse them with small amounts of cold water.
8. Determine the Yield and perform a melting point test of the crude product.
9. Collect 100mg of the crude product and purify it with hexanes and again determine the recrystallized yield and melting point.
   1. Compare the melting point of the crude vs purified
10. Run a FTIR spectrum of the product.
    1. Benzhydrol will have to be dissolved in a few drops of methylene chloride, transferred to a salt plate, and then the methylene chloride must be evaportated.