

## Chemistry 244 Organic Laboratory

Schedule for synthesis lab:

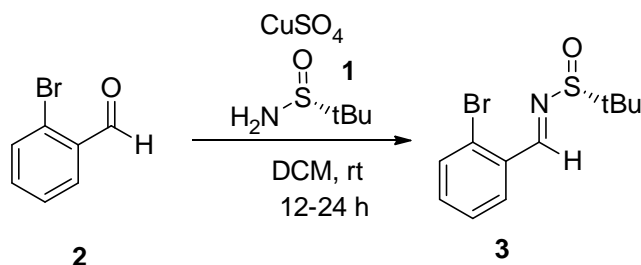
June 22: Set up reaction and pack column;

June 23: Work up reaction and purify product by column chromatography.

### Schiff Base: Synthesis of Enantiomerically Pure N-tert-Butanesulfinyl Imine

#### Introduction

A Schiff base, named after Hugo Schiff, is a compound with a functional group that contains a carbon-nitrogen double bond with the nitrogen atom connected to an aryl or alkyl group. Schiff bases can also be referred to as imines. N-Sulfinyl imines are versatile intermediates in the asymmetric synthesis of many enantiomerically pure organic compounds such as  $\alpha$ -branched amines,  $\alpha,\alpha$ -dibranched amines,  $\alpha$ - and  $\beta$ -amino acids, aziridines, and  $\alpha$ - and  $\beta$ -amino-phosphonic acids. The practical preparation of N-sulfinyl imines is central to their utility in the asymmetric synthesis of chiral amines. In this lab, we use the  $\text{CuSO}_4$ -promoted one-step preparation of the tert-butanesulfinyl imine **3** by the condensation of tert-butanesulfinamide **1** with aldehyde **2**.



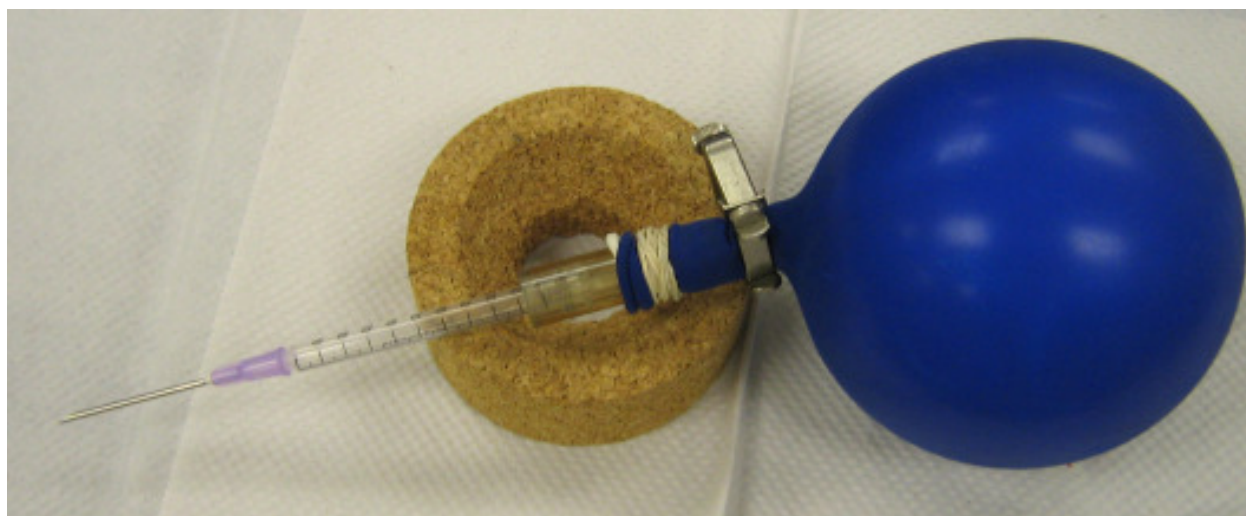
#### Procedure for the Synthesis of tert-Butanesulfinyl Aldimines **3**

To a 0.5 M solution of (R)-**1** (500 mg, 4.13 mmol, 1 equiv) in  $\text{CH}_2\text{Cl}_2$  add 2.2 equiv (1.45 g, 9.08 mmol) of anhydrous  $\text{CuSO}_4$  followed by the aldehyde **2** (1.5 g, 8.2 mmol, 2 equiv). The mixture is stirred at room temperature for 12-24 h. The reaction mixture is then filtered through a pad of Celite, and the filter cake was washed well with  $\text{CH}_2\text{Cl}_2$ . The residue obtained after filtration should be concentrated by rotary evaporation. The resulting product mixture should be purified by chromatography on the second lab period. The final product is transferred to a vial, which should be submitted to your TA for further investigation.

#### Product Analysis

The IR spectra of compounds **1-3** should be collected and compared to show the identity of the functional groups of each compound.

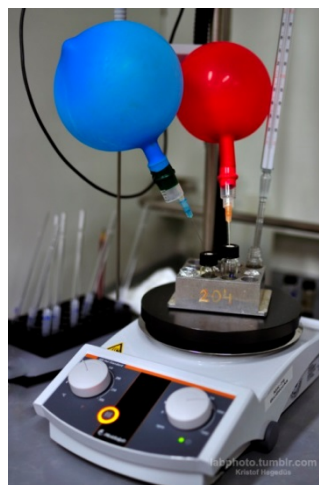
## Set up illustrations



**Figure 1.** Balloons filled with nitrogen gas can be placed on top of a reaction flask to repel moisture and oxygen in the atmosphere.



(a)



(b)

**Figure 2.** (a) Small amount of anhydrous solvent or reagent solution is best transferred by using a syringe. (b) An example of using nitrogen-filled balloons to protect the reaction vials from atmosphere.



**Figure 3.** More examples of using nitrogen-filled balloons to protect the reactions from atmosphere.

### The Report

1. Calculate the theoretical yield of the Schiff base product **3**. Then calculate and record the percentage yield of your product. Submit your product **3** to your TA in a vial labeled with the structure of **3**.
2. Compare and identify the IR peaks present in your product, but was absent in your starting materials, to show you have the desired product.
3. Write a mechanism for the formation of the Schiff base in the presence of  $\text{CuSO}_4$ .