

# Effects of Solvents on the Fischer Esterification

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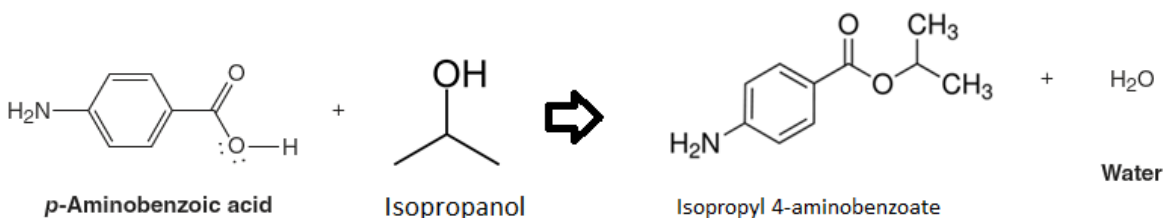
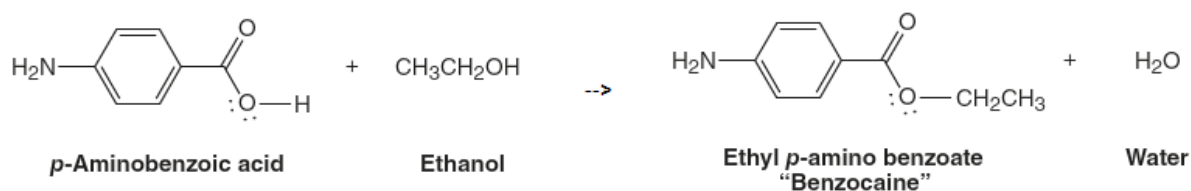
## Introduction

The overall purpose of this experiment was to determine if a “bulky” solvent had any effect on the Fischer esterification reaction. Typically, ethanol is used as the solvent and is known to work very well. For this experiment, we ran two reactions with ethanol and two reactions with isopropanol (which served as our “bulky” solvent) in order to ensure we would have at least one working reaction per solvent, as well as to ensure reproducibility. The effect of the “bulky” solvent on the reaction was determined by comparing yields and time to form a precipitate with that of the ethanol reaction. Our hypothesis was that the isopropanol would work just as well as the ethanol.

The Fischer esterification is good because it is an important reaction in the synthesis of many anesthetics (such as Benzocaine). Having anesthetics in high yield is important as they have many uses in medicine, be it over-the-counter or prescription. In terms of natural synthesis, an acid-catalyzed esterification reaction is actually what causes the production of esters in wine, giving different wines their different flavors (Braymer).

The reaction will utilize reflux prep., vacuum filtration, IR spectroscopy, and melting point analysis to determine if the resulting product matches the intended product, Benzocaine.

## Reaction Scheme



## Experimental

PABA (0.1855g) and ethanol (4.5mL) were placed in a 10.0mL conical flask and a magnetic stirrer was added to stir and help dissolve the solid acid. The PABA completely dissolved in the ethanol. While the mixture was being stirred, 0.15mL of concentrated sulfuric acid was added drop-wise, leading to the formation of a white precipitate. We then attached a water-cooled condenser to prepare for reflux before heating the reaction and letting it gently boil for 60 minutes. The precipitate dissolved, and when the reaction was completed, we detached the reaction apparatus and transferred it to an Erlenmeyer flask that contained 3.0mL of water, which resulted in a clear, liquid solution. This was then cooled to room temperature, at which point we added 10% sodium carbonate solution drop-wise to neutralize the reaction mixture. Sodium carbonate was added until gas evolution had ceased and the pH was greater than 8. At this point, a white precipitate had formed. This precipitate was then filtered using vacuum filtration through a Hirsch funnel. Water was used to aid in transferring the crystals to the funnel. The resulting crude solid weighed 0.0724g. This was then left to set and was characterized during the next lab period, one week later: TLC  $R_F$  = 0.35 (7:3 hexanes/ethyl acetate); m.p. = 88.6 DEGREES C; IR 3419.78, 337.57, 3220.22, 2984.23, 2899.00, 1678.68, 1593.50  $\text{cm}^{-1}$ ; yield = 32.47%.

## Results & Discussion

Both of the initial isopropanol reactions failed to produce a precipitate. In order to attempt to form a precipitate, we ran the experiment one more time using .1800g PABA and 7.0 mL isopropanol instead of the original .1863g PABA/4.5mL isopropanol. This reaction also did not produce any precipitate, so we were unable to characterize product for our isopropanol reactions.

For our reaction with ethanol, both produced a precipitate; however, the product of one reaction was lost so we only characterized one. The IR data from this product showed the presence of sharp, strong peaks at 3419.00  $\text{cm}^{-1}$ , 3311.11  $\text{cm}^{-1}$ , and 3220.60  $\text{cm}^{-1}$  all which indicate the presence of an N-H stretch. There were sharp, weak peaks at 2973.23  $\text{cm}^{-1}$ ,

2929.71  $\text{cm}^{-1}$ , and 2817.17  $\text{cm}^{-1}$  which indicate C-C aromatic stretches as well as  $\text{sp}^3$ -hybridized carbons. Lastly, the presence of sharp, strong peaks at 1680.00  $\text{cm}^{-1}$  indicates the presence of a C=O ester stretch. These peaks all match with expected peaks of Benzocaine. Additionally, the known IR data for Benzocaine looks very similar to the IR spectrum obtained in the experiment.

TLC spotting of the crude solid and the starting material (PABA) was done, as well as a co-spot of both. Results showed an  $R_F$  value of .35 for the crude product, and  $R_F$  value of .16 for the PABA, and two separate spots with  $R_F$  values of .35 and .16 for the co-spot. The separate  $R_F$  values indicate that the product is different from the starting material, and the complete separation of spots in the co-spot indicates that the product is pure. When performing melting point analysis, our crude product melted at 88.6  $^{\circ}\text{C}$ . This melting point matches the known data for Benzocaine, which has a melting point range of 88-90  $^{\circ}\text{C}$ .

## Conclusion

Overall, this experiment is important as it the products can be used in medicines to help treat patients who need local anesthetics. Being able to produce these in high yield can help to make sure there is always medicine available for those who need. The results of the reaction that used the ethanol solvent did appear to be successful; however, the results for the isopropanol reaction were unsuccessful. Given the failure of the isopropanol but the success of the ethanol reaction, our hypothesis was incorrect. In order to avoid this in future experiments, I would re-filter any liquid that remained after the initial filtration. When running this experiment, we only filtered once, which may have led to liquid extract mixed with our precipitate.

## References

*Braymer, Joseph.* Chemistry 215 lecture. University of Michigan. 8 February 2013.

*Reaxsys.* 2.13618.7. Elsevier Properties SA, n.d. Web. 13 Feb. 2013.

*Spectral Database for Organic Compounds SDBS.* National Institute of Advanced Industrial Science and Technology (AIST), n.d. Web. 13 Feb. 2013.