Canizzaro Reaction: The Conversion of p-Nitrobenzaldehyde into p-Nitrobenzoic Acid and p-Nitrobenzyl Alcohol

Ziyue Zhu

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- Introduction

The objectives of this lab were to prepare p-nitrobenzoic acid and p-nitrobenzyl alcohol with p-nitrobenzaldehyde by Cannizzaro reaction. Besides, TLC was used to analyze the products.

Aldehydes contained hydrogens which attached to the α-carbons would have an aldol condensation when met with base solution. Additionally, aldehydes containing no α-hydrogens will occur a self-oxidation-reduction reaction when meet with base solution. 50% of the aldehyde is oxidized into carboxylic acid and another 50% of the aldehyde is reduced into primary alcohol. In this lab, the major reaction refer to Figure 1:

In the first part of this lab was to mix p-nitrobenzaldehyde with KOH solution and to stir the mixture with a magnetic stir bar for 15mins. The mixture was transferred into a centrifuge tube and labeled as 'acid'. Distilled water and dichloromethane was added into the centrifuge tube. The bottom layer was moved into another centrifuge tube labeled 'alcohol'. Dichloromethane was added again into the 'acid' tube and new bottom layer was mixed with the old one in the 'alcohol' tube. The solution in the 'alcohol' tube was washed by distilled water and transferred into a clean test tube. The test tube was labeled as 'alcohol 2'. Na_2SO_4 was added into the 'alcohol 2' tube to dry the dichloromethane. HCl was gradually added into the 'acid' tube. The tube was put into a ice-bath to cool down. Then the crystals were collected by vacuum filtration and washed by distilled water. The crystals were transferred into a Erlenmeyer flask which had methanol in it and were redissolved. After the solution was cooled down, a steam of air was used to reduce the volume of the solution. Petroleum ether was added to precipitate the p-nitrobenzyl alcohol. After the precipitation was finished in the ice-bath, two solutions were prepared with p-nitrobenzoic acid product and p-nitrobenzyl alcohol product by adding acetone and dichloromethane. The second part of this experiment was to analyze the products by TLC and measure the melting points of the products.

- Data

Table 1: The expected yield and actual yield.

Expected yield of Acid	Actual yield of Acid	Error%	Expected yield of alcohol	Actual yield of alcohol	Error%
0.1688g	0.0461g	72.70%	0.1547g	0.1226g	20.75%

Table 2: The melting point range of p-nitrobenzoic acid and p-nitrobenzyl alcohol.

Actual mp of acid	Expected mp of acid	Actual mp of alcohol	Expected mp of alcohol
226^{o}C-231^{o}C	239^{o}C-245^{o}C	75^{o}C-79^{o}C	92^{o}C-94^{o}C

Table 3: R_f value of the products

S	$A_{\text{p-nitrobenzoic acid}}$	$A_{\text{p-nitrobenzyl alcohol}}$	A_{starting}
7.0cm	0.9	4.3	5.2
	R_f of acid	R_f of alcohol	R_f of starting solution
	0.1286	0.6143	0.7429

- Result and Discussion

According to Table 1, the yield of p-nitrobenzoic acid was 0.0461g and the percentage error was 72.70%. Whereas, the yield of p-nitrobenzyl alcohol was 0.1226 and the percentage error was 20.75%. The most possible reason for the error of p-nitrobenzoic acid was that when the HCl was added into the solution, the HCl was poured so quickly that lots of p-nitrobenzoic acid stayed on the centrifuge tube with the foams. The most possible reason for the error of p-nitrobenzyl alcohol was that when the solution was transferred into the flask, some of the p-nitrobenzyl alcohol remained in the test tube with Na_2SO_4.

The Table 2 indicates that the melting points range of p-nitrobenzoic acid was 226°C-231°C which was pretty close to the expected melting points range. The melting points range of p-nitrobenzyl alcohol was 75°C-79°C which is far more away from the expected value. The most reasonable explanation to the error of the melting points range of p-nitrobenzoic acid was the heating button was set to 70 which caused a delay on the reading of the thermometer. Besides, the explanation to the error of p-nitrobenzyl alcohol was that Na_2SO_4 was transferred with the alcohol into the flask. What is more, this can also explain why the error of the yield of alcohol was quite smaller than that of the acid.

The table 3 shows that the R_f values of p-nitrobenzoic acid, p-notrobenzyl alcohol and the starting solution were 0.1286, 0.6143 and 0.7429. This indicates that the acid and alcohol were separated very well and both the acid and the alcohol contained no starting solution.