Discussion:

In this experiment, we performed the synthesis of 4-aryloxybenzaldehyde using nucleophilic aromatic substitution. We were able to produce a small, light brown solid as our product. This experiment went well as we did not mess up much of the procedure. To start, we first acquired 2 mmol of 4-flurobenzaldehyde, which we calculated was equivalent to 5 drops. Alongside this, we acquired .245 g of 4-methoxyphenol and .335 g of potassium carbonate. The potassium carbonate was meant to be in excess, but the recommended amount to use was around .3 g. The potassium carbonate was first added to the test tube, followed by the 4-methoxyphenol, and then the 4-flurobenzaldehyde. After these chemicals were added, 2 mL of DMSO was also added to the test tube. While the chemicals were being obtained, my teammates started the heating process to keep the oil bath between the range of 137 – 145 0C. Once the oil had heated up to an acceptable temperature, we put the test tube in the oil bath. This, however, was not a very good step for us as we had a hard time controlling the temperature range. During the 30 minutes in the oil bath (alongside some time outside to let the oil chill to get in the acceptable range), our oil bath had a range of 1350C – 1520C. A few important observations to note are that within the first minute of being in the oil bath, the solution had become deep purple, and much solid was seen at the bottom. When 9 minutes were left for heating, the solution was much darker, and a brown powder was visible at the bottom of the test tube. Following the 30 minutes of heating, the solution was left out to chill and then put in an ice bath to chill even more. After 8 minutes in the ice bath, water was added to the solution, resulting in a nice pink/light grey layer forming. The test tube contained a solid which had a little trouble at first mixing with the water. The solid mixed fully after 2 minutes with the help of the stir bar. After the solution was well mixed, it was vacuum filtrated. More water was added to the test tube to get as much product out as possible. The final product obtained was a light brown, sand-like solid, which was then used to obtain an IR analysis, NMR analysis, and a melting point analysis.

Our reactants could have undergone 3 possible reactions in this experiment: EAS, NAS addition-elimination, or NAS elimination-edition. As evidenced by the chemical mechanism of this reaction, the aromatic ring of 4-flurobenzaldehyde acts as an electrophile which is attacked by potassium carbonate ions (nucleophile). Therefore, we know that this reaction did not occur via EAS. There is also no strong electrophile/Lewis acid in our reactants such as AlCL3 , which are key indicators of an EAS reaction. Since our reactant includes a halogen (fluorine)/leaving group on the ring, this points to a NAS reaction. Now we must distinguish between NAS elimination-addition and addition-elimination. Our IR spectrum indicated the presence of a C=C ring and an alcohol group. This is not particularly useful as both reactions result in such. On the other hand, our H NMR spectrum displayed the signals of an aldehyde, ether, and para substituted benzene while our C NMR revealed a carbonyl group, CH2, CH3, aromatic carbons, and a C-O bond. These also are useless as they are all seen in both the elimination-addition reaction and the addition-elimination reaction. Our melting point range (42oC-50oC) was lower than the expected values, indicating our product contained some impurities. The impurities can also be seen by our percent yield being greater than 100%. The melting point range would not be a beneficial tool in analyzing which reaction took place as the range not only is close to our product, but also to the melting point range of 4-methoxyphenol (53oC-58oC), which was one of our reactants. Ultimately, the only proof we have that an addition-elimination reaction occurred was the presence of a electron withdrawing group ortho/para to our leaving group. This proves that the product 4-arloxybenzaldehyde was produced via NAS addition-elimination.

Conclusion:

To begin the synthesis of 4-arylbonzaldehyde, we first added our reactants (4-flurobenzaldehyde, 4-methoxyphenol, potassium carbonate & DMSO) to a test tube. The mixture was heated at 140oC for 30 minutes. The reaction mixture was then left to chill in an ice bath. The crude product, a light brown, fine, sandy powder, was finally collected and was used to obtain IR, NMR, and melting point. We had a percent yield of 103.06 % with a melting point range of 42 oC – 50 oC. These are acceptable values as the literature review reports an average yield of 42% with a melting point range of 52oC-59oC and 64oC-65oC. Our IR spectrum revealed the presence of an O-H group, indicating the presence of water or 4-methoxyphenol in our final product. These impurities my have also skewed our obtained melting point range. Overall, this experiment was a success as we fulfilled our objective to perform a Nucleophilic Aromatic Substitution reaction by synthesizing a diarylether and analyzing the reaction product.