The Synthesis of Novel Ionic Liquids

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Synthesis of Ionic Liquids

The general procedure for the synthesis of these compounds was as follows. In the specific case of the synthesis of mono-2-pyridyltri(p-tolyl)phosphonium bistriflimide we combined bromo-pyridine, lithium bistriflimide, and tri(p-tolyl) phosphine in a 1:1:1 molar ratio in a sealed reaction vessel with a nitrogen rich atmosphere. The nitrogen rich atmosphere was necessary because of the highly hygroscopic nature of lithium bistriflimide. The reaction vessel was then placed into a silicon oil bath that was at 170˚C. The time the reaction was allowed to run varied per experiment, but we normally had it in the oil bath overnight.

Once we allowed the reaction to cool from the oil bath we would add a mixture of distilled water and dichloromethane to dissolve the products. Once the products were dissolved, we would separate the water phase and dichloromethane phase of the product with a set funnel as shown in the photo at left.

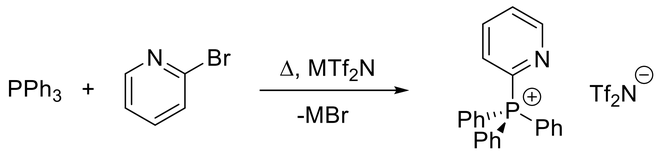
We would then mix the dichloromethane phase of the product with ethanol to pull our final product out of the dichloromethane solution. We then placed this solution into a Roto-evaporator to separate our product from the dichloromethane and ethanol.

Compound	Yield (%)	Melting Point (°C)
[Mopyphos]Tf_2N	82	83-85
^{6pic}[Mopyphos]Tf_2N	54	97-98
^{5pic}[Mopyphos]Tf_2N	63	68-72
^{4pic}[Mopyphos]Tf_2N	53	77-79
^{otol}[Mopyphos]Tf_2N	N/A	N/A
^{ptol}[Mopyphos]Tf_2N	62	69-72
^{OPh}[Mopyphos]Tf_2N	N/A	N/A
[Mopyarse]Tf_2N	**COVID**	**COVID**

This project's results in green

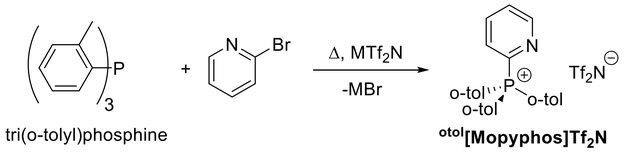
Introduction

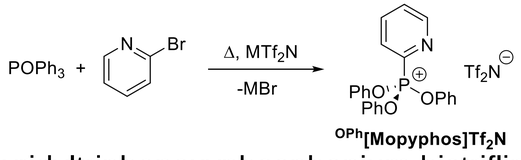
An ionic liquid is a salt that has a relatively low melting point (often below 100ºC). Ionic Liquids have several applications in multiple industries from high-temperature cooling to use as green solvents. Our group has recently developed a method for producing mono-2-pyridyl(triphenylphosphonium) ([Mopyphos]$^+$) salts. The bis-trifluromethylsulfonylimide (bistriflimide or Tf_2N^-) salts of Mopyphos]+ have shown to have melting points low enough to be considered ionic liquids. For this project, we investigated whether this chemistry could be expanded into other groups.



Mono-2-pyridyltri(o-tolyl)phosphonium bistriflimide

The synthesis procedure of this compound was as stated in the synthesis section of this poster with the exception of using tri(o-tolyl) phosphine instead of tri(p-tolyl) phosphine . We attempted this reaction multiple times; however, from the NMR data we gathered it appears we were unsuccessful. We theorized that the methyl groups of the tri(o-tolyl) phosphine may be preventing the reaction.



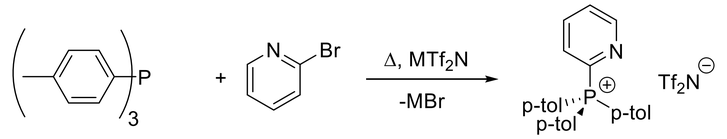


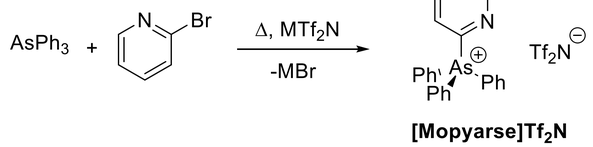
Mono-2-pyridyltriphenoxyphosphonium bistriflimide

The synthesis procedure of this compound was as stated in the synthesis section of this poster with the exception of using triphenyle phosphite instead of tri(p-tolyl) phosphine. According to our NMR data it appears this reaction did not work; however, we are planning to reattempt the experiment at a lower temperature to see if our final product is decaying at the higher temperature.

Mono-2-pyridyltri(p-tolyl)phosphonium bistriflimide

The synthesis procedure of this compound was as stated above. According to the NMR data we gathered it appears that we managed to successfully make this compound.





Mono-2-pyridyltriphneylarsenium bistriflimide

The synthesis procedure of this compound was as stated in the synthesis section of this poster with the exception of using triphenyl arsine instead of tri(p-tolyl) phosphine. Due to the COVID-19 situation we were unable to acquire NMR data for this product; however, when we finished processing this reaction, we were left with a white solid which makes us think this reaction may have worked.

Conclusions

Over the last semester we have managed to synthesize at least two novel ionic liquids. Our next steps are to find the thermal stability of these products and to continue to synthesize more of these ionic liquids to possibly pursue an application of using these compounds as green solvents.

Acknowledgements

I would like to acknowledge my advisor Dr. Benjamin Wicker for his guidance in this research experience.