Progress of the Synthesis of 1-Methylsilatranone

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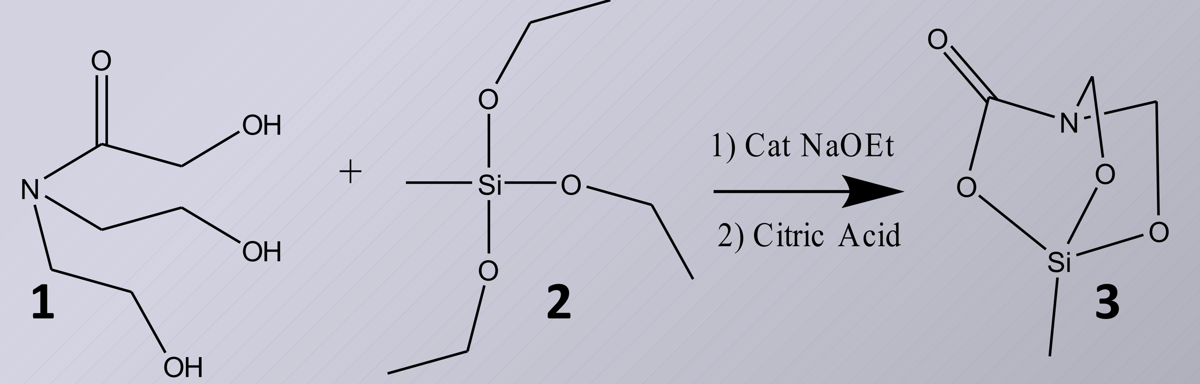
Introduction:

Silatranes have been synthesized and used in many different practical applications such as the making of polymers and adhesives, as well as medicinal applications, including but not limited to anti-cancer, anti-viral, and anti-bacterial agents. In addition, silatranes can be used to help regenerate and reconnect collagen tissue.



Scheme 1. Hypothetical synthesis of 1-methylsilatranone

Little attention has been paid to the nitrogen atom in regards to how it affects the central dative bond. The synthesis of 1-methylsilatranone (**3**) is a very good contribution to understanding silatranes. Resonance may play an important part, with the addition of the carbonyl adjacent to the nitrogen that may create a double bond at the [3.3.3] lactam bridge-head, in changing the strength of the interaction between the nitrogen and silicon.

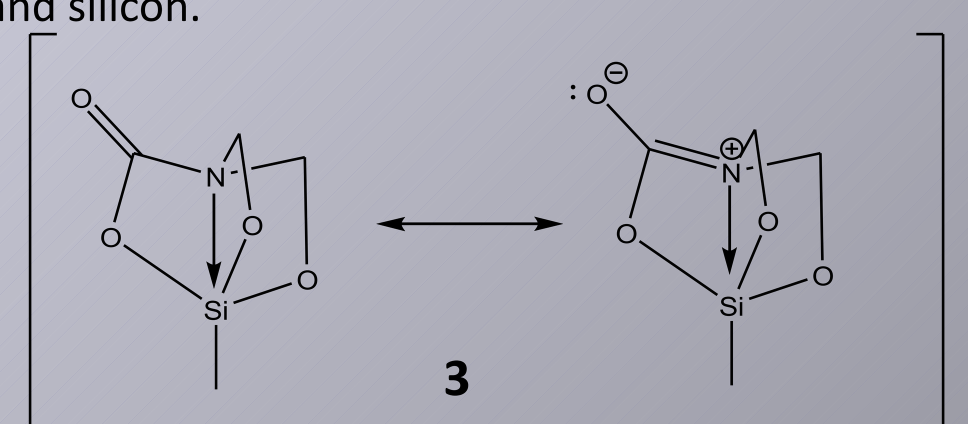
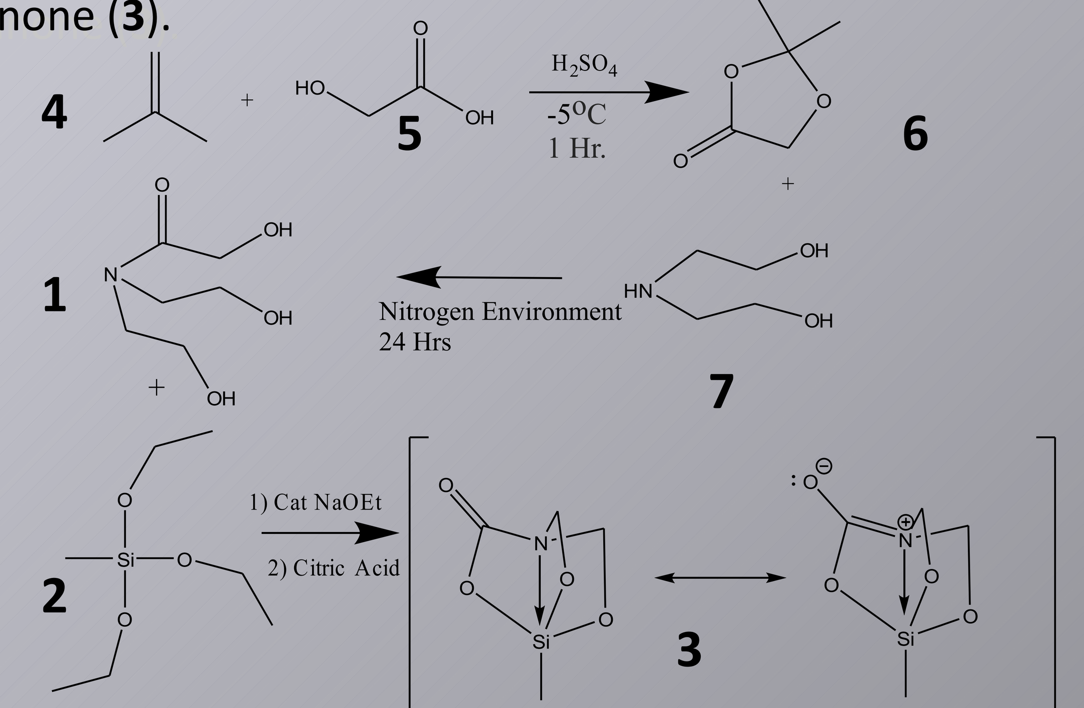


Figure 1. Possible resonance structures of 1-methylsilatranone

Experimental Work:

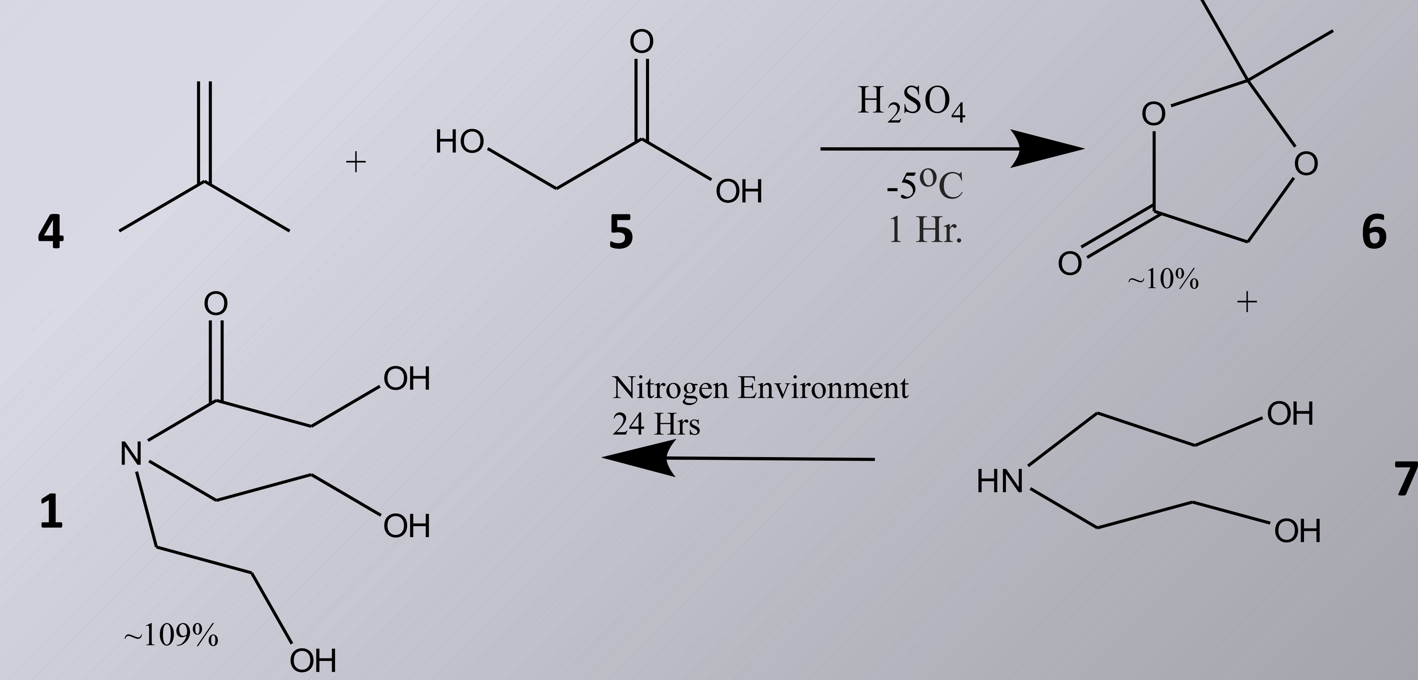
The project began with the synthesis of starting materials with acetone (**4**) and glycolic acid (**5**) to produce glycolic acid acetonide (**6**). This product was then reacted with diethanolamine (**7**) to give N-glycolyl-diethanolamine (**1**). The final step is a reaction with triethoxymethylsilane (**2**) which would provide the target product of 1-methylsilatranone (**3**).



Scheme 2. Proposed synthetic pathway from starting materials to 1-methylsilatranone

Results and Discussion:

Glycolic acid acetonide (**1**) was successfully synthesized from acetone (**4**) and glycolic acid (**5**) in low yield. Next, the glycolic acid acetonide (**1**) was reacted with diethanolamine (**7**) to give N-glycolyl-diethanolamine (**1**). The product was then purified by column chromotography with ethanol, however, not in high enough yield to proceed further with the experiment.



Scheme 3. Synthesis of N-glycolyl-diethanolamine starting material

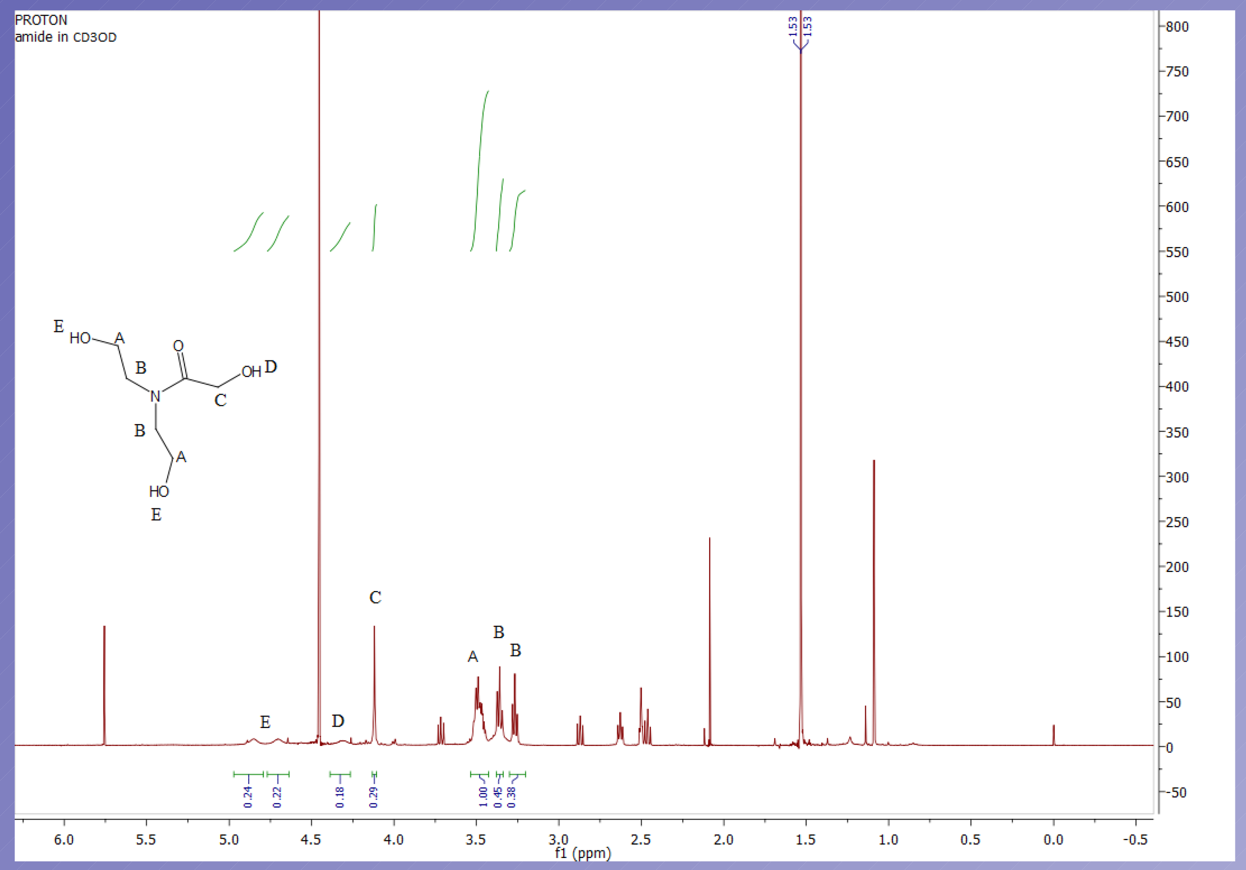


Figure 2. ^{1}H NMR of N-glycolyl-diethanolamine in $CDCD_3OD$

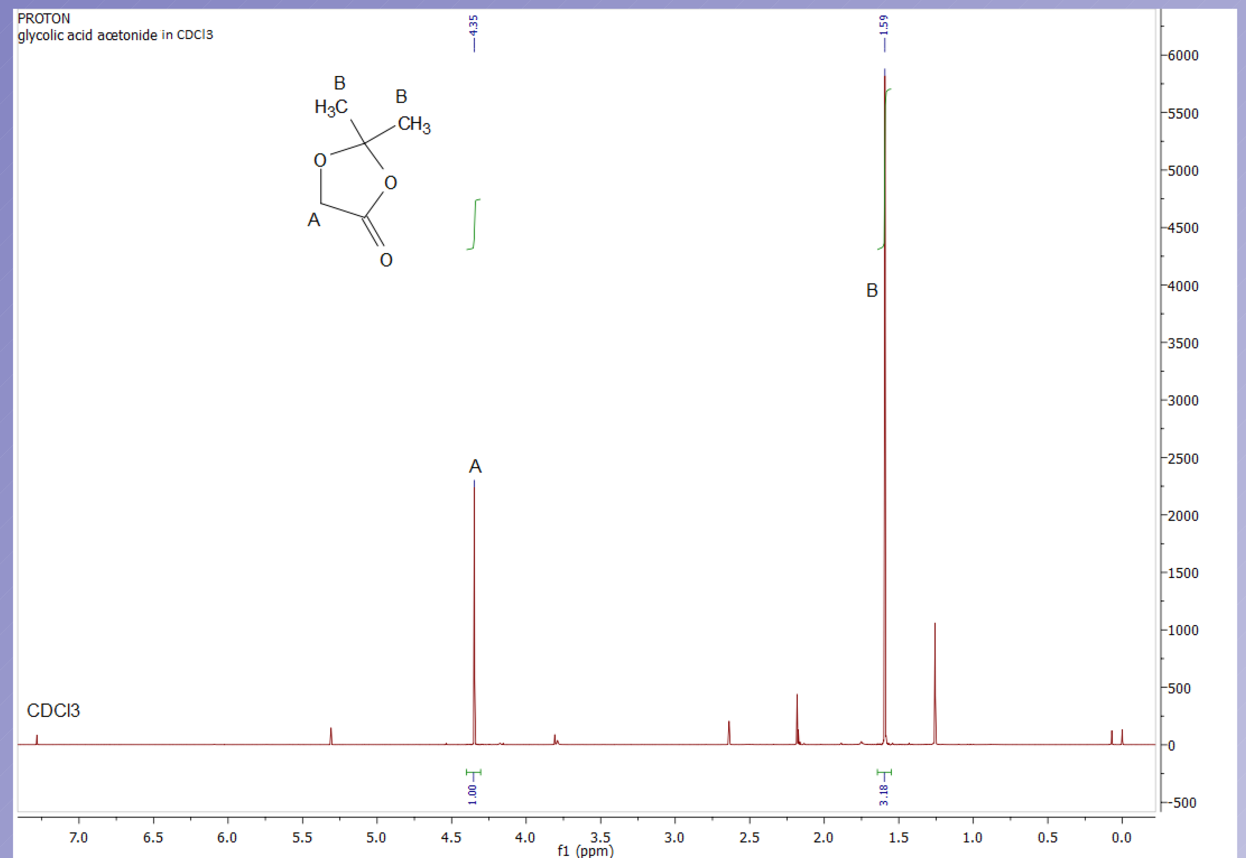


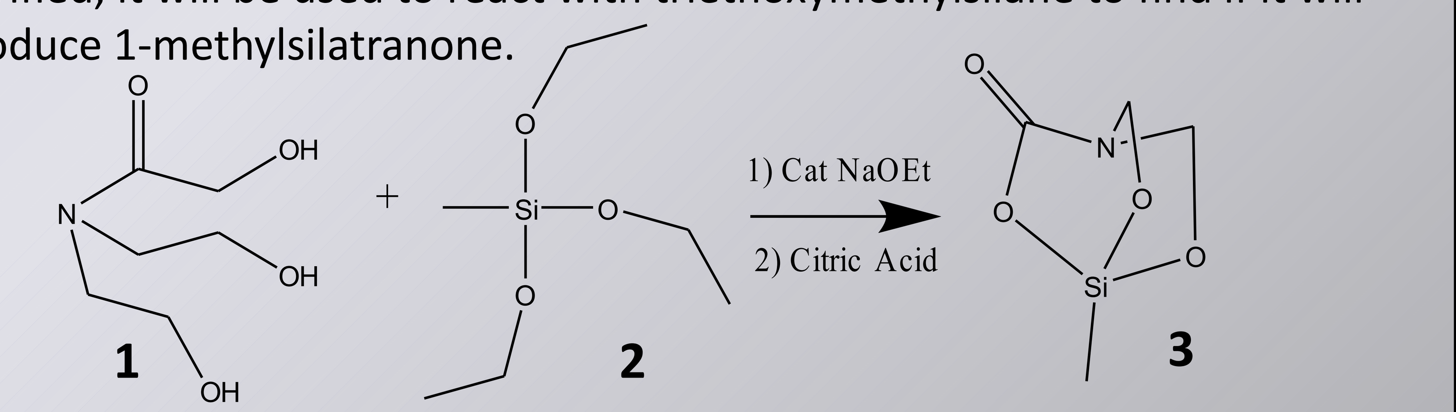
Figure 3. ^{1}H NMR of glycolic acid acetonide in $CDCl_3$

Conclusions:

Although incomplete, the synthesis looks promising. The necessary starting materials have successfully been synthesized and progress has been made toward the final product of the synthesis of 1-methylsilatranone.

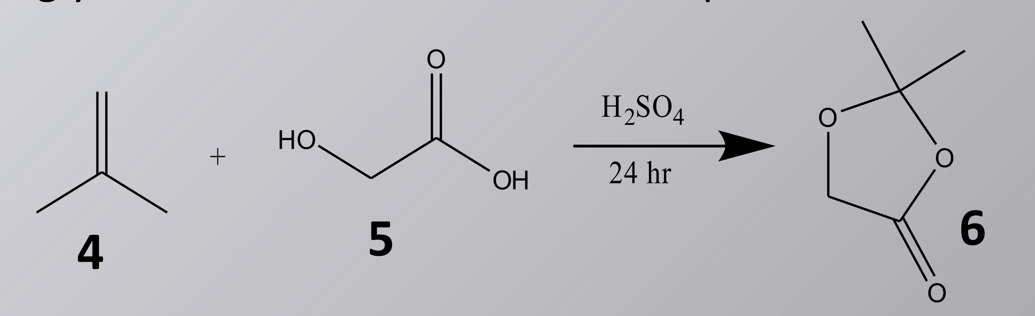
Future Work:

When enough of the pure N-glycolyl-diethanolamine is synthesized and purified, it will be used to react with triethoxymethylsilane to find if it will produce 1-methylsilatranone.



Scheme 4. Future reaction for the synthesis of 1-methylsilatranone

In addition, other reaction environments will be explored for the synthesis of starting materials as the yield was relatively low, especially with the synthesis of glycolic acid acetonide. The subsequent reactions are higher yield.



Scheme 5. Future change in scheme for synthesis of glycolic acid acetonide

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