Synthesis, Comparison, and Nuclear Magnetic Resonance Spectroscopy Analysis of New PPDO-TSC Compounds

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Introduction

This poster will present the synthesis and characterization by Nuclear Magnetic Resonance (NMR) spectroscopy of a series of new thiosemicarbazone molecules. We have successfully synthesized these compounds using α-Isonitrosopropiophenone and an appropriate thiosemicarbazide. These new molecules seem to exhibit different conformations depending upon the reaction conditions. This research has experimented with different reaction procedures in the hope of finding the cleanest and most efficient synthesis route. Our use of NMR has enabled us to determine the most appropriate synthesis procedures. New thiosemicarbazone ligands are important in designing new anti-cancer drugs, so this research is providing new molecules for testing.

Synthesis of PPDO-TSC Compounds

PPDO-ETSC was synthesized two separate times using the same procedure, but two different solvents.

During the first synthesis, .6500g (3.90 mmol) of α-Isonitrosopropiophenone was combined with .5196g (4.40 mmol) of the thiosemicarbazide in a 50mL Erlenmeyer flask. A stir bar, one drop of concentrated sulfuric acid, and approximately 20mL of 50% Ethanol/5% Acetic Acid solution were added and the solution was stirred and heated to 60°C for roughtly 24 hours. A white precipitate was observed. The solid was gravity filtered, dried, and weighed. The dry weight of the PPDO-ETSC product collected was .9480g. This was then repeated using Isopropanol as the solvent. The reaction yielded .8103g product.

PPDO-MTSC was synthesized two separate times using the same procedure as above, with two different solvents. One solvent used was Isopropanol and the reaction yielded .2660g of product. The other solvent was 50% Ethanol/5% Acetic Acid and that reaction yielded .5288g of product.

- ❖ All compounds were analyzed using Nuclear Magnetic Resonance Spectroscopy

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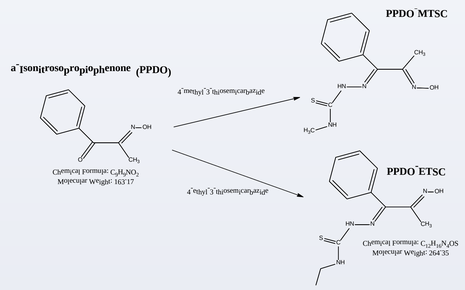

Reaction Scheme

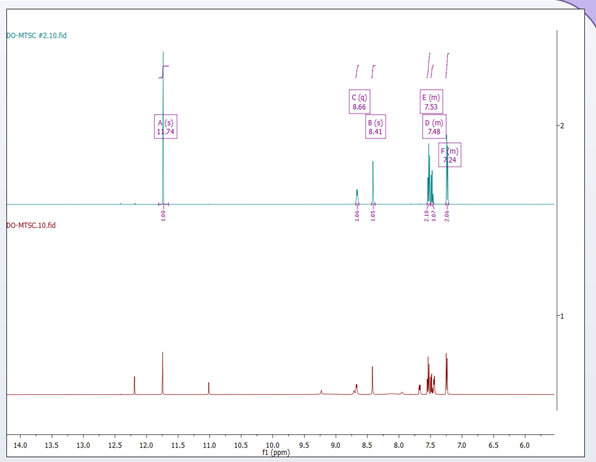

Figure 1

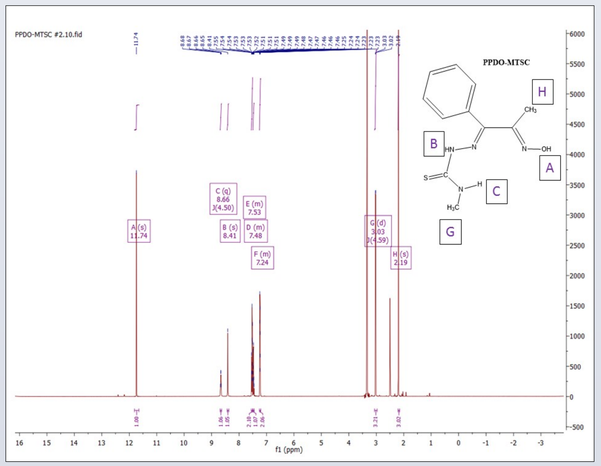

Figure 2

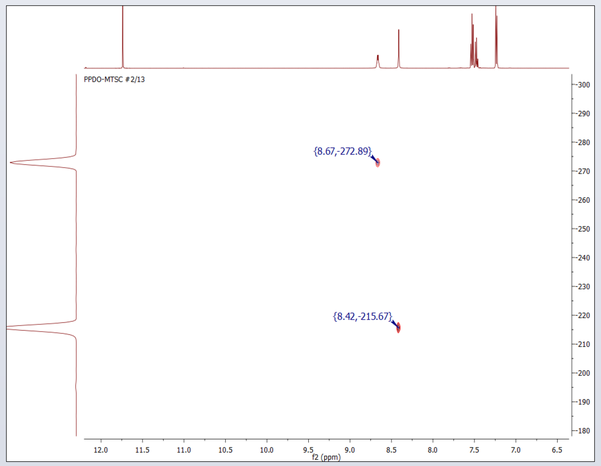

Figure 3

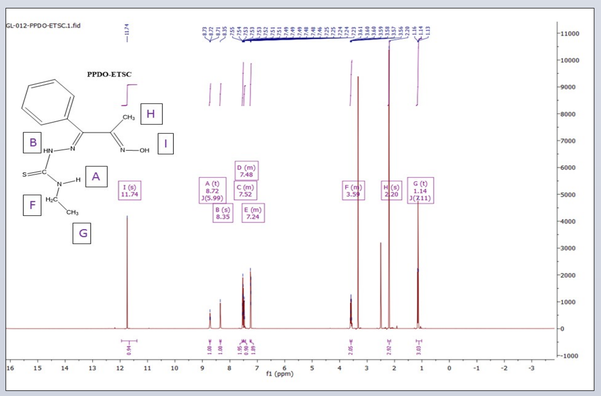

Figure 4

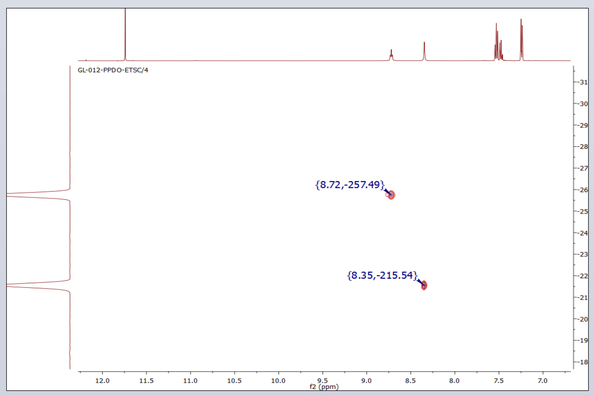

Figure 5

Results and Discussion

After collecting all of the NMR data, I began analyzing and comparing the products in order to determine which procedure and solvent had the best product result. When looking at the MTSC compounds, Figure 1 shows a stacked diagram of the HNMR results from each product. The first MTSC synthesis was messy and did not react well with Isopropanol. The HNMR for this MTSC product is located in the bottom half of Figure 1. After examining the HNMR, we determined that Isopropanol was not the best option for synthesizing this compound. The top half of the HNMR is the PPDO-MTSC product synthesized in 50% Ethanol/5% Acetic Acid. When looking at the NMR data for this product, it was shown to have a much more pure product structure as well as yield more product. Figure 2 shows the HNMR for the PPDO-MTSC in 50% Ethanol/5% Acetic Acid and clearly labels each hydrogen to the corresponding peak on the HNMR. This gives evidence that the structure of the product is in fact what it should be and that 50% Ethanol/5% Acetic Acid is the optimal solvent for this reaction. The HSQC test for PPDO-MTSC in 50% Ethanol/5% Acetic Acid is Figure 3. This shows that the product is consistent with our theory and that the N-H bond is able to resonate, unlike the N-OH bond seen in the structure. Figures 4 and 5 show the HNMR and the HSQC test for the PPDO-ETSC product that was made. Both of these tests give evidence that the theorized structure of our product was correct. The PPDO-ETSC product made in Isopropanol was very messy, similar to the MTSC produced. from it.

Conclusion

In conclusion, the best reaction condition for making these PPDO compounds is synthesizing it in a 50% Ethanol/5% Acetic Acid solvent. Both PPDO-MTSC and PPDO-ETSC reacted the best in this solution and gave us the best product results along with the highest yield of product. I will continue to make new PPDO compounds, as well as further explore these existing PPDO compounds

Acknowledgements

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