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Synthesis and Characterization of a Tris(phenolate)amine Ligand for the Production of an Iron(III)-centered Catalyst

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Introduction

• The use of tris(phenol)amines in the synthesis of iron(III)-based catalysts has been essential to the production of high glasstransition aliphatic and semiaromatic polyesters from epoxides and cyclic anhydrides.¹



- Many aliphatic and semiaromatic polymers can be produced from sustainable resources, which serve as a great alternative to petroleum based products.
- Aliphatic polymers degrade very easily and can be used for a range of medical purposes, due to their high biocompatibility.
- High glass-transition polymers can be used in higher stress environments, such as machinery.

Research Objectives

- Our goal is to optimize the literature synthesis of the catalyst by improving the ligand synthesis considering the following variables:²
 - Time
 - Temperature
 - Stoichiometric ratio
 - Purification methods



Intended Product

Synthesis and Characterization of a Tris(phenolate)amine Ligand for the Production of an **Iron(III)-centered Catalyst**

Caden Pensak, Marie Heidler, Abby Betar, Dr. Ursula Williams **Department of Chemistry** Juniata College

Results

Stoichiometric ratio (phenol:HMTA)	Temperature (°C)	Time (hrs)
8:1	110	24
6:1	130	40
		48

Experiments monitored via ¹H NMR









Intermediate

Purification Methods

- mixtures:

- recrystallization

Conclusions

Future Goals

- synthesize the catalyst

Acknowledgments

We would like to thank Cian Kelly and Kerry Casey for their guidance and introduction to this project, Michael Delosh for his insight and help on our team, and Juniata College for funding.

References

- 6394.

• 4 methods were tested to purify crude reaction

Recrystallization from acetone

Column chromatography in dichloromethane

• Recrystallization in diethyl ether and hexanes

• Trituration in 2-propanol, followed by acetone

• The only method that resulted in pure product at reasonable yields was the trituration technique.

• Increased stoichiometric ratio eliminates the need of a 2nd addition of 2,4-dimethylphenol

• Longer reaction times maximize conversion to product

• Trituration in 2-propanol is a critical purification step.

• Establish a proper internal standard for quantitative NMR

• Use isolated product for future metalation reactions to

• Evaluate the potential of this and related compounds to act as a catalyst in polymer chemistry

(1) Sanford, Maria J., et al. *Macromolecules*, **2016**, 49,

(2) Chandrasekaran, A., Day, R. O.; Holmes, R. R., J. Am. *Chem. Soc.*, **2000,** 122, 1066