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Exploring the Decarbonylation of Cyclic Imides Utilizing a Nickel Catalyst

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INTRODUCTION

Previous research established that the decarbonylative nickel-mediated cross coupling reaction of N-substituted cyclic imides with diorganozinc reagents is possible with a range of phthalimide substitution. The scope of the reaction was further expanded with a wide variety of diorganozinc reagents.

Further research is underway to develop reaction conditions that promote catalyst turnover, and to expand the scope to imides with saturated backbones.

Reaction Scope

The decarbonylation of N-substituted phthalimides proceeds successfully with a variety of substitutions and nucleophiles. In order to widen the scope of the reaction, we are exploring imides with a variety of backbones and phthalimides directly substituted with heteroatoms. Preliminary results indicate that imides with a cyclohexyl backbone will decarbonylate a small amount in toluene using tri-t-butyl phosphine tetrafluoroborate as a ligand.

CATALYTIC DECARBONYLATION

Upon further investigation of the decarbonylation of these cyclic imides, it was found that catalyst turnover is achieved with the use of electron deficient N-substitutions.

Preliminary results:

*conversions determined by GC

MECHANISTIC EXPLORATION

In order to better understand the mechanics of the reaction, specifically the hitch in past attempts to decarbonylate with a saturated backbone, 13C NMR spectra were taken after each component of the reaction was individually added. The reaction was further tracked by in situ IR. Both studies suggest that the Ni does not insert into imides with a cyclohexyl backbone. These findings encourage the use of more sigma donating ligands, such as tri-t-butyl phosphine tetrafluoroborate. Mechanistic studies via 13C NMR and in situ IR are ongoing in order to better support our preliminary findings, and increase our understanding of the unsaturated mechanism.

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