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Attempts at the Synthesis of a Chiral Cycloruthenated Complex of N,N-dimethyl-1-ferrocenylethylamine

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Abstract

Chiral ferrocene-containing ligands which exhibit both central and planar chirality have figured prominently in the area of catalytic stereoselective organic synthesis. In addition, Ru(II) has been shown to be catalytically active in a variety of organic reactions (hydrogenation, oxidation, insertion, metathesis, etc.). In this study, our short-term goal was to prepare a cycloruthenated complex (**3**) of the known chiral ligand, N,N-dimethyl-1-ferrocenylethylamine (**1**). In compound **3**, in addition to the central and planar chirality, the Ru(II) center (being in a pseudo-tetrahedral environment) adds an extra element of chirality. The long-term goal of this project is to investigate the combined effect of these chirality elements on the stereochemical outcome of a series of organic transformations.

In regards to our short-term goal, we have prepared the enantiomerically-pure (*R*)-(+)-N,N-dimethyl-1-ferrocenylethylamine (**1**) based on a published procedure. The ortho-mercurated derivative (**2**) was then prepared as an orange solid by reacting compound **1** with *t*-butyl lithium (in dry pentane) followed by the slow addition of a THF solution of HgCl₂ at -78 °C. The synthesis of the ortho-ruthenated derivative (**3**) was attempted through a transmetallation reaction between compound **2** and the ruthenium(II) dimeric complex [(C₆H₆)RuCl₂]₂. The product mixture contained two distinct Ru-containing species (as indicated by ¹H NMR), one of which showed the placement of Ru at the ortho position of the ferrocene ring. However, no "cycloruthenated" product could be isolated despite several attempts. The isolated Ru complex of **1** is currently being investigated for its catalytic activity in the stereoselective insertion reactions of small molecules (e.g. CO, RN≡C, CH₂=CH₂, SO₂) into the Ru-C bond. Our synthetic efforts, plus the latest results from the latter investigations, will be discussed in this presentation.