

Correction to Synthesis and Structures of Scandium and Lutetium Benzyl Complexes

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In the article, the broad signals observed in the ^1H NMR spectrum of $[\text{Lu}(\text{CH}_2\text{Ph})_3(\text{THF})_3]$, obtained in Edmonton, in $\text{THF}-d_8$ at RT and the two sets of signals at LT were attributed to some fluxional behavior. Subsequently, KS discovered¹ that the actual reason for this was the presence of the ate-complex, $\text{K}[\text{Lu}(\text{CH}_2\text{Ph})_4(\text{THF})_x]$, in the sample. The Edmonton synthesis was repeated several times, but in each case, it was accompanied by the formation of variable amounts of the ate-complex contamination, as determined by ^1H NMR spectroscopy in $\text{THF}-d_8$. Although crystals of $[\text{Lu}(\text{CH}_2\text{Ph})_3(\text{THF})_3]$ were obtained from such preparations, in our hands, pure neutral tribenzyl was best obtained by trituration/extraction of the crude product with toluene,

which gave $[\text{Lu}(\text{CH}_2\text{Ph})_3(\text{THF})_2]$ with two coordinated THF molecules (structure at hand) but with loss of yield. Figure 1 shows the ^1H NMR spectra of pure $[\text{Lu}(\text{CH}_2\text{Ph})_3(\text{THF})_3]$, the ate-complex, and mixture of the two in $\text{THF}-d_8$ at LT; the ^1H NMR spectra of the former two complexes are temperature-invariant except for minor changes in chemical shifts, whereas that of the mixture shows broad signals at RT due to intermolecular exchange.

JT regrets the error and wishes to emphasize that the problem with ate-complex formation was only observed in the Edmonton synthetic protocol and workup procedure. Alternative, large-scale synthesis of $[\text{Ln}(\text{CH}_2\text{Ph})_3(\text{THF})_3]$ ² and most recently including that of $[\text{Lu}(\text{CH}_2\text{Ph})_3(\text{THF})_3]$ has appeared.³

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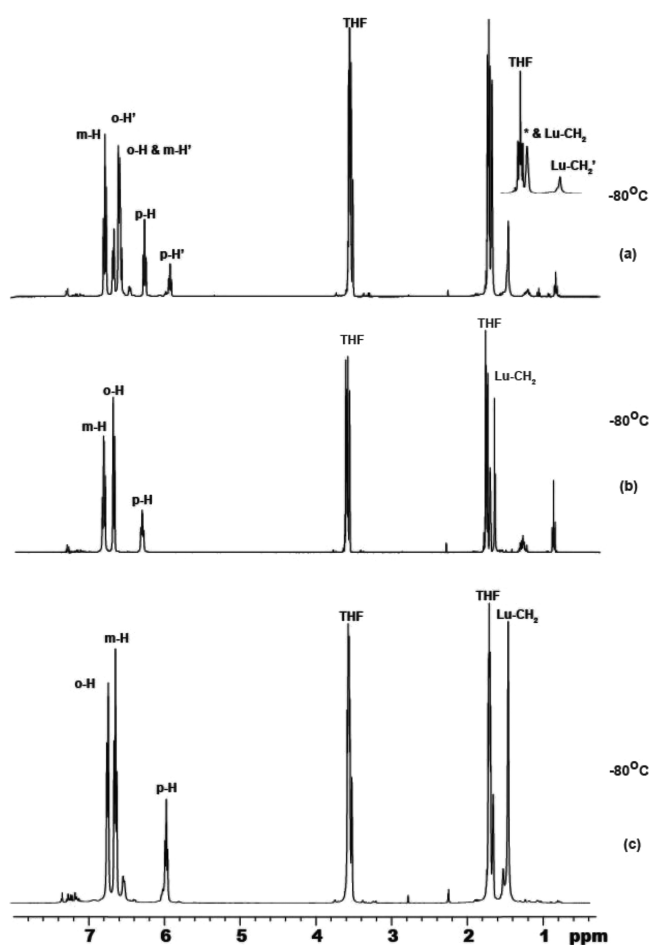


Figure 1. ^1H NMR spectra of (a) mixture of $[\text{Lu}(\text{CH}_2\text{Ph})_3(\text{THF})_3]/\text{K}[\text{Lu}(\text{CH}_2\text{Ph})_4(\text{THF})_x]$ (shown primed), (b) $[\text{Lu}(\text{CH}_2\text{Ph})_3(\text{THF})_3]$, and (c) $\text{K}[\text{Lu}(\text{CH}_2\text{Ph})_4(\text{THF})_x]$ in $\text{THF}-d_8$ at -80°C .

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