

Reactions of Cp_2M ($\text{M} = \text{Ni}, \text{V}$) with dilithium diamido-aryl reagents; retention and oxidation of the transition metal ion†Francesca A. Stokes,^a Mark A. Vincent,^b Ian H. Hillier,^b Tanya K. Ronson,^a Alexander Steiner,^c Andrew E. H. Wheatley,^{a*} Paul T. Wood^a and Dominic S. Wright^{a*}

The reactions of dilithium 1,2-diamidobenzene, $[\text{1,2-(NH)}_2\text{C}_6\text{H}_4]\text{Li}_2$ (L^1H_2) Li_2 and dilithium 1,8-diamidonaphthalene, $[\text{1,8-(NH)}_2\text{C}_{10}\text{H}_6]\text{Li}_2$ (L^2H_2) Li_2 with Cp_2Ni and Cp_2V have been used to obtain the new complexes $(\text{L}^1\text{H}_2)_2\text{NiLi}(\text{THF})_2$ (**3**), $(\text{L}^1\text{H}_2)_2\text{VLi}(\text{THF})_2$ (**4**) and $(\text{L}^1\text{H}_2)_2\text{NiLi}[(\text{L}^1\text{H}_2)_2(\text{L}^1\text{H}_2)_2\text{NiLi}(\text{THF})]^{2-} \cdot 2[\text{Li}(\text{THF})_4]^+$ (**5**), in which retention or oxidation of the initial metal(s) centre is observed. Whereas **3** and **4** contain one transition metal ion within ion-paired structures, **5** has a complicated co-crystalline composition which contains octahedral Ni_6 -cages constructed from six square-planar (16e) Ni^0 centres.

Received 19th June 2013

Accepted 27th July 2013

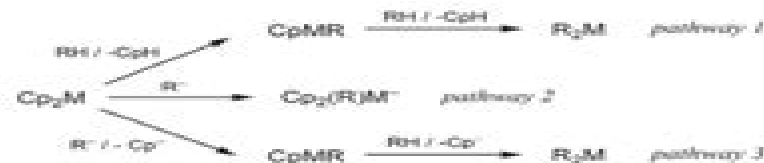
DOI: 10.1039/C3DT51632F

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Introduction

In contrast to ferrocene (Cp_2Fe), in which the Cp–Fe interactions are predominantly covalent, the other first-row metal-lobenes are far more polar. As a result, these species have extensive synthetic utility as sources of the metal atoms in a range of organometallic and metallo-organic compounds. Extensive synthetic studies involving a broad range of N-, O- and C-based organic acids and nucleophiles have identified three main types of reactivity: (i) the deprotonation of organic acids (RH) by the anionic Cp ligands (pathway 1, Scheme 1),¹ (ii) the addition of weaker nucleophiles (R^-) to the transition metal centre (M) without displacement of the Cp ligands (pathway 2),² and (iii) nucleophilic displacement of the Cp ligands by stronger heteroatomic and organometallic nucleophiles (R^-) (pathway 3).³ A common feature of all of these reactions is that the +2 oxidation state of the transition metal ion is maintained throughout, so that Cp_2M has functioned as an organically-soluble source of M^{2+} ions.

The ability to maintain the oxidation state of the transition metal in the course of each of the reactions in Scheme 1 is of particular interest in the formation of multiply-bonded



Scheme 1 Common reactivity patterns of polar metallocenes with organic acids and nucleophiles.

transition metal compounds in which the retention of a low oxidation state is important for the participation of the metal d-orbitals in bonding. Some of our most recent studies have shown that the use of Cp_2M ($\text{M} = \text{V}, \text{Cr}, \text{Mn}, \text{Ni}$) as metal precursors can indeed lead to formally multiply-bonded transition metal compounds. For example, the reaction of $(\text{hpp})\text{Li}$ ($\text{hppH} = 1,3,4,6,7,8\text{-hexahydro-2,2'-pyrimido}[1,2-a]\text{pyrimidine}$) with Cp_2V gives the remarkable complex $[(\text{V}_2(\text{hpp})_4)\text{Li}(\mu^3\text{-Cp})\text{Li}(\mu^3\text{-Cp})\text{Li}[\text{V}_2(\text{hpp})_4]]^{+} \cdot [(\eta^3\text{-Cp})\text{Li}(\eta^3\text{-Cp})\text{Li}(\eta^3\text{-Cp})]^{-}$ in which a $\text{V}=\text{V}$ triply-bonded fragment $\text{V}_2(\text{hpp})_4$ functions as a metal-based Lewis base ligand in the trapping of a $[(\text{V}_2(\text{hpp})_4)\text{Li}(\mu^3\text{-Cp})\text{Li}(\mu^3\text{-Cp})\text{Li}[\text{V}_2(\text{hpp})_4]]^{+}$ cation.² Meanwhile, whereas reaction of the sterically-undemanding lithium amidinate $[\text{MeN}=\text{C}(\text{H})=\text{NMe}]\text{Li}$ with Cp_2Cr has given the classical, lantern-shaped compound $\text{Cr}_2[\text{MeN}=\text{C}(\text{H})=\text{NMe}]_4$, which contains a Cr–Cr quadruple bond,^{3d} the use of the dilithiate of 2,3-diphenyl guanidine $[(\text{PhNH})_2\text{C}=\text{NH}=\text{LiH}_2]$ gives $[\text{Cr}_2(\text{LH})_4][\text{Li}(\text{THF})_4]_2[\text{LiCp}]_2$, which contains a quadruply-bonded Cr^{II} tetranion.⁴

Most recently, however, it was found that the tendency to maintain the metal +2 oxidation state in these reactions may

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† Electronic supplementary information (ESI) available: DFT-coordinates for **3a** and **3b**[†], CCDC 943396–943398. For ESI and crystallographic data in CIF or other electronic format see DOI: 10.1039/C3DT51632F



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 - The Rise of Digital Reading Practical Guide To Cp M Dilithium Press Series On Cp M
 - Advantages of eBooks Over Traditional Books
2. Identifying Practical Guide To Cp M Dilithium Press Series On Cp M
 - Exploring Different Genres
 - Considering Fiction vs. Non-Fiction
 - Determining Your Reading Goals
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 - Popular eBook Platforms
 - Features to Look for in an Practical Guide To Cp M Dilithium Press Series On Cp M
 - User-Friendly Interface
4. Exploring eBook Recommendations from Practical Guide To Cp M Dilithium Press Series On Cp M
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 - Practical Guide To Cp M Dilithium Press Series On Cp M User Reviews and Ratings
 - Practical Guide To Cp M Dilithium Press Series On Cp M and Bestseller Lists
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 - Dealing with Digital Eye Strain
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 - Carving Out Dedicated Reading Time
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 - Distinguishing Credible Sources
- 13. Promoting Lifelong Learning
 - Utilizing eBooks for Skill Development
 - Exploring Educational eBooks
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