

Reactions of  $\text{Cp}_2\text{M}$  ( $\text{M} = \text{Ni}, \text{V}$ ) with dilithium diamido-aryl reagents; retention and oxidation of the transition metal ion†Francesca A. Stokes,<sup>a</sup> Mark A. Vincent,<sup>b</sup> Ian H. Hillier,<sup>b</sup> Tanya K. Ronson,<sup>a</sup> Alexander Steiner,<sup>c</sup> Andrew E. H. Wheatley,<sup>a\*</sup> Paul T. Wood<sup>a</sup> and Dominic S. Wright<sup>a\*</sup>

The reactions of dilithium 1,2-diamidobenzene,  $[\text{1,2-(NH)}_2\text{C}_6\text{H}_4]\text{Li}_2$  ( $\text{L}^1\text{H}_2$ ) $\text{Li}_2$  and dilithium 1,8-diamidonaphthalene,  $[\text{1,8-(NH)}_2\text{C}_{10}\text{H}_6]\text{Li}_2$  ( $\text{L}^2\text{H}_2$ ) $\text{Li}_2$  with  $\text{Cp}_2\text{Ni}$  and  $\text{Cp}_2\text{V}$  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  $\text{Ni}_6$ -cages constructed from six square-planar  $(\text{Li}6\text{e})\text{Ni}^0$  centres.

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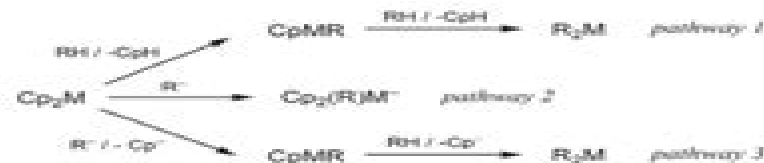
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## Introduction

In contrast to ferrocene ( $\text{Cp}_2\text{Fe}$ ), in which the Cp–Fe interactions are predominantly covalent, the other first-row metalloenes 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),<sup>1</sup> (ii) the addition of weaker nucleophiles ( $\text{R}^-$ ) to the transition metal centre (M) without displacement of the Cp ligands (pathway 2),<sup>2</sup> and (iii) nucleophilic displacement of the Cp ligands by stronger heteroatomic and organometallic nucleophiles ( $\text{R}^-$ ) (pathway 3).<sup>3</sup> A common feature of all of these reactions is that the +2 oxidation state of the transition metal ion is maintained throughout, so that  $\text{Cp}_2\text{M}$  has functioned as an organically-soluble source of  $\text{M}^{2+}$  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  $\text{Cp}_2\text{M}$  ( $\text{M} = \text{V}, \text{Cr}, \text{Mn}, \text{Ni}$ ) as metal precursors can indeed lead to formally multiply-bonded transition metal compounds. For example, the reaction of  $(\text{hpp})\text{Li}$  ( $\text{hppH} = 1,3,4,6,7,8\text{-hexahydro-2,2'-pyrimido}[1,2\text{-}a]\text{pyrimidine}$ ) with  $\text{Cp}_2\text{V}$  gives the remarkable complex  $[(\text{V}_2(\text{hpp})_4)\text{Li}(\mu^3\text{-Cp})\text{Li}(\mu^3\text{-Cp})\text{Li}[\text{V}_2(\text{hpp})_4]]^{+} [(\eta^3\text{-Cp})\text{Li}(\eta^3\text{-Cp})\text{Li}(\eta^3\text{-Cp})]^{-}$  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.<sup>2</sup> Meanwhile, whereas reaction of the sterically-undemanding lithium amidinate  $[\text{MeN}=\text{C}(\text{H})=\text{NMe}]\text{Li}$  with  $\text{Cp}_2\text{Cr}$  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,<sup>3d</sup> 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  $\text{Cr}^{\text{II}}$  tetranion.<sup>4</sup>

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**<sup>†</sup>, CCDC 943396–943398. For ESI and crystallographic data in CIF or other electronic format see DOI: 10.1039/C3DT51632F



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