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Synthesis of bipyridine and biquinoline complexes of group 4 metallocenes

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Abstract

The complexes Cp_2ML_2, with M = Ti, Zr and Hf, and L_2 = bipyridyl, biquinoline and 4,4′-dimethyl-2,2′-bipyridyl, were prepared in excellent yields via magnesium reduction of the respective group 4 metallocene dichlorides in THF solution. ^{1}H NMR spectroscopy in benzene-d_6 indicates that this synthetic route affords $Cp_2Ti(bpy)$ as a diamagnetic product. © 1998 Elsevier Science S.A.

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1. Introduction

The group 4 metallocene moieties Cp_2M (Cp = η^5-cyclopentadienyl; M = Ti, Zr, Hf) have found wide application in organic and organometallic chemistry since their discovery in the 1950s [1,2]. [1] Titanocene has been utilized to convert dinitrogen to ammonia [4] as well as to incorporate nitrogen into various organic carbonyl compounds [5]. In addition to reducing olefins [6] and organic halides [7,8], it has directly converted aldehydes, esters, and 1,2-oxides to alkanes [9].

Recently, intramolecular C–H activation of vinyl titanocene complexes has resulted in highly regioselective methane elimination reactions [10]. Catalytically, titanocene can cyclize enynes which, upon insertion of an isocyanide, leads to the formation of iminocyclopentenes and ultimately to bicyclic cyclopentenones [11].

Zirconocene has also attracted much recent attention because it can cyclize enynes and diynes [12], for example, and can be used to trap otherwise unstable species such as benzyne [13]. A zirconocene-catalyzed system has also been developed in which dienes are reductively cyclized to yield dimagnesium reagents [14],

Herein, we report the general syntheses of group 4 metallocene complexes having biquinoline and bipyridine ligands. These complexes are extremely reactive sources of metallocenes. Commercially-available group 4 metallocene dichlorides are reduced by magnesium in the presence of the bidentate nitrogen ligands to yield extremely air- and moisture-sensitive 18 electron species. We have found $Cp_2Ti(bpy)$ (**1**) to be a diamagnetic species via this synthetic route and its ^{1}H NMR data is reported.

2. Experimental details

2.1. Equipment and techniques.

The syntheses of complexes **1–8** were conducted under argon using standard Schlenk techniques. NMR spectra were obtained using either a Varian FT NMR Unity 300 or a Varian XL-300 spectrometer. Infrared data were

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[1] For a review of the chemistry of titanocene and zirconocene see Ref. [3].

collected with a Nicolet 510 FT-IR Spectrometer. Low-and high-resolution mass spectra (HRMS) were performed at the University of Illinois Mass Spectrometry Laboratory.

2.2. Materials

Solvents used in synthesis were purified by distillation from sodium benzophenone (THF, Et_2O) or calcium hydride (hexanes, toluene) under argon immediately prior to use. Commercially-available titanocene dichloride, zirconocene dichloride, hafnocene dichloride, 2,2′-bipyridine, 2,2′-biquinoline, and 4,4′-dimethyl-2,2′-bipyridyl (Aldrich) were used without purification. Grignard-grade magnesium turnings were dried at 150°C and crushed under an argon atmosphere immediately prior to use.

2.3. Syntheses of complexes

2.3.1. $Cp_2Ti(bpy)$(1) [15,16]

Cp_2TiCl_2 (1.0 g, 4.0 mmol) and 2,2′-bipyridine (0.63 g, 4.0 mmol) dissolved in ca. 100 ml of THF were treated with freshly-crushed magnesium turnings (0.5 g, 20 mmol). An immediate reaction ensued and the solution gradually darkened to a deep-green. After stirring for 10 h the solvent was removed in vacuo. The residue was extracted with ca. 200 ml of hexane, the solution was filtered and concentrated until solid began to deposit (ca. 50 ml). The solution was cooled to -78°C and complex **1** was deposited as extremely air-sensitive green–black needles (1.2 g, 87%). ^{1}H NMR (C_6D_6, 20°C): δ5.49 (10 H, Cp), 7.67, 7.20, 7.10, 6.42 (8H, bpy). Mass spectrum (70 eV) m/e 334 (M^+, 74%), 269 (M^+- Cp, 10%), 178 (Cp_2Ti^+, 100%), 156 (bpy^+, 5%).

2.3.2. $Cp_2Zr(bpy)$ (**2**) [17,18]

A solution of Cp_2ZrCl_2 (0.50 g, 1.7 mmol) and 2,2′-bipyridine (0.27 g, 1.7 mmol) in ca. 75 ml of THF containing magnesium turnings (0.16 g, 6.6 mmol) was stirred magnetically under argon at room temperature. The solution gradually darkened, eventually turning dark purple. After 15 h the THF was removed in vacuo. The deep purple residue was dissolved in hexane, filtered through 1 cm of anhydrous sodium sulfate in a Schlenk fritte and the solvent was concentrated in vacuo. Cooling to 20°C caused **2** to deposit as violet–black crystals (0.53 g, 83%). ^{1}H NMR (C_6D_6, 20°C): δ7.41 (d, $J=9.0$ Hz, 2H, bpy), 6.93 (d, $J=9.0$ Hz, 2H, bpy), 6.31 (dd, $J=6.0$ Hz, 2H, bpy), 5.40 (s, 10H, Cp), 5.35 (dd, 2H, bpy) ^{13}C NMR (C_6D_6, 20°C): δ115.8, 121.2, 125.6, 136.5, 148.1, 149.2. IR (KBr, cm^{-1}): 1585 (s), 1456 (s), 1263 (s), 1094 (br), 1023 (br), 866 (w), 803 (s), 757 (s), 622 (w). Mass spectrum (70 eV) m/e 376 (M^+, 100%), 220 (Cp_2Zr^+, 91%), 156 (bpy^+, 51%). HRMS (m/e) calcd for $C_{20}H_{18}N_2Zr$: 376.051707. Found: 376.051200.

2.3.3. $Cp_2Hf(bpy)$ (**3**)

The reaction was run similar to the procedure for **2**. An etheral solution was filtered and the solvent was removed in vacuo. Complex **3** was obtained as dark purple crystals (0.43 g, 72%). ^{1}H NMR (C_6D_6, 20°C): δ7.20 (d, $J=6.0$ Hz, 2H, bpy), 6.82 (d, $J=9.0$ Hz, 2H, bpy), 6.19 (dd, $J=6.0$ Hz, 2H, bpy), 5.35 (s, 10H, Cp), 5.29 (dd, $J=9.0$, 6.0 Hz, 2H, bpy). ^{13}C NMR (C_6D_6, 20°C): δ103.5, 124.1, 126.3, 126.6, 141.7, 146.4. The extreme sensitivity of this compound prevented accurate elemental analysis.

2.3.4. $Cp_2Ti(biq)$ (**4**)

2,2′-Biquinoline (0.51 g, 2.0 mmol), Cp_2TiCl_2 (0.50 g, 2.0 mmol), and freshly-crushed magnesium turnings (0.20 g, 8.2 mmol) in ca. 75 ml of THF were stirred magnetically at room temperature. The mixture gradually turned deep yellow–green. After 16 h the solvent was stripped in vacuo. The residue was extracted with toluene, filtered through anhydrous sodium sulfate and stripped to dryness. Complex **4** was obtained as a blue–green crystalline solid (0.51 g, 59%). Mp 128–131°C (dec.). ^{1}H NMR (C_6D_6, 20°C): δ7.56 (d, $J=12$ Hz, 2H, biq), 6.94 (dd, $J=9.0$ Hz, $J=6.0$ Hz, 2H, biq), 6.77 (dd, $J=6.0$ Hz, $J=9.0$ Hz, 2H, biq), 6.55 (d, $J=12$ Hz, 2H, biq), 6.28 (d, $J=9$ Hz, 2H, biq), 6.19 (d, $J=9$ Hz, 2H, biq), 5.64 (s, 10H, Cp). IR (KBr, cm^{-1}): 1697 (w), 1575 (br), 1541 (s), 1509 (s), 1489 (w), 1422 (w), 1399 (w), 1321 (br), 1275 (s), 1073 (w), 1018 (s), 862 (w), 810 (s, br), 760 (s), 734 (w), 697 (w). Mass spectrum (70 eV) m/e 434 (M^+, 40%), 256 (biq^+, 100%), 178 (Cp_2Ti^+, 100%). HRMS (m/e) calcd for $C_{28}H_{22}N_2Ti$: 434.126246. Found: 434.126674.

2.3.5. $Cp_2Zr(biq)$ (**5**)

The reaction was ran similar to the procedure for **4**. An etheral solution was filtered and concentrated to ca. 15 ml. Cooling to -78°C caused **5** to deposit as deep-violet crystals (0.78 g, 96%). Mp 128–131°C (dec.) ^{1}H NMR (C_6D_6, 20°C): δ7.74 (d, $J=12$ Hz, 2H, biq), 7.51 (d, $J=12$ Hz, 2H, biq), 7.04 (d, $J=12$ Hz, 2H, biq), 6.87 (d, $J=9$ Hz, 2H, biq), 6.81 (d, $J = 9$ Hz, 2H, biq), 6.45 (d, $J = 15$ Hz, 2H, biq), 5.67 (s, 10H, Cp). IR (KBr, cm^{-1}): 1545 (w), 1479 (w), 1448 (s), 1313 (s), 1262 (s), 1098 (br), 1017 (br), 799 (br), 754 (br). Mass spectrum (70 eV) m/e 476 (M$^+$, 100%), 256 (biq$^+$, 88%), 220 (Cp_2Zr^+, 60%). HRMS (m/e) calcd for $C_{28}H_{22}N_2Zr$: 476.083007. Found: 476.0824.

2.3.6. Cp_2Hf(biq) (**6**)

The reaction was ran similar to the procedure for **4**. After filtration and concentration to ca. 20 ml, an etheral solution was cooled to −78°C. Complex **6** formed violet–black crystals (0.73 g, 99%). Mp 173–177°C (dec.) ^{1}H NMR (C_6D_6, 20°C): δ7.08 (d, 2H, biq), [2] 6.99 (dd, $J = 6$ Hz, $J = 6$ Hz, 2H, biq), 6.82 (d, $J = 12$ Hz, 2H, biq), 6.73 (d, $J = 15$ Hz, 2H, biq), 6.25 (d, $J = 12$ Hz, 2H, biq), 5.77 (s, 10H, Cp), 5.61 (d, $J = 9$ Hz, 2H, biq). IR (KBr, cm^{-1}): 1509 (w), 1479 (w), 1448 (w), 1418 (w), 1397 (w), 1305 (s), 1281 (w), 1260 (s), 1096 (w), 1018 (br), 919 (w), 881 (w), 810 (br), 785 (w), 749 (s). Mass spectrum (70 eV) m/e 562 (M$^+$, 2%), [3] 308 (Cp_2Hf^+, 5%), 243 (CpHf$^+$, 3%).

2.3.7. Cp_2Ti(dmbpy) (**7**)

Cp_2TiCl_2 (0.25 g, 1.0 mmol),4,4′-dimethyl-2,2′-bipyridyl (0.18 g, 0.98 mmol), and magnesium turnings (0.29 g, 11.9 mmol) in ca. 50 ml of THF were stirred magnetically at ambient temperature. The reaction mixture gradually turned dark brown–green. After 14 h the mixture was filtered through a Schlenk fritte and the solvent was removed in vacuo. The black–green residue was extracted with hexanes and filtered through a Schlenk fritte. The solvent was then removed in vacuo and complex **7** was isolated as a dark black–green solid (0.29 g, 81%). ^{1}H NMR (C_6D_6, 20°C) δ6.50–6.47 (m, dmbpy), 6.30–6.28 (m, dmbpy), 5.47 (s, 10H, Cp) 2.35–2.34 (Me, dmbpy). IR (Nujol, cm^{-1}): 2728 (w), 1463 (s, br), 1382 (s), 1277 (m, br), 1172 (w), 1120 (w), 1075 (w), 1043 (m, br), 1017 (m), 973 (m), 940 (w), 893 (m, br), 834 (m), 800 (m), 782 (w), 772 (m), 735 (w), 720 (m, br). Mass spectrum (70 eV) m/e 362 (M$^+$, 25%), 184 (dmbpy$^+$, 58%), 178 (Cp_2Ti^+, 100%). HRMS (m/e) calcd for $C_{22}H_{22}N_2Ti$: 362.126246. Found: 362.126425.

2.3.8. Cp_2Zr(dmbpy) (**8**)

The reaction was run similar to the procedure for **7**. A toluene solution was filtered and the solvent was removed in vacuo. Complex **8** was obtained as a dark purple crystalline solid in good yield. ^{1}H NMR (C_6D_6, 20°C) δ7.45 (d, $J = 6.6$ Hz, 2H, dmbpy), 6.82 (s, dmbpy), 5.41 (s, 10H, Cp), 5.30–5.27 (m, dmbpy), 1.98 (s, 6H, Me). IR (Nujol, cm^{-1}): 1601 (m), 1470 (s), 1453 (s), 1381 (m), 1328 (w), 1287 (m), 1268 (m), 1173 (w), 1068 (w), 1039 (m, br), 1013 (m, br), 951 (w), 889 (m, br), 839 (w), 822 (w), 791 (s), 766 (w), 758 (m), 723 (w). Mass spectrum (70 eV) m/e 404 (M$^+$, 60%), 220 (Cp_2Zr^+, 57%), 184 (dmbpy$^+$, 100%). HRMS (m/e) calcd for $C_{22}H_{22}N_2Zr$: 404.083007. Found: 404.082400.

3. Results and discussion

3.1. Syntheses of complexes.

Research in our laboratories has focused on the synthesis of group 4 metallocene derivatives of the weakly-coordinating bidentate nitrogen ligands bipyridyl and biquinoline. We obtained the Cp_2M(bpy) and Cp_2M(biq) complexes **1–6** (M = Ti, Zr and Hf; bpy = bipyridyl, biq = 2,2′-biquinoline) as well as Cp_2M(dmbpy) complexes **7** and **8** (M = Ti, Zr; dmbpy = 4,4′-dimethyl-2,2′-bipyridyl) as green–black (**1, 4, 7**) or deep-violet (**2, 3, 5, 6, 8**), crystalline solids in 59–99% yields via Mg reduction of the respective metallocene dichlorides in THF in the presence of one equivalent of ligand. We isolated **1–8** after extracting the stripped reaction mixtures with hexane, Et_2O, or toluene, followed by filtering and crystallization at low temperature. Complexes **1–8** are exceedingly sensitive to air and moisture.

Compounds **1** and **2** have been previously synthesized. Fischer and Amtmann [15] treated $Cp_2Ti(CO)_2$ with 2,2′-bipyridyl to obtain **1** in 72% yield. [4] Alternatively, Calderazzo et al. [16] added dilithium bipyridyl dropwise to a solution of Cp_2TiCl_2, isolating **1** in 81% yield. The synthesis of **2** was reported by Wailes and Weigold [17] in which

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[2] The coupling constant for this peak was difficult to determine due to the overlap of the deuterated solvent peak.

[3] This particular sample required the aid of mercuric chloride in order for the reduction to ensue. The resulting mass spectrum was a mixture of species.

[4] Cp_2Ti(bpy) (**1**) was first reported in 1967. See Refs. [15,16]. Both papers indicated that Cp_2Ti(bpy) (**1**) is paramagnetic. Our method of synthesis affords diamagnetic product. See Ref. [19].

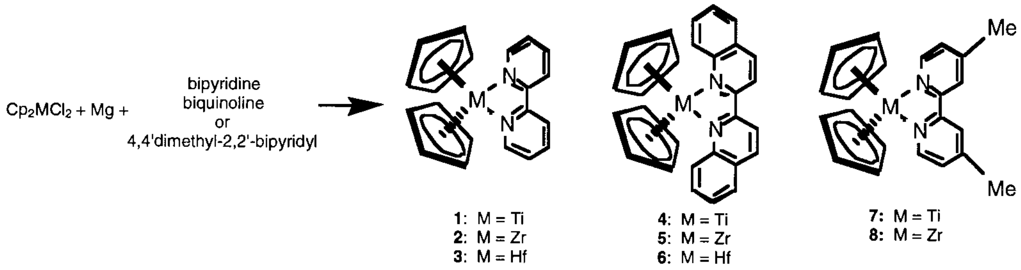
Table 1
^1H NMR spectral data for the Cp signals of complexes **1–8**[a] and related complexes

Complex	Cp	Complex	Cp
$Cp_2Ti(bpy)$(**1**)	5.49	$Cp_2Ti(PMe_3)_2$[20]	4.51
$Cp_2Zr(bpy)$(**2**)	5.40	$Cp_2Zr(PMe_3)_2$[19]	4.75
$Cp_2Hf(bpy)$(**3**)	5.35	$Cp_2Ti(PMe_3)(CO)$[21]	4.62
$Cp_2Ti(biq)$(**4**)	5.64	$Cp_2Zr(PMe_3)(CO)$[21]	4.91
$Cp_2Zr(biq)$(**5**)	5.67	$Cp_2Hf(PMe_3)(CO)$[20]	4.78
$Cp_2Hf(biq)$(**6**)	5.77	$Cp_2Ti(CO)_2$[26]	4.53
$Cp_2Ti(dmbpy)$(**7**)	5.47	$Cp_2Ti(CNC_6H_3Me_2)_2$[27]	5.10
$Cp_2Zr(dmbpy)$(**8**)	5.41	$Cp_2Zr(CNC_6H_3Me_2)_2$[27]	5.40

[a] Spectra for **1–8** are reported in ppm vs. TMS and were obtained at 300 MHz at 20°C in C_6D_6.

Cp_2ZrCl_2 was reduced in the presence of disodium bipyridyl to give the desired product in 72% yield. [5] Beckhaus and Thiele [18] obtained **2** in 49% yield by reacting vinyl zirconocene with 2,2′-bipyridyl.

Our preparation of **1** and **2** resulted in yields of 87 and 83%, respectively. In addition, by utilizing magnesium as a reducing agent, milder reaction conditions are employed. As in the previous cases, both compounds were extracted in similar solvents and are extremely air-sensitive. Compound **1** [15,16] has been reported previously as a paramagnetic compound while only ^{13}C NMR data has been reported for compound **2** [17,18]. We have found them to be diamagnetic and report their ^1H NMR spectra.



The ^1H NMR spectral signals for the Cp protons for complexes **1–8** are given in Table 1. A comparison of the bipyridyl complexes **1–3** reveals that signals assigned to the Cp ring shift upfield on proceeding down the group 4 triad. This observation contrasts with a comparison of the Cp resonances for $Cp_2Ti(PMe_3)_2$ [20] (δ4.51) vs. $Cp_2Zr(PMe_3)_2$ [19] (δ4.75), that reveals a downfield shift on descending from titanium to zirconium, and with the series [21] $Cp_2Ti(PMe_3)(CO)$(δ4.62), $Cp_2Zr(PMe_3)(CO)$(δ4.91) and $Cp_2Hf(PMe_3)(CO)$ (δ4.78) that indicates that the Cp rings are most deshielded in the zirconocene complex. In all cases the Cp signals of the bipyridyl and biquinoline complexes **1–8** are downfield of those reported for other Cp_2ML_2 complexes, with $L = PMe_3$ and CO, which indicates there is significantly lower electron density on Cp (and, presumably, on the metal) for these bipyridyl and biquinoline complexes than is observed for analogous complexes having phosphine or carbonyl ligands.

In all cases, the aromatic signals for the three ligands in the complexes shift upfield with respect to the free ligand (Table 2). In correspondence with the presumed lower electron density on the Cp rings as well as the metal, one may propose that greater electron density is delocalized within the ligand. Interestingly, the Zr species **5** and **8** show a downfield shift in comparison to the corresponding Ti complexes **4** and **7**, thus indicating more electron density on Zr.

As previously mentioned, we are reporting the ^1H NMR spectrum for the known complex $Cp_2Ti(bpy)$ (**1**). The ^1H NMR spectrum of $Cp_2Ti(biq)$ (**4**) has signals for the complex that are somewhat broad, indicating that the bidentate species is slightly paramagnetic. $Cp_2Ti(dmbpy)$ (**7**) behaves as a paramagnetic species. [6] McPherson et al. [22] have studied the electronic properties of **1** and discovered that it has a triplet state which is thermally accessible from the ground state. At room temperature, **1** gave rise to an intense EPR signal. As the temperature increased, the signal lost its intensity. This temperature-dependent EPR spectrum suggested a diamagnetic species in the ground state but a paramagnetic excited state. Therefore, our synthetic route and NMR conditions made it possible in our case to obtain an ^1H NMR spectrum of **1**. We believe to be on the threshold for the proper temperature in order to obtain a spectrum, for some attempts were not successful.

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[5] $Cp_2Zr(bpy)$ (**2**) has been reported previously. See Refs. [17,18]. No ^1H NMR spectral data were given.

[6] ^1H NMR spectra has only been obtained once for this complex. Signals due to the complex, in this case, were broad.

Table 2
^{1}H NMR spectral data[a] for the aromatic signals of complexes **1–8** and free ligands

Ligand	Aromatics	Complex	Aromatics
bpy	8.75–6.67	$Cp_2Ti(bpy)$(**1**)	7.67–6.42
		$Cp_2Zr(bpy)$(**2**)	7.41–6.31
		$Cp_2Hf(bpy)$(**3**)	7.20–5.29
biq	9.13–7.17	$Cp_2Ti(biq)$(**4**)	7.56–6.19
		$Cp_2Zr(biq)$(**5**)	7.74–6.45
		$Cp_2Hf(biq)$(**6**)	7.08–5.61
dmbpy	8.70–6.61	$Cp_2Ti(dmbpy)$(**7**)	6.50–6.28
		$Cp_2Zr(dmbpy)$(**8**)	7.45–5.30

[a] Spectra for **1–8** and free ligands are reported in ppm vs. TMS and were obtained at 300 MHz at 20°C in C_6D_6.

We are currently investigating the reactivity of these complexes, in particular, $Cp_2Zr(biq)$ (**5**). For example, treatment of **5** with 1 atm of CO leads to the formation of the dicarbonyl $Cp_2Zr(CO)_2$ [23–25].

4. Conclusions

The bipyridine and biquinoline complexes of the group 4 metallocenes are readily available via the magnesium reduction of the respective dichlorides in the presence of the bidentate ligands. The extreme reactivity of these metallocene derivatives will be the focus of future studies.

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