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Studies on cyclomercuration of bisferrocenylimines

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Abstract

Cyclomercuration of bisferrocenylimines $[(\eta^5\text{-}C_5H_5)Fe\{(\eta^5\text{-}C_5H_4)\text{–}CR{=}N\text{–}(C_6H_3\text{-}2\text{-}R')\}]_2$, (R = H, CH_3; R′ = H, CH_3, Cl) occurred predominantly in the *ortho*-position of the substituted ferrocenyl ring to yield double 2-mercurated ferrocenylimines, which arise from activation of two $\sigma(C_{sp^2,\ ferrocene}\text{–}H)$ bonds. The composition and structure of the metallocycles were characterized by elemental analysis, IR, ^1H NMR and 2D $^{13}C\{^1H\}$-COSY spectra, which confirmed the existence of two five-membered mercuriocycles fused with the ferrocenyl moieties.

Keywords: Bisferrocenylimine; Cyclomercuration

1. Introduction

Cyclometallated compounds have attracted much research interest in recent decades owing to their potential utility in organic synthesis, catalysis, asymmetric synthesis and photochemistry [1]. Organomercurials have been used extensively in organic synthesis as well as in mechanism investigation due to their ability to accommodate practically all of the important organic functional groups and their ease in undergoing transmetallation for the synthesis of other organometallic compounds [2]. It has been found out that chiral cyclomercurated ferrocenylimines can be used as transmetallation reagents for the synthesis of other chiral organometallic compounds with the retention of the absolute configuration in the ferrocene moiety [3], thus they provide a new and convenient method for the synthesis of chiral 1,2-disubstituted ferrocenes. Studies on the cyclomercuration of ferrocenylimines in our laboratory have been systematically reported [4]. It just intrigued us to find out whether related bisfunctional ligands, such as the bisferrocenylimines **3** and **4** (Scheme 1) could undergo the activation of two $\sigma(C_{sp^2,\ ferrocene}\text{–}H)$ bonds.

2. Results and discussion

Bisferrocenylimines **3** and **4** were synthesized by condensation of benzidine or *ortho*-substituted benzidine with formylferrocene **1** and acetylferrocene **2**, respectively, in the presence of freshly activated Al_2O_3 and using toluene as solvent. Cyclomercuration of **3** and **4** with mercuric acetate and subsequent treatment with lithium chloride at room temperature gave double cyclomercurated bisferrocenylimines **5** and **6**. Purification of compounds **5** and **6** was performed by chromatography of the reaction mixture on silica gel, since both of them exhibited higher R_f values than those of the corresponding starting materials. All the double cyclomercurated compounds were red or orange solids, stable in air and soluble in dimethylsulfoxide (DMSO), chloroform, acetone and benzene, but insoluble in methanol and hexane. The products **3**–**6** were characterized by elemental analysis, IR and ^1H NMR spectra. It is noteworthy that the double cyclomercurated bisferrocenylimines were the main product even with equimolar amounts of bisferrocenylimines and mercuric acetate, which indicates that the chloromercurio groups were directed by imino nitrogen to two substituted Cp-rings simultaneously.

Elemental analyses of complexes **3**–**6** were consistent with the proposed formulae. The IR spectra of ligands **3** and **4** exhibited a sharp and intense band at approxi-

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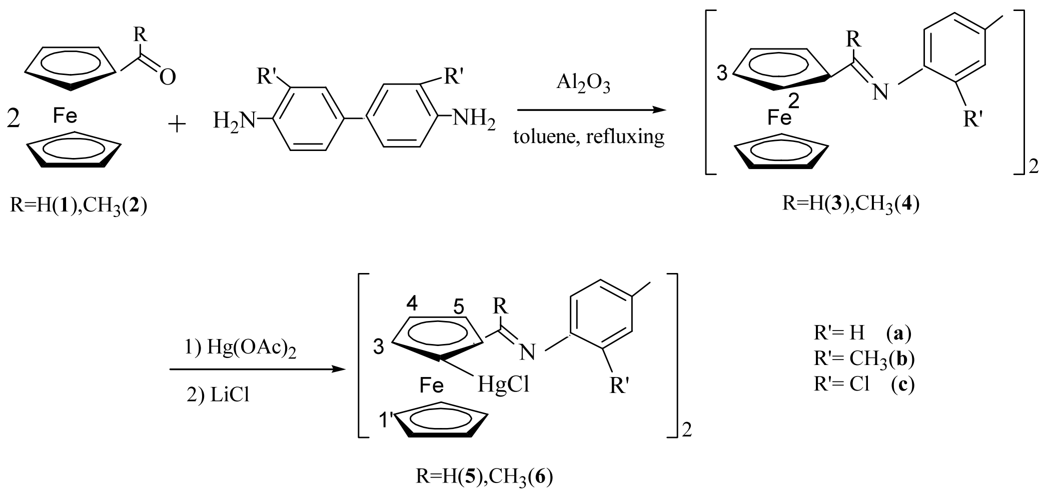

Scheme 1.

mately 1625 cm^{-1}, which was assigned to the asymmetric stretching of the >C=N– group. The C=N absorption of **5** and **6** were shifted to lower energy ranging from 5 to 19 cm^{-1}, indicating a N→Hg intramolecular coordination. These results are well in agreement with those reported on related cyclomercurated mono-ferrocenylimines and 1,1′-bisferrocenylimines [5], and show that mercuration of the bisferrocenylimines **3** and **4** afforded double cyclomercurated bisferrocenylimines. All the compounds showed absorptions at approximately 1000 and 1100 cm^{-1}, indicating an unsubstituted Cp-ring [6]. The ^1H NMR spectra of the free ligand, for example **3a**, showed two signals at 4.82 and 4.50 ppm, ascribed to protons **2** and **3**, respectively. However, three singlets at 4.55 (H-3), 4.71 (H-4) and 4.83 ppm (H-5), ascribed to three types of non-equivalent protons of the substituted ferrocenyl unit were observed in compound **5a**. The chemical shifts of the protons of the ferrocenyl ring demonstrated no apparent difference between compounds **a** and **b**, and there was only a slightly low field shift presented in **c**. This indicates that the *N*-phenyl ring and C=N double bond were in different planes, and there was no effective conjugation between the ferrocenyl ring and the *N*-phenyl ring.

Two-dimensional C, H-COSY (Fig. 1) was also measured for **5b** for elucidating the structure in solution. The ^1H NMR and C, H-COSY spectra showed that the corresponding proton and carbon atoms at the two halves of the molecule did not behave differently in identical magnetic environments. Moreover, the proton signals correlated with the corresponding carbon signals; the proton signals for substituted and unsubstituted Cp-rings occurred at 4.94 (H-5), 4.72 (H-4), 4.58 (H-3) and 4.30 ppm (H-1′), that would be corresponding to the carbon signals at 72.79 (C-5), 73.43 (C-4), 77.28 (C-3) and 69.94 ppm (C-1′), respectively. It was confirmed by our experiment that the double cyclomercurated bisferrocenylimines existed as one isomer.

3. UV–Vis spectra

The UV–Vis spectra were recorded in CH_2Cl_2. All the examined compounds showed two characteristic bands in the UV–Vis spectra. The absorption around 460 nm (ε, 1108 l mol^{-1} cm^{-1}) was assigned to the d–d transition. A higher energy band around 350 nm (ε, 14985 l mol^{-1} cm^{-1}) was assigned as the Fe–Cp transition. An examination of UV–Vis spectra of the cyclomercurated bisferrocenylimines showed that an intramolecular coordination, N→Hg, existed in these molecules, with red shifts by approximately 10 nm on comparing with the corresponding bands in the bisferrocenylimines.

4. Experimental

Melting points (m.p.) were measured on an XT-4A apparatus and were uncorrected. Elemental analyses were determined with a Carlo Erba 1160 Elemental Analyser. ^1H NMR spectra were recorded on a Bruker DPX 400 spectrometer, using $CDCl_3$ as solvent and TMS as an internal standard, C, H-COSY using DMSO as solvent. IR spectra were recorded on a Bruker VECTOR 22 spectrophotometer. UV–Vis spectra of the complexes were measured with a Shimadzu UV-2100 spectrophotometer, using CH_2Cl_2 as solvent. Chromatographic work was carried out using silica gel under reduced pressure. All the solvents were purified according to the standard methods. Benzidine and *ortho*-substituted benzidines were obtained from commercial sources and used as received.

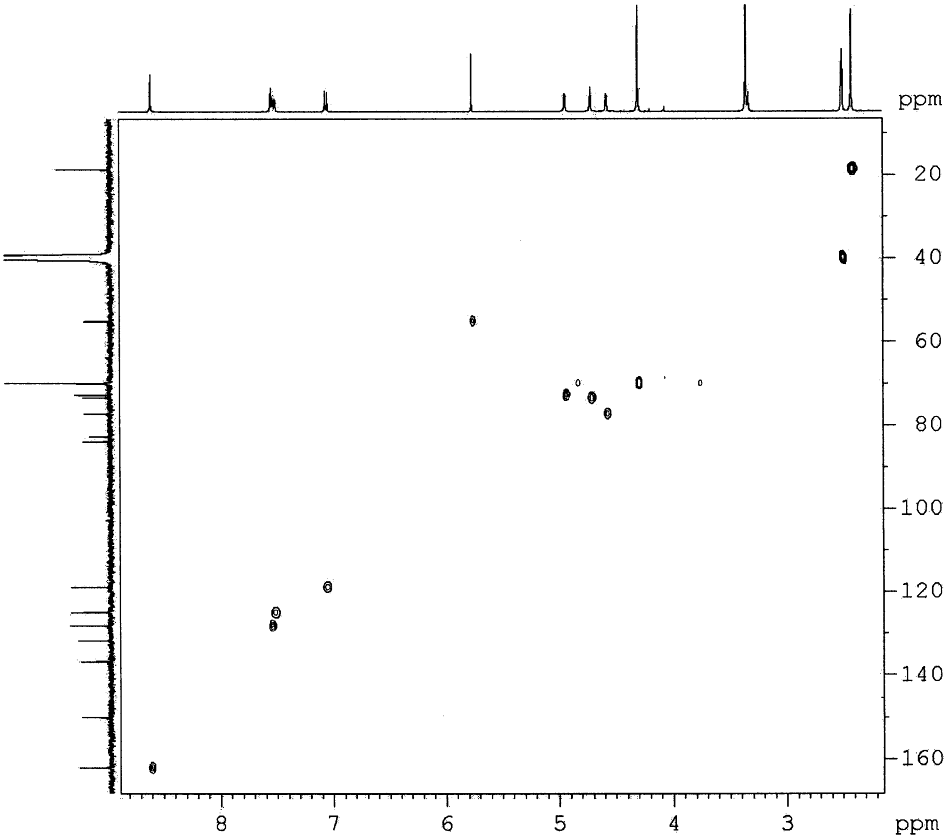

Fig. 1. Two-dimensional C, H-COSY of compound **5b**.

*4.1. General procedure for the synthesis of compounds **3** and **4***

Formylferrocene or acetylferrocene (1 mmol) was dissolved in toluene (20 ml) at room temperature and the corresponding benzidine (1 mmol) was added. The red solution was refluxed under argon atmosphere for about 3 days. Every day an addition of small quantities of freshly activated Al_2O_3 was necessary to complete the reaction. The hot solution was carefully filtered, and the filtrate was concentrated to dryness in a rotary evaporator. The residue was washed with ethanol to remove unreacted starting materials and then dried under reduced pressure to give bisferrocenylimines **3** or **4**. Characterization data are as follows:

$[(\eta^5\text{-}C_5H_5)Fe\{(\eta^5\text{-}C_5H_4){-}CH{=}N{-}(C_6H_4)\}]_2$ (**3a**): orange red solid, yield 82%; m.p. > 247 °C (dec.); IR (KBr pellet): 3083, 1625, 1587, 1498, 1460, 1109, 1009, 824, 489 cm^{-1}; UV–Vis (CH_2Cl_2) λ_{max} (ε, 1 mol^{-1} cm^{-1}): 462 (1041), 345 (14490) nm; ^1H NMR (ppm): δ 4.34 (s, 10H, unsubstituted Cp-ring), 4.50 (bs, 4H, H-3), 4.82 (bs, 4H, H-2), 8.40 (s, 2H, *CH*), 7.23–7.63 (m, 8H, protons on phenyl ring). Anal. Calc. for $C_{34}H_{28}Fe_2N_2$: C, 70.86; H, 4.89; N, 4.86. Found: C, 71.17; H, 4.74; N, 4.92%.

$[(\eta^5\text{-}C_5H_5)Fe\{(\eta^5\text{-}C_5H_4){-}CH{=}N{-}(C_6H_3\text{-}2\text{-}CH_3)\}]_2$ (**3b**): orange solid, yield 89%; m.p. 222–223 °C; IR (KBr pellet): 3101, 1623, 1599, 1488, 1463, 1221, 1106, 1001, 824, 503 cm^{-1}; UV–Vis (CH_2Cl_2) λ_{max} (ε, 1 mol^{-1} cm^{-1}): 460 (1108), 350 (14985) nm; ^1H NMR (ppm): δ 4.26(s, 10H, unsubstituted Cp-ring), 4.51 (bs, 4H, H-3), 4.86 (bs, 4H, H-2), 8.29 (s, 2H, *CH*), 2.42 (s, 6H, CH_3), 6.94–7.46 (m, 6H, protons on phenyl ring). Anal. Calc. for $C_{36}H_{32}Fe_2N_2$: C, 71.55; H, 5.34; N, 4.64. Found: C, 71.61, H, 5.33, N, 4.74%.

$[(\eta^5\text{-}C_5H_5)Fe\{(\eta^5\text{-}C_5H_4){-}CH{=}N{-}(C_6H_3\text{-}2\text{-}Cl)\}]_2$ (**3c**): orange solid, yield 80%; m.p. > 230 °C (dec.); IR (KBr pellet): 3090, 1624, 1455, 1104, 1217, 1043, 999, 821, 495 cm^{-1}; UV–Vis (CH_2Cl_2) λ_{max} (ε, 1 mol^{-1} cm^{-1}): 467 (1005), 340 (14700) nm; ^1H NMR (ppm): δ 4.30 (s, 10H, unsubstituted Cp-ring), 4.61 (s, 4H, H-3), 4.80 (s, 4H, H-2), 8.31 (s, 2H, *CH*), 6.80–7.67 (m, 6H, protons on phenyl ring). Anal. Calc. for $C_{34}H_{26}Cl_2Fe_2N_2$: C, 63.29; H, 4.06; N, 4.34. Found: C, 63.05; H, 3.83; N, 4.60%.

$[(\eta^5\text{-}C_5H_5)Fe\{(\eta^5\text{-}C_5H_4){-}C(CH_3){=}N{-}(C_6H_4)\}]_2$ (**4a**): yellow orange, yield 77%; m.p. > 260 °C (dec.); IR (KBr pellet): 3089, 3028, 1628, 1497, 1459, 1286, 1234, 1111, 1008, 830, 495 cm^{-1}; UV–Vis (CH_2Cl_2) λ_{max} (ε, 1 mol^{-1} cm^{-1}): 449 (650), 329 (4432) nm; ^1H NMR (ppm): δ

4.23 (s, 10H, unsubstituted Cp-ring); 4.42 (s, 4H, H-3), 4.84 (s, 4H, H-2), 2.16 (s, 6H, *CH*$_3$), 6.84–7.60 (m, 8H, protons on phenyl ring). Anal. Calc. for $C_{36}H_{32}Fe_2N_2$: C, 71.56; H, 5.34; N, 4.63. Found: C, 71.63; H, 5.52; N, 4.73%.

$[(\eta^5\text{-}C_5H_5)Fe\{(\eta^5\text{-}C_5H_4)\text{–}C(CH_3){=}N\text{–}(C_6H_3\text{-2-}CH_3)\}]_2$ (**4b**): orange solid, yield 79%; m.p. 208–209 °C; IR (KBr pellet): 3094, 3017, 1627, 1467, 1366, 1287, 1234, 1119, 1001, 824, 489 cm^{-1}; UV–Vis (CH_2Cl_2) λ_{max} (ε, l mol^{-1} cm^{-1}): 447 (672), 339 (4800) nm; ^{1}H NMR (ppm): δ 4.24 (s, 10H, unsubstituted Cp-ring), 4.44 (s, 4H, H-3), 4.85 (s, 4H, H-2), 2.08 (s, 6H, *CH*$_3$), 2.22 (s, 6H, *CH*$_3$ on phenyl ring), 6.66–7.46 (m, 6H, protons on phenyl ring). Anal. Calc. for $C_{38}H_{36}Fe_2N_2$: C, 72.10; H, 5.74; N, 4.43. Found: C, 71.82; H, 5.75; N, 4.44%.

$[(\eta^5\text{-}C_5H_5)Fe\{(\eta^5\text{-}C_5H_4)\text{–}C(CH_3){=}N\text{–}(C_6H_3\text{-2-Cl})\}]_2$ (**4c**): orange solid, yield 78%; m.p. > 180 °C (dec.); IR (KBr pellet): 3094, 1626, 1460, 1367, 1291, 1233, 1115, 1054, 1002, 825, 487 cm^{-1}; UV–Vis (CH_2Cl_2) λ_{max} (ε, l mol^{-1} cm^{-1}): 450 (663), 341 (5075) nm; ^{1}H NMR (ppm): δ 4.28 (s, 10H, unsubstituted Cp-ring); 4.47 (s, 4H, H-3), 4.86 (s, 4H, H-2), 2.11 (s, 3H, *CH*$_3$), 6.82–7.65 (m, 6H, protons on phenyl ring). Anal. Calc. for $C_{36}H_{30}Cl_2Fe_2N_2$: C, 64.23; H, 4.49; N, 4.16. Found: C, 64.32; H, 4.63; N, 4.09%.

4.2. General procedure for the synthesis of compounds **5** and **6**

0.5 mmol of bisferrocenylimines **3** or **4** and 10 ml methylene dichloride were added into a 50 ml flask equipped with a magnetic stirrer and an equilibrated addition funnel. 1 mmol of mercuric acetate was dissolved in a sufficient amount of methanol, and was added dropwise to the flask over a period of 30 min. Subsequently, 1.5 mmol of lithium chloride dissolved in 25 ml methanol was slowly added, and then the mixture was stirred vigorously for 30 min at room temperature, and a large quantity of solid was formed. The contents of the flask were transferred to a separatory funnel, and an additional 20 ml of methylene dichloride and 30 ml water were added. The combined organic phase was washed with two 30 ml brine, and then dried over Na_2SO_4. The resulting solution was evaporated in vacuo to a minimum amount and subjected to a short dry column (200 mm × ∅ 25 mm) of silica gel, eluted with methylene dichloride. The first band was compound **5** or **6**. The products were characterized as following:

$[HgCl(\eta^5\text{-}C_5H_5)Fe\{(\eta^5\text{-}C_5H_3)\text{–}CH{=}N\text{–}(C_6H_4)\}]_2$ (**5a**): orange solid, yield 35%; m.p. 212–213 °C; IR (KBr pellet): 3089, 1612, 1589, 1491, 1440, 1331, 1203, 1108, 1032, 998, 825, 503 cm^{-1}; UV–Vis (CH_2Cl_2) λ_{max} (ε, l mol^{-1} cm^{-1}): 468 (1380), 363 (1862) nm; ^{1}H NMR (ppm): δ 4.28 (s, 10H, unsubstituted Cp-ring); 4.55 (bs, 2H, H-3), 4.71 (bs, 2H, H-4), 4.83 (bs, 2H, H-5), 8.82 (s, 2H, *CH*), 7.40, 7.65 (d, d, 8H, $J = 8.4$ Hz, protons on phenyl ring). Anal. Calc. for $C_{34}H_{26}Cl_2Fe_2Hg_2N_2$: C, 39.03; H, 2.50; N, 2.68. Found: C, 38.87; H, 2.20; N, 2.52%.

$[HgCl(\eta^5\text{-}C_5H_5)Fe\{(\eta^5\text{-}C_5H_3)\text{–}CH{=}N\text{–}(C_6H_3\text{-2-}CH_3)\}]_2$ (**5b**): orange solid, yield 37%; m.p. 185–186 °C; IR (KBr pellet): 3088, 1605, 1485, 1444, 1202, 1116, 997, 814, 509 cm^{-1}; UV–Vis (CH_2Cl_2) λ_{max} (ε, l mol^{-1} cm^{-1}): 466 (1346), 357 (15018) nm; ^{1}H NMR (ppm): δ 4.29 (bs, 10H, unsubstituted Cp-ring); 4.53 (bs, 2H, H-3), 4.70 (t, 2H, $J = 2.4$ Hz, H-4), 4.83 (d, 2H, $J = 1.6$ Hz, H-5), 8.55 (s, 2H, *CH*), 2.50 (s, 6H, *CH*$_3$), 6.92–7.46 (m, 6H, protons on phenyl ring). $^{13}C\{^1H\}$ NMR (DMSO): δ 18.66 (CH_3), 69.94 (CH in unsubstituted Cp-ring), 72.79, 73.44, 77.28 (CH in substituted Cp-ring), 82.73, 83.84 (C in substituted Cp-ring), 118.9, 125.0, 128.2, (CH in phenyl ring), 131.8, 136.9, 150.1 (C in phenyl ring), 162.1 (CHN). Anal. Calc. for $C_{36}H_{30}Cl_2Fe_2Hg_2N_2$: C, 40.24; H, 2.81; N, 2.61. Found: C, 40.51; H, 2.93; N, 2.86%.

$[HgCl(\eta^5\text{-}C_5H_5)Fe\{(\eta^5\text{-}C_5H_3)\text{–}CH{=}N\text{–}(C_6H_3\text{-2-Cl})\}]_2$ (**5c**): orange solid, yield 30%; m.p. 198–199 °C; IR (KBr pellet): 3089, 1618, 1481, 1439, 1206, 1109, 1044, 999, 823, 494 cm^{-1}; UV–Vis (CH_2Cl_2) λ_{max} (ε, l mol^{-1} cm^{-1}): 478 (1416), 355 (14862) nm; ^{1}H NMR (ppm): δ 4.32 (s, 10H, unsubstituted Cp-ring), 4.58 (bs, 2H, H-3), 4.74 (t, 2H, $J = 2.4$ Hz, H-4), 4.84 (bs, 2H, H-5), 8.60 (s, 6H, *CH*), 7.04–7.66 (m, 6H, protons on phenyl ring). Anal. Calc. for $C_{34}H_{24}Cl_4Fe_2Hg_2N_2$: C, 36.62; H, 2.17; N, 2.51. Found: C, 36.69; H, 2.51; N, 2.34%.

$[HgCl(\eta^5\text{-}C_5H_5)Fe\{(\eta^5\text{-}C_5H_3)\text{–}C(CH_3){=}N\text{–}(C_6H_4)\}]_2$ (**6a**): orange solid, yield 38%; m.p. > 167 °C (dec.); IR (KBr pellet): 3088, 3029, 1609, 1492, 1437, 1219, 1106, 1004, 831, 493 cm^{-1}; UV–Vis (CH_2Cl_2) λ_{max} (ε, l mol^{-1} cm^{-1}): 455 (943), 347 (5709) nm; ^{1}H NMR (ppm): δ 4.26 (bs, 10H, unsubstituted Cp-ring); 4.47 (bs, 2H, H-3), 4.66 (bs, 2H, H-4), 4.86 (bs, 2H, H-5), 2.17 (s, 6H, *CH*$_3$), 6.91, 7.60 (d, d, 8H, $J = 6.9$ Hz, $J = 7.2$ Hz, protons on phenyl ring). Anal. Calc. for $C_{36}H_{30}Cl_2Fe_2Hg_2N_2$: C, 40.24; H, 2.81; N, 2.61. Found: C, 40.60; H, 2.99; N, 2.22%.

$[HgCl(\eta^5\text{-}C_5H_5)Fe\{(\eta^5\text{-}C_5H_3)\text{–}C(CH_3){=}N\text{–}(C_6H_3\text{-2-}CH_3)\}]_2$ (**6b**): orange red solid, yield 36%; m.p. > 220 °C (dec.); IR (KBr pellet): 3091, 1612, 1483, 1439, 1365, 1223, 1110, 994, 827, 489 cm^{-1}; UV–Vis (CH_2Cl_2) λ_{max} (ε, l mol^{-1} cm^{-1}): 453 (858), 345 (4926) nm; ^{1}H NMR (ppm): δ 4.27 (bs, 10H, unsubstituted Cp-ring); 4.47 (bs, 2H, H-3), 4.67 (bs, 2H, H-4), 4.86 (bs, 2H, H-5), 2.14 (s, 6H, *CH*$_3$), 2.29 (s, 6H, *CH*$_3$ on phenyl ring), 6.62–7.46 (m, 6H, protons on phenyl ring). Anal. Calc. for $C_{38}H_{34}Cl_2Fe_2Hg_2N_2$: C, 41.30; H, 3.10; N, 2.54. Found: C, 41.50; H, 3.18; N, 2.60%.

$[HgCl(\eta^5\text{-}C_5H_5)Fe\{(\eta^5\text{-}C_5H_3)\text{–}C(CH_3){=}N\text{–}(C_6H_3\text{-2-Cl})\}]_2$ (**6c**): orange red solid, yield 22%; m.p. > 206 °C (dec.); IR (KBr pellet): 3090, 1618, 1475, 1437, 1369, 1219, 1145, 1056, 999, 831, 492 cm^{-1}; UV–Vis

(CH_2Cl_2) λ_{max} (ε, l mol^{-1} cm^{-1}): 456 (984), 345 (5568) nm; ^{1}H NMR (ppm): δ 4.34 (bs, 10H, unsubstituted Cp-ring), 4.52 (bs, 2H, H-3), 4.72 (bs, 2H, H-4), 4.90 (bs, 2H, H-5), 2.15 (s, 6H, CH_3), 6.80–7.65 (m, 6H, protons on phenyl ring). Anal. Calc. for $C_{36}H_{28}Cl_4Fe_2Hg_2N_2$: C, 37.87; H, 2.47; N, 2.45. Found: C, 37.61; H, 2.05; N, 2.24%.

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