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Resolution of cycloplatinated ferrocenylketimines

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Abstract

The resolution of cycloplatinated ferrocenylketimines was carried out by using *S*-leucine as chiral auxiliary and a pair of diastereomers was obtained. The optically active derivatives of the cycloplatinated ferrocenylketimines have been prepared and characterized. The structures and absolute configurations of (−)-(S_p,*S*)-[Pt{(η^5-C_5H_3CMe=NC_6H_4-4-CH_3)Fe(η^5-C_5H_5)}(*S*-leu)] and (−)-(S_p)-[Pt{(η^5-C_5H_3CMe=NC_6H_4-4-Br)Fe(η^5-C_5H_5)}(PPh_3)Cl] were determined by X-ray diffraction, on the basis of which the absolute configurations of other optically active compounds studied were ascertained.

1. Introduction

Recently, metallocene chemistry has undergone something of a renaissance.[1] Ferrocene is an ideal framework into which planar chirality can be introduced. Chiral and optically active 1,2-disubstituted ferrocenes have proved to be of particular interest for their application in asymmetric syntheses.[2] The common way to obtain the planar chirality in the ferrocene moiety is through use of a chiral precursor.[3–5] Sokolov et al. have reported the asymmetric cyclopalladation of N,N-dimethylaminoethylferrocene with the salts of optically active carboxylic acids.[6] However, this method was only suitable when the reaction experienced a diastereomeric transition state. The only report of the resolution of cyclometallated ferrocene derivatives was given by Nonoyama and coworkers.[7] They resolved N,N-dimethylaminomethylferrocene into enantiomerically pure form by using *S*-proline as the chiral auxiliary.

Although chiral cyclopalladated ferrocene derivatives have already been studied, chiral cycloplatinated ferrocene derivatives have not been reported. In this paper we described the resolution of cycloplatinated ferrocenylketimines, via the derivatives of *S*-leucine, and the preparation of their optically active derivatives (Scheme 1).

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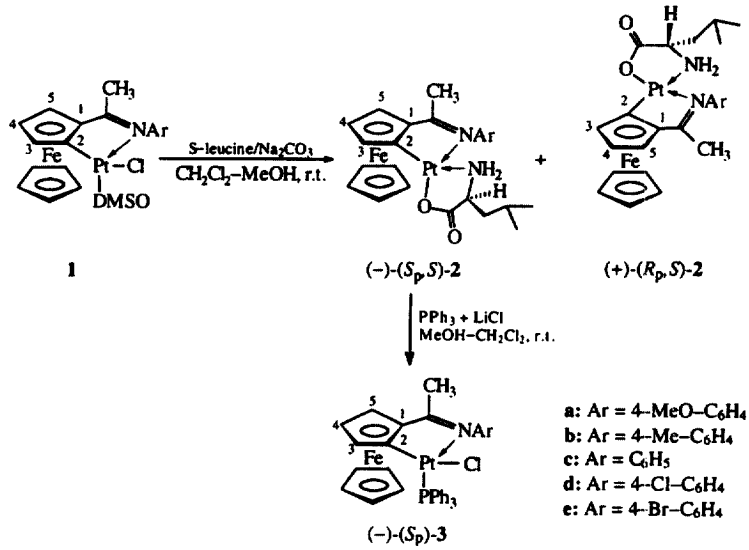

1 (−)-(S_p,S)-2 (+)-(R_p,S)-2 (−)-(S_p)-3

Scheme 1.

2. Results and discussion

The reaction of cycloplatinated ferrocenylketimines **1a–e** with *S*-leucine and sodium carbonate in MeOH:CH_2Cl_2 at room temperature produced (−)-(S_p,S)-**2a–e** (higher R_f value) and (+)-(R_p,S)-**2a–e** in 48–72% yields, after thin layer chromatography (CH_2Cl_2:acetone 3.5:1). Both of the compounds were characterized by EA, IR, and ^{1}H NMR spectra. IR absorptions at about 3230, 3320 and 1640 cm^{-1} indicated the presence of the NH_2 group and carboxylate group. At about 1542 cm^{-1} were the imine absorptions, which were similar to those of compounds **1a–e**.[8] The ^{1}H NMR spectra of (−)-(S_p,S)-**2a–e** and (+)-(R_p,S)-**2a–e** had different chemical shifts of the C-3 proton, which could be used as an indication of the complete separation of the diastereomers. The CD spectra of the pair of diastereomers **2b** were nearly enantiomeric to each other (Fig. 1), which suggested that the two compounds are a pair of diastereomers resulting from the planar chirality. The structural relationships were elucidated by ^{1}H NMR, thin-layer chromatography and X-ray crystal and structural analysis of (−)-(S_p,S)-**2b** (Fig. 2).

Compounds (−)-(S_p,S)-**2a–e** were stirred with lithium chloride and triphenyl phosphine in MeOH:CH_2Cl_2 at room temperature to produce the new compounds (−)-(S_p)-**3a–e** which were characterized by EA, IR, and ^{1}H NMR spectra. The IR absorptions of the NH_2 group and carboxylate group disappeared. The ^{1}H NMR spectra were similar to that of the corresponding compounds **1a–e**[8] except that the C-3 proton on the substituted Cp ring shifted to a higher field owing to the shield effect of the PPh_3 group. Compound (−)-(S_p)-**3a–e** remained optically active and maintained the same absolute configuration in the ferrocene moiety as that of compound (−)-(S_p,S)-**2a–e**, which was confirmed by single crystal X-ray analysis of compound (−)-(S_p)-**3e** (Fig. 3).

As shown in Figs. 2 and 3, both ferrocene moieties have *S* configurations and the amino acid moiety in (−)-(S_p,S)-**2b** also has *S* configuration. Each platinum atom in the metallocycle is in a slightly distorted square-planar coordination environment. The angles between adjacent atoms in the coordination sphere of platinum lie in the range of 80.2–99.3° for (−)-(S_p,S)-**2b** and 80.0–96.7° for (−)-(S_p)-**3e**. The angles N(1)–Pt–P(1) and C(1)–Pt–Cl(1) in (−)-(S_p)-**3e** are 171.7° and 169.4°, respectively, which have larger distortion than the angles of 175.0° for C(9)–Pt–N(2) and of 174.4° for N(1)–Pt–O(1) in (−)-(S_p,S)-**2b**, due to the steric interaction between the PPh_3 group and substituted Cp ring. In both structures, the

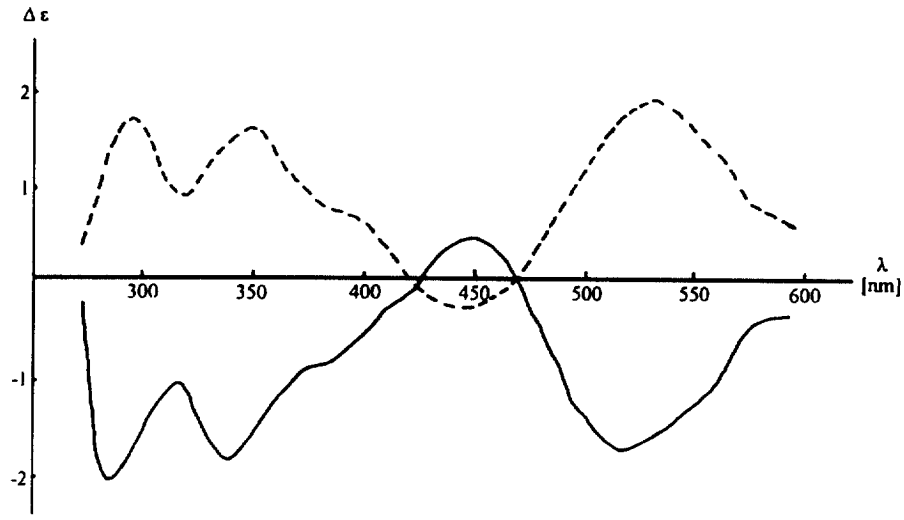

Figure 1. CD spectra of methanol solutions of (−)-(S_p,S)-**2b** (—) and (+)-(R_p,S)-**2b** (- - -)

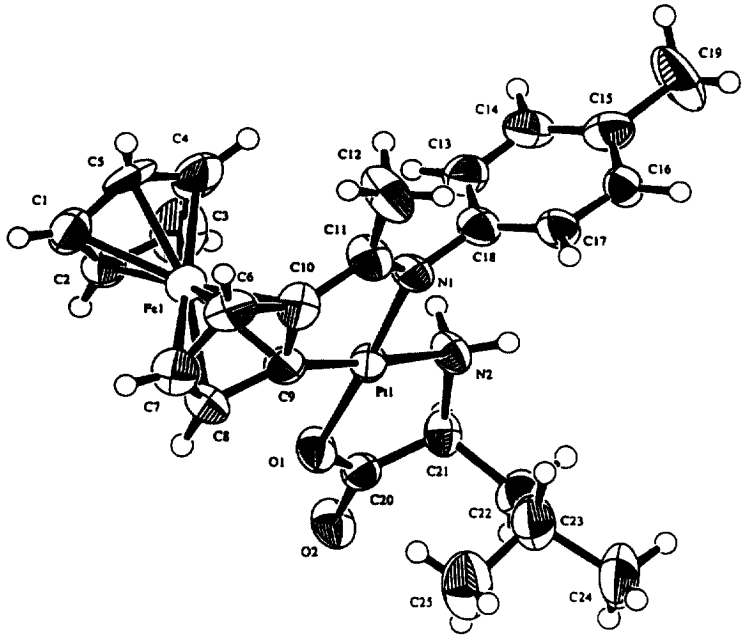

Figure 2. ORTEP view of the molecular structure of compound (−)-(S_p,S)-**2a**. Selected bond lengths (Å) and angle (°): Pt–C(9) 1.96(2), Pt–O(1) 2.02(1), Pt–N(1) 2.03(1), Pt–N(2) 2.15(1), N(1)–C(11) 1.32(2), N(2)–C(21) 1.53(2), O(1)–C(20) 1.29(2), C(9)–C(10) 1.44(2), C(10)–C(11) 1.42(3), N(1)–Pt–C(9) 80.8(6), Pt–C(9)–C(10) 113(1), C(9)–C(10)–C(11) 114(1), N(1)–C(11)–C(10) 115(1), Pt–N(1)–C(11) 115(1), O(1)–Pt–N(2) 80.2(5), Pt–N(2)–C(21) 106.2(9), N(2)–C(21)–C(20) 110(1), O(1)–C(20)–C(21) 120(1), Pt–O(1)–C(20) 116(1), O(1)–Pt–N(1) 174.4(6), O(1)–Pt–C(9) 99.3(6), N(1)–Pt–N(2) 99.1(5), N(2)–Pt–C(9) 170.5(7)

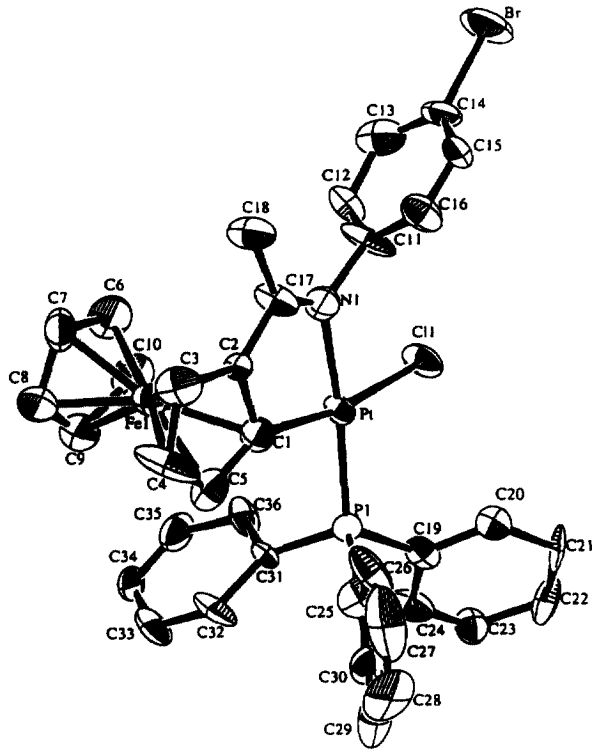

Figure 3. ORTEP view of the molecular structure of compound (−)-(S_p)-**3b**. Selected bond lengths (Å) and angle (°): Pt–C(1) 2.05(2), Pt–Cl 2.365(5), Pt–N 2.15(1), Pt–P 2.206(5), C(1)–C(2) 1.50(2), C(2)–C(17) 1.43(3), N–C(17) 1.29(3), N–Pt–C(1) 80.0(8), Pt–C(1)–C(2) 109(1), C(1)–C(2)–C(17) 119(1), N–C(17)–C(2) 114(1), Pt–N–C(17) 116(1), Cl–Pt–P 96.9(2), P–Pt–C(1) 92.0(6), Cl–Pt–N(1) 91.3(4), Cl–Pt–C(1) 169.4(6), P–Pt–N 171.7(4)

cyclopentadienyl rings are planar and nearly parallel to each other [tilt angle: 1.54° for (−)-(S_p,S)-**2b** and 3.01° for (−)-(S_p)-**3e**] and the N-phenyl ring has a large angle with the substituted Cp ring [dihedral angle: 74.09° for (−)-(S_p,S)-**2b** and 103.35° for (−)-(S_p)-**3e**]. In the structure of (−)-(S_p,S)-**2b**, the two five-member metallocycles are approximately co-planar with the dihedral angle of 2.15°. Owing to the significant *trans* effect of the σ-Pt–C functionality, the Pt–N(2) length (2.15 Å) is larger than Pt–N(1) (2.03 Å) in (−)-(S_p,S)-**2b**.

3. Experimental

Melting points were determined on a WC-1 microscopic apparatus and were uncorrected. Optical rotations were measured in $CHCl_3$ in a 1 dm cell at 20°C with a Perkin–Elmer Model 341 polarimeter. Elemental analyses were determined with a Carlo Erba 1160 elemental analyzer. ^1H NMR were recorded on a Bruker DPX 400 instrument using DMSO-d_6 or $CDCl_3$ as the solvent and TMS as an internal standard. CD spectra were recorded on GJASCO J-20C automatic recording spectropolarimeter at 20°C. Cycloplatinated ferrocenylketimines **1** were synthesized according to the literature procedures.[8] All solvents were dried according to the standard methods.

3.1. Preparation of (−)-(S_p,S)-2 and (+)-(R_p,S)-2

A solution of cycloplatinated ferrocenylketimine **1** (0.2 mmol) in dichloromethane was added to a solution of 0.2 mmol of *S*-leucine and an equimolecular amount of anhydrous Na_2CO_3 in methanol. The mixture was stirred overnight at room temperature, followed by evaporation of the solvent in vacuo on a rotatory evaporator at room temperature. The resulting red solid was chromatographed on silica gel TLC plates using dichloromethane:acetone (3.5:1) as developing agent. The two successive purplish bands were collected and washed with methanol, respectively. The first band with higher R_f value contained (−)-(S_p,*S*)-**2** and the second band contained (+)-(R_p,*S*)-**2**. After removal of the methanol, the residue was dissolved in CH_2Cl_2 and filtered. The filtrates were reduced and the solids separated were recrystallized from dichloromethane–*n*-hexane to give (−)-(S_p,*S*)-**2** and (+)-(R_p,*S*)-**2** as red blocks, respectively.

Characterization data for (−)-(S_p,*S*)-**2a**: Yield: 31%. M.p. 140–142°C. $[\alpha]_D^{20}$ −2636.4 (0.0044 in $CHCl_3$). Anal. calcd for $C_{25}H_{30}FeN_2O_3Pt$: C, 45.66; H, 4.60; N, 4.26. Found: C, 45.40; H, 4.81; N, 4.25. Selected ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.97 (s, 3H, CH_3C=N), 3.18 (m, 1H, C*H*NH_2), 3.80 (s, 3H, $C_6H_4OCH_3$), 4.23 (s, 5H, C_5H_5), 4.44 [bs, 1H, $H^3(C_5H_3)$], 4.50 [bs, 1H, $H^4(C_5H_3)$], 4.62 [bs, 1H, $H^5(C_5H_3)$].

For (+)-(R_p,*S*)-**2a**: Yield: 25%. M.p. 116–120°C (dec.). $[\alpha]_D^{20}$ +2825.0 (0.0040 in $CHCl_3$). Anal. calcd for $C_{25}H_{30}FeN_2O_3Pt$: C, 45.66; H, 4.60; N, 4.26. Found: C, 45.57; H, 4.79; N, 4.17. Selected ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.97 (s, 3H, CH_3C=N), 3.14 (m, 1H, C*H*NH_2), 3.79 (s, 3H, $C_6H_4OCH_3$), 4.23 (s, 5H, C_5H_5), 4.46 [bs, 1H, $H^3(C_5H_3)$], 4.50 [bs, 1H, $H^4(C_5H_3)$], 4.62 [bs, 1H, $H^5(C_5H_3)$].

For (−)-(S_p,*S*)-**2b**: Yield: 35%. M.p. 148–150°C (dec.). $[\alpha]_D^{20}$ −3241.0 (0.0054 in $CHCl_3$). Anal. calcd for $C_{25}H_{30}FeN_2O_2Pt$: C, 46.81; H, 4.71; N, 4.37. Found: C, 47.01; H, 4.82; N, 4.24. Selected ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.96 (s, 3H, CH_3C=N), 2.34 (s, 3H, $C_6H_4CH_3$), 3.18 (m, 1H, C*H*NH_2), 4.21 (s, 5H, C_5H_5), 4.45 [bs, 1H, $H^3(C_5H_3)$], 4.51 [bs, 1H, $H^4(C_5H_3)$], 4.63 [d, *J*=2.0 Hz, 1H, $H^5(C_5H_3)$].

For (+)-(R_p,*S*)-**2b**: Yield: 37%. M.p. 112–115°C. $[\alpha]_D^{20}$ +2400.0 (0.0050 in $CHCl_3$). Anal. calcd for $C_{25}H_{30}FeN_2O_2Pt$: C, 46.81; H, 4.71; N, 4.37. Found: C, 46.64; H, 4.79; N, 4.11. Selected ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.96 (s, 3H, CH_3C=N), 2.34 (s, 3H, $C_6H_4CH_3$), 3.13 (m, 1H, C*H*NH_2), 4.23 (s, 5H, C_5H_5), 4.46 [bs, 1H, $H^3(C_5H_3)$], 4.51 [bs, 1H, $H^4(C_5H_3)$], 4.63 [d, *J*=2.0 Hz, 1H, $H^5(C_5H_3)$].

For (−)-(S_p,*S*)-**2c**: Yield: 23%. M.p. 124–126°C (dec.). $[\alpha]_D^{20}$ −2903.2 (0.0050 in $CHCl_3$). Anal. calcd for $C_{24}H_{28}FeN_2O_2Pt$: C, 45.94; H, 4.50; N, 4.47. Found: C, 45.65; H, 4.81; N, 4.13. Selected ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.98 (s, 3H, CH_3C=N), 3.17 (m, 1H, C*H*NH_2), 4.25 (s, 5H, C_5H_5), 4.46 [bs, 1H, $H^3(C_5H_3)$], 4.52 [bs, 1H, $H^4(C_5H_3)$], 4.65 [bs, 1H, $H^5(C_5H_3)$].

For (+)-(R_p,*S*)-**2c**: Yield: 25%. M.p. 132–134°C. $[\alpha]_D^{20}$ +2660.0 (0.0050 in $CHCl_3$). Anal. calcd for $C_{24}H_{28}FeN_2O_2Pt$: C, 45.94; H, 4.50; N, 4.47. Found: C, 45.89; H, 4.67; N, 4.18. Selected ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.98 (s, 3H, CH_3C=N), 3.12 (bs, 1H, C*H*NH_2), 4.24 (s, 5H, C_5H_5), 4.48 [bs, 1H, $H^3(C_5H_3)$], 4.52 [bs, 1H, $H^4(C_5H_3)$], 4.65 [bs, 1H, $H^5(C_5H_3)$].

For (−)-(S_p,*S*)-**2d**: Yield: 23%. M.p. 222–224°C (dec.). $[\alpha]_D^{20}$ −3543.0 (0.0046 in $CHCl_3$). Anal. calcd for $C_{24}H_{27}ClFeN_2O_2Pt$: C, 43.55; H, 4.11; N, 4.23. Found: C, 43.76; H, 4.17; N, 4.26. ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.98 (s, 3H, CH_3C=N), 3.17 (m, 1H, C*H*NH_2), 4.24 (s, 5H, C_5H_5), 4.48 [bs, 1H, $H^3(C_5H_3)$], 4.54 [bs, 1H, $H^4(C_5H_3)$], 4.66 [bs, 1H, $H^5(C_5H_3)$].

For (+)-(R_p,*S*)-**2d**: Yield: 33%. M.p. 112–114°C. $[\alpha]_D^{20}$ +2812.5 (0.0032 in $CHCl_3$). Anal. calcd for $C_{24}H_{27}ClFeN_2O_2Pt$: C, 43.55; H, 4.11; N, 4.23. Found: C, 43.71; H, 3.97; N, 4.44. Selected ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.98 (s, 3H, CH_3C=N), 3.08 (m, 1H, C*H*NH_2), 4.24 (s, 5H, C_5H_5), 4.49 [bs, 1H, $H^3(C_5H_3)$], 4.53 [bs, 1H, $H^4(C_5H_3)$], 4.66 [bs, 1H, $H^5(C_5H_3)$].

For (−)-(S_p,*S*)-**2e**: Yield: 33%. M.p. >200°C (dec.). $[\alpha]_D^{20}$ −3142.8 (0.0028 in $CHCl_3$). Anal. calcd for $C_{25}H_{29}BrFeN_2O_2Pt$: C, 40.81; H, 3.85; N, 3.97. Found: C, 41.10; H, 3.91; N, 3.85. Selected ^{1}H NMR

(400 MHz, DMSO-d_6): δ 1.97 (s, 3H, CH_3C=N), 3.20 (m, 1H, C*H*NH_2), 4.24 (s, 5H, C_5H_5), 4.48 [bs, 1H, $H^3(C_5H_3)$], 4.54 [bs, 1H, $H^4(C_5H_3)$], 4.66 [bs, 1H, $H^5(C_5H_3)$].

For (+)-(R_p,*S*)-**2e**: Yield: 26%. M.p. >200°C (dec.). $[\alpha]_D^{20}$ +2865.7 (0.0034 in $CHCl_3$). Anal. calcd for $C_{25}H_{29}BrFeN_2O_2Pt \cdot 0.5C_6H_{14}$: C, 43.27; H, 4.57; N, 3.74. Found: C, 43.31; H, 4.29; N, 3.69. Selected ^{1}H NMR (400 MHz, DMSO-d_6): δ 1.97 (s, 3H, CH_3C=N), 3.08 (m, 1H, C*H*NH_2), 4.24 (s, 5H, C_5H_5), 4.50 [bs, 1H, $H^3(C_5H_3)$], 4.54 [bs, 1H, $H^4(C_5H_3)$], 4.65 [bs, 1H, $H^5(C_5H_3)$].

3.2. Preparation of (−)-(S_p)-3

(−)-(S_p,*S*)-**2** (10 mg) was added to a solution of equimolecular amounts of PPh_3 and LiCl in dichloromethane:methanol (5:1) and the mixture was stirred at room temperature for about 5 h. After the evaporation of the solvent in vacuo, the residue was purified by passing it through a short silica gel column with dichloromethane as eluent. Concentration of the eluted solution gave (−)-(S_p)-**3** which was recrystallized from dichloromethane–*n*-hexane as red needles.

Characterization data for (−)-(S_p)-**3a**: Yield: 70%. M.p. 224–226°C (dec.). $[\alpha]_D^{20}$ −1873.0 (0.0118 in $CHCl_3$). Anal. calcd for $C_{37}H_{33}ClFeNOPPt$: C, 53.86; H, 4.03; N, 1.70. Found: C, 53.56; H, 4.16; N, 1.51. Selected ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.08 (s, 3H, CH_3C=N), 3.46 [bs, 1H, $H^3(C_5H_3)$], 3.81 (s, 3H, $C_6H_4OCH_3$), 3.91 (s, 5H, C_5H_5), 4.37 [bs, 1H, $H^4(C_5H_3)$], 4.58 [bs, 1H, $H^5(C_5H_3)$].

For (−)-(S_p)-**3b**: Yield: 65%. M.p. 148–150°C (dec.). $[\alpha]_D^{20}$ −1733.0 (0.0105 in $CHCl_3$). Anal. calcd for $C_{37}H_{33}ClFeNPPt$: C, 54.93; H, 4.11; N, 1.73. Found: C, 54.60; H, 4.10; N, 1.58. Selected ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.08 (s, 3H, CH_3C=N), 2.35 (s, 3H, $C_6H_4CH_3$), 3.34 [bs, 1H, $H^3(C_5H_3)$], 3.91 (s, 5H, C_5H_5), 4.23 [bs, 1H, $H^4(C_5H_3)$], 4.47 [bs, 1H, $H^5(C_5H_3)$].

For (−)-(S_p)-**3c**: Yield: 49%. M.p. 244–246°C (dec.). $[\alpha]_D^{20}$ −2150.0 (0.0100 in $CHCl_3$). Anal. calcd for $C_{36}H_{31}ClFeNPPt \cdot 0.5C_6H_{14}$: C, 55.89; H, 4.57; N, 1.67. Found: C, 56.08; H, 4.66; N, 1.59. Selected ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.08 (s, 3H, CH_3C=N), 3.38 [bs, 1H, $H^3(C_5H_3)$], 3.92 (s, 5H, C_5H_5), 4.26 [bs, 1H, $H^4(C_5H_3)$], 4.50 [bs, 1H, $H^5(C_5H_3)$].

For (−)-(S_p)-**3d**: Yield: 68%. M.p. >220°C (dec.). $[\alpha]_D^{20}$ −1953.5 (0.0086 in $CHCl_3$). Anal. calcd for $C_{36}H_{30}Cl_2FeNPPt$: C, 52.13; H, 3.65; N, 1.69. Found: C, 51.91; H, 3.65; N, 1.63. ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.08 (s, 3H, CH_3C=N), 3.39 [bs, 1H, $H^3(C_5H_3)$], 3.90 (s, 5H, C_5H_5), 4.28 [bs, 1H, $H^4(C_5H_3)$], 4.50 [bs, 1H, $H^5(C_5H_3)$].

For (−)-(S_p)-**3e**: Yield: 69%. M.p. 276–278°C (dec.). $[\alpha]_D^{20}$ −1893.6 (0.0084 in $CHCl_3$). Anal. calcd for $C_{36}H_{30}BrClFeNPPt$: C, 49.48; H, 3.46; N, 1.60. Found: C, 49.56; H, 3.53; N, 1.51. ^{1}H NMR (400 MHz, $CDCl_3$): δ 2.08 (s, 3H, CH_3C=N), 3.38 [d, J=2.0 Hz, 1H, $H^3(C_5H_3)$], 3.89 (s, 5H, C_5H_5), 4.27 [t, J=2.4 Hz, 1H, $H^4(C_5H_3)$], 4.49 [d, J=2.4 Hz, 1H, $H^5(C_5H_3)$].

3.3. Crystallography

(−)-(S_p,*S*)-**2b**: $C_{25}H_{30}FeN_2O_2Pt$, M=641.46, monoclinic, space group $P2_1$ (No. 4), a=10.941(9) Å, b=7.614(2) Å, c=15.059(5) Å, β=108.77(6)°, Z=2, Dc=1.793 g cm^{-3}, F(000)=628.00, μ(MoKα)=64.93 cm^{-1}. Of the 2482 reflections collected, 2147 reflections (I>3.00σ(I)) were used for the refinement. The final residuals were R=0.048, R_w=0.062, and GOF=1.21. For (−)-(S_p)-**3e**: $C_{36}H_{30}BrClFeNPPt$, M=873.91, orthorhombic, space group $P2_12_12_1$ (No. 19), a=15.780(5) Å, b=16.438(5) Å, c=12.313(3) Å, Z=4, Dc=1.817 g cm^{-3}, F(000)=1696.00, μ(MoKα)=62.26 cm^{-1}. Of the 3161 reflections collected, 2664 reflections (I>3.00σ(I)) were used for the refinement. The final residuals were R=0.065, R_w=0.090, and GOF=1.22.

Intensity data were collected on a Rigaku RAXIS-IV imaging plate area detector at 288 K using graphite monochromated Mo-Kα radiation (λ=0.71070 Å) to a maximum 2θ value of 55.0°. The data were corrected for Lorentz and polarization effects and also for secondary excitation. The structures were solved by direct methods and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. All calculations were performed using the teXsan crystallographic software package of Molecular Structure Corporation. [9] The absolute configuration of (−)-(S_p)-**3e** was confirmed by the significance of the difference between the two sigma weighted R factors, which was judged by the Hamilton test.[10]

Acknowledgements

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