Chiral Norbornadienes as Efficient Ligands for the Rhodium-Catalyzed Asymmetric 1,4-Addition of Arylboronic Acids to Fumaric and Maleic Compounds

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Supporting Information

I. General

All manipulations were carried out with standard Schlenk techniques under nitrogen.

1,4-Dioxane and Et₂O were distilled over benzophenone ketyl under nitrogen.

N-methylmaleimide (Aldrich), *N*-benzylmaleimide (ICN Biomedicals), phenylboronic acid (TCI), and LiAlH₄ (Wako Chemicals) were used as received. [RhCl(C₂H₄)₂]₂¹ and (1*R*, 4*R*)-2,5-dibenzylbicyclo[2.2.1]hepta-2,5-diene (**5a**)² were prepared following the literature procedures. (1*R*, 4*R*)-2,5-bis(2,4,6-trimethylphenylmethyl)bicyclo[2.2.1]hepta-2,5-diene (**5b**) was synthesized following the procedure for (*R,R*)-**5a**. *N*-cyclohexylmaleimide³ and di-*tert*-butyl fumarate⁴ were synthesized following the literature procedures.

All other chemicals and solvents were purchased from Aldrich, Wako Chemicals, TCI, or Kanto Chemicals and used as received.

II. Spectra Data for (*R,R*)-**5b** and Its Synthetic Intermediates

(1*R*,4*R*)-Spiro[5-(2,4,6-trimethylphenylmethyl)bicyclo[2.2.1]hepta-5-ene-2,2'-[1,3]dioxolane]

¹H NMR (CDCl₃): δ 6.82 (s, 2H), 5.24 (d, ³J_{HH} = 1.6 Hz, 1H), 3.98-3.77 (m, 4H), 3.44 (dd, ²J_{HH} = 17.1 Hz and ⁴J_{HH} = 1.8 Hz, 1H), 3.31 (dd, ²J_{HH} = 17.1 Hz and ⁴J_{HH} = 1.8 Hz, 1H), 2.59-2.55 (m, 1H), 2.54-2.52 (m, 1H), 2.25 (s, 3H), 2.20 (s, 6H), 1.87 (dd, ²J_{HH} = 12.3 Hz and ³J_{HH} = 3.8 Hz, 1H), 1.70 (d, ²J_{HH} = 8.6 Hz, 1H), 1.64 (ddt, ²J_{HH} = 8.6 Hz, ³J_{HH} = 3.5 Hz, and ⁴J_{HH} = 1.8 Hz, 1H), 1.46 (dd, ²J_{HH} = 12.3 Hz and ³J_{HH} = 3.5 Hz, 1H). ¹³C NMR (CDCl₃): δ 151.0, 136.7, 135.2, 132.6, 128.7, 125.3, 119.4, 64.7, 64.3, 49.6, 48.3, 44.1, 39.8, 30.3, 20.9, 19.7. Anal. Calcd for C₁₉H₂₄O₂: C, 80.24; H, 8.51. Found: C, 80.41; H, 8.79.

¹ Cramer, R. *Inorg. Synth.* **1974**, *15*, 16.

² Hayashi, T.; Ueyama, K.; Tokunaga, N.; Yoshida, K. *J. Am. Chem. Soc.* **2003**, *125*, 11508.

³ Kita, Y.; Tobo, T.; Sakamoto, K.; Baba, M.; Nakagawa, Y. *Jpn. Kokai Tokkyo Koho* **1988**, *7*.

⁴ Tsuge, O.; Kanemasa, S.; Takenaka, S. *Bull. Chem. Soc., Jpn.* **1985**, *58*, 3137.

(1R,4R)-2-(2,4,6-Trimethylphenylmethyl)[2.2.1]hepta-2-en-5-one

^1H NMR (CDCl_3): δ 6.82 (s, 2H), 5.23-5.20 (m, 1H), 3.52 (dd, $^2J_{\text{HH}} = 17.6$ Hz and $^4J_{\text{HH}} = 2.0$ Hz, 1H), 3.35 (dd, $^2J_{\text{HH}} = 17.6$ Hz and $^4J_{\text{HH}} = 2.0$ Hz, 1H), 2.92-2.89 (m, 2H), 2.25 (s, 3H), 2.18 (s, 6H), 2.13 (ddt, $^2J_{\text{HH}} = 11.0$ Hz, $^3J_{\text{HH}} = 4.4$ Hz, and $^4J_{\text{HH}} = 2.4$ Hz, 1H), 1.98 (dd, $^2J_{\text{HH}} = 16.5$ Hz and $^3J_{\text{HH}} = 3.7$ Hz, 1H), 1.92 (d, $^2J_{\text{HH}} = 11.0$ Hz, 1H), 1.78 (dd, $^2J_{\text{HH}} = 16.5$ Hz and $^3J_{\text{HH}} = 4.4$ Hz, 1H). ^{13}C NMR (CDCl_3): δ 214.5, 155.7, 136.4, 135.7, 131.3, 128.8, 122.0, 55.9, 49.6, 43.2, 37.0, 30.8, 20.8, 19.7. Anal. Calcd for $\text{C}_{17}\text{H}_{20}\text{O}$: C, 84.96; H, 8.39. Found: C, 85.11; H, 8.39.

(1R,4R)-2-(2,4,6-trimethylphenylmethyl)-5-trifluoromethanesulfonyloxybicyclo[2.2.1]hepta-2,5-diene

^1H NMR (CDCl_3): δ 6.82 (s, 2H), 6.30-6.27 (m, 1H), 5.95-5.92 (m, 1H), 3.53 (d, $^2J_{\text{HH}} = 17.6$ Hz, 1H), 3.40 (d, $^2J_{\text{HH}} = 17.6$ Hz, 1H), 3.33-3.28 (m, 2H), 2.37-2.34 (m, 1H), 2.25 (s, 3H), 2.20-2.17 (m, 1H), 2.16 (s, 6H). ^{13}C NMR (CDCl_3): δ 168.2, 157.7, 136.4, 135.5, 132.2, 132.1, 128.8, 124.2, 118.6 (q, $^1J_{\text{CF}} = 320$ Hz), 72.1, 51.9, 51.4, 31.3, 20.9, 19.6. Anal. Calcd for $\text{C}_{18}\text{H}_{19}\text{F}_3\text{O}_3\text{S}$: C, 58.05; H, 5.14. Found: C, 58.00; H, 5.15.

(1R,4R)-2,5-Di(2,4,6-trimethylphenylmethyl)bicyclo[2.2.1]hepta-2,5-diene (5b)

^1H NMR (CDCl_3): δ 6.80 (s, 4H), 5.72 (dt, $^3J_{\text{HH}} = 3.9$ Hz and $^4J_{\text{HH}} = 1.9$ Hz, 2H), 3.46 (dd, $^2J_{\text{HH}} = 17.4$ Hz and $^4J_{\text{HH}} = 1.9$ Hz, 2H), 3.27 (dd, $^2J_{\text{HH}} = 17.4$ Hz and $^4J_{\text{HH}} = 1.9$ Hz, 2H), 3.15-3.12 (m, 2H), 2.25 (s, 6H), 2.14 (s, 12H), 1.98-1.96 (m, 2H). ^{13}C NMR (CDCl_3): δ 157.1, 136.4, 135.0, 133.5, 132.5, 128.5, 71.5, 53.5, 31.6, 20.9, 19.7. $[\alpha]_D^{20} -77.3$ (c 1.00, CHCl_3). Anal. Calcd for $\text{C}_{27}\text{H}_{32}$: C, 90.95; H, 9.05. Found: C, 91.95; H, 9.16.

III. 1,4-Addition Reactions

General Procedure for Table 1.

A solution of $[\text{RhCl}(\text{C}_2\text{H}_4)_2]_2$ (1.9 mg, 9.8 μmol Rh) and chiral ligand (11 μmol) in 1,4-dioxane (1.0 mL) was stirred for 10 min at room temperature. KOH (0.20 mL, 20 μmol ; 0.1 M aqueous) was then added to it, and the resulting solution was stirred for additional 10 min at room temperature. After addition of $\text{ArB}(\text{OH})_2$ (0.60 mmol) and stirring for 5 min, this mixture was transferred to a solution of di-*tert*-butyl fumarate (45.6 mg, 0.20 mmol) in 1,4-dioxane (1.0 mL). The resulting mixture was stirred for 3 h

at 50 °C, and was then passed through a pad of silica gel with Et₂O, and the solvent was removed under vacuum. The residue was purified by silica gel PTLC with EtOAc/hexane afford the desired 1,4-adduct **6**.

Table 1, entry 6 (6a). (CAS 159528-25-3) Pale yellow oil. 90% yield. The ee was determined on a Daicel Chiralpak AD-H column with hexane : isopropanol = 95 : 5, flow = 1.0 mL/min. Retention times: 8.1 min [(*R*)-enantiomer], 16.6 min [(*S*)-enantiomer]. 90% ee. $[\alpha]_D^{20} +57.4$ (c 0.99, CHCl₃).

¹H NMR (CDCl₃): δ 7.75-7.23 (m, 5H), 3.92 (dd, ³*J*_{HH} = 10.0 Hz and 5.7 Hz, 1H), 3.01 (dd, ²*J*_{HH} = 16.5 Hz and ³*J*_{HH} = 10.0 Hz, 1H), 2.55 (dd, ²*J*_{HH} = 16.5 Hz and ³*J*_{HH} = 5.7 Hz, 1H), 1.40 (s, 9H), 1.39 (s, 9H).

The absolute configuration was determined by comparison of the optical rotation with that reported in the literature,⁵ after derivatization to 2-phenylbutane-1,4-diol **7** (CAS 6837-05-4) following the procedure below:

LiAlH₄ (7.6 mg, 0.20 mmol) was added to a solution of the 1,4-adduct (30.9 mg, 0.10 mmol) in Et₂O (1.0 mL), and the mixture was stirred for 10 h at room temperature. After quenching with a drop of water, this was passed through a pad of silica gel with Et₂O to afford the desired diol as a colorless oil (8.4 mg, 0.51 mmol; 51% yield). $[\alpha]_D^{20} +28.2$ (c 0.84, MeOH).

¹H NMR (CDCl₃): δ 7.32 (t, ³*J*_{HH} = 7.4 Hz, 2H), 7.27-7.19 (m, 3H), 3.77 (d, ³*J*_{HH} = 6.8 Hz, 2H), 3.68 (dt, ²*J*_{HH} = 10.8 Hz and ³*J*_{HH} = 5.6 Hz, 1H), 3.57 (ddd, ²*J*_{HH} = 10.8 Hz and ³*J*_{HH} = 8.1 Hz and 5.6 Hz, 1H), 2.95 (quint, ³*J*_{HH} = 6.8 Hz, 1H), 2.24 (bs, 2H), 2.02 (ddt, ²*J*_{HH} = 13.8 Hz and ³*J*_{HH} = 12.2 Hz and 5.6 Hz, 1H), 1.89 (ddt, ²*J*_{HH} = 13.8 Hz and ³*J*_{HH} = 8.1 Hz and 5.6 Hz, 1H).

Table 1, entry 7 (6b). Pale yellow oil. 78% yield. The ee was determined on a Daicel Chiralpak AD-H column with hexane : isopropanol = 95 : 5, flow = 1.0 mL/min. Retention times: 13.8 min [(*R*)-enantiomer], 30.1 min [(*S*)-enantiomer]. 86% ee. $[\alpha]_D^{20} +55.7$ (c 1.31, CHCl₃). The absolute configuration was assigned by analogy with Table 2, entry 1.

¹H NMR (CDCl₃): δ 7.19 (d, ³*J*_{HH} = 8.7 Hz, 2H), 6.84 (d, ³*J*_{HH} = 8.7 Hz, 2H), 3.86 (dd, ³*J*_{HH} = 10.0 Hz and 5.8 Hz, 1H), 3.78 (s, 3H), 2.98 (dd, ²*J*_{HH} = 16.4 Hz and ³*J*_{HH} = 10.0 Hz, 1H), 2.52 (dd, ²*J*_{HH} = 16.4 Hz and ³*J*_{HH} = 5.8 Hz, 1H), 1.40 (s, 9H), 1.38 (s, 9H). ¹³C NMR (CDCl₃): δ 172.3, 170.9, 158.7, 130.6, 128.7, 114.0, 80.8, 80.6, 55.2, 47.6, 39.2, 28.0, 27.9. Anal. Calcd for C₁₉H₂₈O₅: C, 67.83; H, 8.39. Found: C, 67.64; H, 8.32.

⁵ Agami, C.; Couty, F.; Evano, G. *Eur. J. Org. Chem.* **2002**, 29.

Table 1, entry 8 (6c). Pale yellow oil. 85% yield. The ee was determined on a Daicel Chiralpak AD-H column with hexane : isopropanol = 95 : 5, flow = 1.0 mL/min. Retention times: 11.0 min [(*R*)-enantiomer], 24.9 min [(*S*)-enantiomer]. 90% ee. $[\alpha]_D^{20} +56.2$ (*c* 0.94, CHCl₃). The absolute configuration was assigned by analogy with Table 2, entry 1.

¹H NMR (CDCl₃): δ 7.24 (dd, ³*J*_{HH} = 8.7 Hz and ³*J*_{HF} = 5.3 Hz, 2H), 6.99 (tt, ³*J*_{HH} = 8.7 Hz and ³*J*_{HF} = 2.2 Hz, 2H), 3.90 (dd, ³*J*_{HH} = 9.5 Hz and 6.1 Hz, 1H), 2.99 (dd, ²*J*_{HH} = 16.5 Hz and ³*J*_{HH} = 9.5 Hz, 1H), 2.54 (dd, ²*J*_{HH} = 16.5 Hz and ³*J*_{HH} = 6.1 Hz, 1H), 1.40 (s, 9H), 1.38 (s, 9H). ¹³C NMR (CDCl₃): δ 171.9, 170.6, 162.0 (d, ¹*J*_{CF} = 245 Hz), 134.2 (d, ⁴*J*_{CF} = 3.6 Hz), 129.3 (d, ³*J*_{CF} = 8.3 Hz), 115.5 (d, ²*J*_{CF} = 21.1 Hz), 81.1, 80.8, 47.7, 39.1, 28.0, 27.9. Anal. Calcd for C₁₈H₂₅FO₄: C, 66.65; H, 7.77. Found: C, 66.53; H, 7.85.

Table 1, entry 9 (6d). White solid. 91% yield. The ee was determined on a Daicel Chiralpak AD-H column with hexane : isopropanol = 95 : 5, flow = 1.0 mL/min. Retention times: 11.6 min [(*R*)-enantiomer], 26.6 min [(*S*)-enantiomer]. 87% ee. $[\alpha]_D^{20} +61.5$ (*c* 1.62, CHCl₃). The absolute configuration was assigned by analogy with Table 2, entry 1.

¹H NMR (CDCl₃): δ 7.84-7.70 (m, 4H), 7.50-7.39 (m, 3H), 4.10 (dd, ³*J*_{HH} = 9.9 Hz and 5.7 Hz, 1H), 3.12 (dd, ²*J*_{HH} = 16.6 Hz and ³*J*_{HH} = 9.9 Hz, 1H), 2.64 (dd, ²*J*_{HH} = 16.6 Hz and ³*J*_{HH} = 5.7 Hz, 1H), 1.40 (s, 9H), 1.39 (s, 9H). ¹³C NMR (CDCl₃): δ 172.1, 170.8, 136.0, 133.5, 132.6, 128.3, 127.8, 127.6, 126.6, 126.1, 125.9, 125.8, 81.1, 80.8, 48.1, 39.1, 28.0, 27.9. Anal. Calcd for C₂₂H₂₈O₄: C, 74.13; H, 7.92. Found: C, 73.84; H, 7.92.

Table 1, entry 10 (6e). Pale yellow oil. 80% yield. The ee was determined on a Daicel Chiralcel OD-H column with hexane : isopropanol = 500 : 1, flow = 1.0 mL/min. Retention times: 8.0 min [(*R*)-enantiomer], 12.2 min [(*S*)-enantiomer]. 91% ee. $[\alpha]_D^{20} +78.8$ (*c* 1.28, CHCl₃). The absolute configuration was assigned by analogy with Table 2, entry 1.

¹H NMR (CDCl₃): δ 7.22-7.08 (m, 4H), 4.21 (dd, ³*J*_{HH} = 9.9 Hz and 5.6 Hz, 1H), 3.00

(dd, $^2J_{\text{HH}} = 16.5$ Hz and $^3J_{\text{HH}} = 9.9$ Hz, 1H), 2.50 (dd, $^2J_{\text{HH}} = 16.5$ Hz and $^3J_{\text{HH}} = 5.6$ Hz, 1H), 2.41 (s, 3H), 1.39 (s, 9H), 1.37 (s, 9H). ^{13}C NMR (CDCl_3): δ 172.4, 171.1, 137.0, 136.1, 130.5, 127.0, 126.5, 126.2, 80.8, 80.6, 44.0, 38.4, 28.0, 27.9, 19.8. Anal. Calcd for $\text{C}_{19}\text{H}_{28}\text{O}_4$: C, 71.22; H, 8.81. Found: C, 71.44; H, 9.03.

General Procedure for Table 2.

A solution of $[\text{RhCl}(\text{C}_2\text{H}_4)_2]_2$ (1.9 mg, 9.8 μmol Rh) and chiral ligand (11 μmol) in 1,4-dioxane (1.0 mL) was stirred for 10 min at room temperature. KOH (0.20 mL, 20 μmol ; 0.1 M aqueous) was then added to it, and the resulting solution was stirred for additional 10 min at room temperature. After addition of $\text{ArB}(\text{OH})_2$ (0.60 mmol) and stirring for 5 min, this mixture was transferred to a solution of maleimide (0.20 mmol) in 1,4-dioxane (1.0 mL). The resulting mixture was stirred for 3 h at 50 $^\circ\text{C}$, and was then passed through a pad of silica gel with Et_2O , and the solvent was removed under vacuum. The residue was purified by silica gel PTLC with EtOAc /hexane to afford the desired 1,4-adduct **8**.

Table 2, entry 6 (8a). (CAS 86-34-0) 88% conversion. The ee was determined on a Daicel Chiralpak AD-H column with hexane : isopropanol = 90 : 10, flow = 1.0 mL/min. Retention times: 15.2 min [(*R*)-enantiomer], 18.1 min [(*S*)-enantiomer]. 85% ee. The absolute configuration was assigned by analogy with Table 2, entry 8.

^1H NMR (CDCl_3): δ 7.36 (t, $^3J_{\text{HH}} = 7.2$ Hz, 2H), 7.30 (tt, $^3J_{\text{HH}} = 7.2$ Hz and $^4J_{\text{HH}} = 1.3$ Hz, 1H), 7.21 (d, $^3J_{\text{HH}} = 7.2$ Hz, 2H), 4.02 (dd, $^3J_{\text{HH}} = 9.5$ Hz and 4.8 Hz, 1H), 3.20 (dd, $^2J_{\text{HH}} = 18.4$ Hz and $^3J_{\text{HH}} = 9.5$ Hz, 1H), 3.06 (s, 3H), 2.83 (dd, $^2J_{\text{HH}} = 18.4$ Hz and $^3J_{\text{HH}} = 4.8$ Hz, 1H). ^{13}C NMR (CDCl_3): δ 177.8, 176.2, 137.1, 129.2, 127.9, 127.4, 45.9, 37.1, 25.2.

Table 2, entry 7 (8b). (CAS 139477-41-1) White solid. 85% yield. The ee was determined on a Daicel Chiralcel OD-H column with hexane : isopropanol = 90 : 10, flow = 1.0 mL/min. Retention times: 9.3 min [(*S*)-enantiomer], 11.1 min [(*R*)-enantiomer]. 87% ee. $[\alpha]_{\text{D}}^{20} -34.0$ (c 0.82, CHCl_3). The absolute configuration was assigned by analogy with Table 2, entry 8.

^1H NMR (CDCl_3): δ 7.35 (t, $^3J_{\text{HH}} = 7.3$ Hz, 2H), 7.29 (t, $^3J_{\text{HH}} = 7.3$ Hz, 1H), 7.19 (d, $^3J_{\text{HH}} = 7.3$ Hz, 2H), 4.03 (tt, $^3J_{\text{HH}} = 12.7$ Hz and 3.7 Hz, 1H), 3.93 (dd, $^3J_{\text{HH}} = 9.7$ Hz and 4.5 Hz, 1H), 3.12 (dd, $^2J_{\text{HH}} = 18.4$ Hz and $^3J_{\text{HH}} = 9.7$ Hz, 1H), 2.74 (dd, $^2J_{\text{HH}} = 18.4$ Hz and $^3J_{\text{HH}} = 4.5$ Hz, 1H), 2.24-2.13 (m, 2H), 1.88-1.79 (m, 2H), 1.70-1.58 (m, 3H), 1.41-1.15 (m, 3H). ^{13}C NMR (CDCl_3): δ 177.8, 176.3, 138.0, 129.1, 127.8, 127.2, 52.0, 45.6, 37.1, 28.9, 28.7, 25.84, 25.82, 25.0.

Table 2, entry 8 (8c). (CAS 42856-57-5) White solid. 88% yield. The ee was determined on a Daicel Chiralcel OD-H column with hexane : isopropanol = 90 : 10, flow = 1.0 mL/min. Retention times: 21.0 min [(*S*)-enantiomer], 25.7 min [(*R*)-enantiomer]. 69% ee. $[\alpha]_D^{20}$ -33.7 (*c* 1.20, CHCl₃). The absolute configuration was determined by comparison of the optical rotation with that reported in the literature.⁶

¹H NMR (CDCl₃): δ 7.40 (d, ³*J*_{HH} = 6.6 Hz, 2H), 7.36-7.26 (m, 6H), 7.15 (d, ³*J*_{HH} = 6.8 Hz, 2H), 4.72 (d, ²*J*_{HH} = 14.1 Hz, 1H), 4.70 (d, ²*J*_{HH} = 14.1 Hz, 1H), 4.01 (dd, ³*J*_{HH} = 9.6 Hz and 4.8 Hz, 1H), 3.19 (dd, ²*J*_{HH} = 18.5 Hz and ³*J*_{HH} = 9.6 Hz, 1H), 2.81 (dd, ²*J*_{HH} = 18.5 Hz and ³*J*_{HH} = 4.8 Hz, 1H). ¹³C NMR (CDCl₃): δ 177.4, 175.7, 137.2, 135.8, 129.2, 128.8, 128.7, 128.0, 127.9, 127.4, 45.9, 42.8, 37.2.

Table 2, entry 9 (8d). White solid. 77% yield. The ee was determined on a Daicel Chiralcel OD-H column with hexane : isopropanol = 90 : 10, flow = 1.0 mL/min. Retention times: 13.2 min [(*R*)-enantiomer], 16.0 min [(*S*)-enantiomer]. 84% ee. $[\alpha]_D^{20}$ -34.6 (*c* 1.13, CHCl₃). The absolute configuration was assigned by analogy with Table 2, entry 8.

¹H NMR (CDCl₃): δ 7.10 (d, ³*J*_{HH} = 8.7 Hz, 2H), 6.88 (d, ³*J*_{HH} = 8.7 Hz, 2H), 4.02 (tt, ³*J*_{HH} = 12.4 Hz and 3.6 Hz, 1H), 3.87 (dd, ³*J*_{HH} = 9.6 Hz and 4.6 Hz, 1H), 3.79 (s, 3H), 3.10 (dd, ²*J*_{HH} = 18.4 Hz and ³*J*_{HH} = 9.6 Hz, 1H), 2.70 (dd, ²*J*_{HH} = 18.4 Hz and ³*J*_{HH} = 4.6 Hz, 1H), 2.24-2.12 (m, 2H), 1.87-1.78 (m, 2H), 1.69-1.57 (m, 3H), 1.37-1.16 (m, 3H). ¹³C NMR (CDCl₃): δ 178.1, 176.4, 159.2, 129.7, 128.3, 114.6, 55.3, 52.0, 44.8, 37.2, 28.9, 28.8, 25.84, 25.82, 25.0. Anal. Calcd for C₁₇H₂₁NO₃: C, 71.06; H, 7.37. Found: C, 70.88; H, 7.41.

Table 2, entry 10 (8e). White solid. 88% yield. The ee was determined on a Daicel Chiralpak AD-H column with hexane : isopropanol = 90 : 10, flow = 1.0 mL/min. Retention times: 13.4 min [(*R*)-enantiomer], 53.5 min [(*S*)-enantiomer]. 86% ee. $[\alpha]_D^{20}$ -36.5 (*c* 0.96, CHCl₃). The absolute configuration was assigned by analogy with Table 2, entry 8.

¹H NMR (CDCl₃): δ 7.17 (dd, ³*J*_{HH} = 8.6 Hz and ³*J*_{HF} = 5.2 Hz, 2H), 7.05 (t, ³*J* = 8.6 Hz, 2H), 4.03 (tt, ³*J*_{HH} = 12.5 Hz and 3.7 Hz, 1H), 3.92 (dd, ³*J*_{HH} = 9.7 Hz and 4.7 Hz, 1H), 3.12

⁶ Bettoni, G.; Franchini, C.; Morlacchi, F.; Tangari, N.; Tortorella, V. *J. Org. Chem.* **1976**, *41*, 2780.

(dd, $^2J_{\text{HH}} = 18.4$ Hz and $^3J_{\text{HH}} = 9.7$ Hz, 1H), 2.70 (dd, $^2J_{\text{HH}} = 18.4$ Hz and $^3J_{\text{HH}} = 4.7$ Hz, 1H), 2.23-2.13 (m, 1H), 1.87-1.79 (m, 2H), 1.69-1.58 (m, 3H), 1.38-1.16 (m, 3H). ^{13}C NMR (CDCl_3): δ 177.6, 176.0, 162.2 (d, $^1J_{\text{CF}} = 246$ Hz), 133.4 (d, $^4J_{\text{CF}} = 3.6$ Hz), 128.9 (d, $^3J_{\text{CF}} = 8.1$ Hz), 116.1 (d, $^2J_{\text{CF}} = 21.6$ Hz), 52.1, 44.8, 37.0, 28.9, 28.8, 25.82, 25.80, 25.0. Anal. Calcd for $\text{C}_{16}\text{H}_{18}\text{FNO}_2$: C, 69.80; H, 6.59. Found: C, 69.71; H, 6.59.

Table 2, entry 11 (8f). White solid. 92% yield. The ee was determined on a Daicel Chiralpak AD-H column with hexane : isopropanol = 90 : 10, flow = 1.0 mL/min. Retention times: 19.1 min [(*R*)-enantiomer], 56.6 min [(*S*)-enantiomer]. 82% ee. $[\alpha]_{\text{D}}^{20} -26.5$ (c 1.00, CHCl_3). The absolute configuration was assigned by analogy with Table 2, entry 8.

^1H NMR (CDCl_3): δ 7.32-7.27 (m, 2H), 7.21-7.18 (m, 1H), 7.10-7.06 (m, 1H), 4.03 (tt, $^3J_{\text{HH}} = 12.4$ Hz and 3.6 Hz, 1H), 3.91 (dd, $^3J_{\text{HH}} = 9.7$ Hz and 4.7 Hz, 1H), 3.14 (dd, $^2J_{\text{HH}} = 18.4$ Hz and $^3J_{\text{HH}} = 9.7$ Hz, 1H), 2.72 (dd, $^2J_{\text{HH}} = 18.4$ Hz and $^3J_{\text{HH}} = 4.7$ Hz, 1H), 2.24-2.13 (m, 2H), 1.88-1.80 (m, 2H), 1.66-1.56 (m, 3H), 1.38-1.17 (m, 3H). ^{13}C NMR (CDCl_3): δ 177.1, 175.8, 139.5, 135.0, 130.4, 128.1, 127.7, 125.4, 52.2, 45.2, 36.9, 28.9, 28.8, 25.87, 25.85, 25.0. Anal. Calcd for $\text{C}_{16}\text{H}_{18}\text{ClNO}_2$: C, 65.86; H, 6.22. Found: C, 65.61; H, 6.17.

Table 2, entry 12 (8g). White solid. 93% yield. The ee was determined on a Daicel Chiralpak AD-H column with hexane : isopropanol = 90 : 10, flow = 1.0 mL/min. Retention times: 19.6 min [(*R*)-enantiomer], 96.7 min [(*S*)-enantiomer]. 84% ee. $[\alpha]_{\text{D}}^{20} -46.4$ (c 1.11, CHCl_3). The absolute configuration was assigned by analogy with Table 2, entry 8.

^1H NMR (CDCl_3): δ 7.86-7.76 (m, 3H), 7.67 (s, 1H), 7.52-7.45 (m, 2H), 7.28-7.23 (m, 1H), 4.14-4.02 (m, 2H), 3.19 (dd, $^2J_{\text{HH}} = 18.4$ Hz and $^3J_{\text{HH}} = 9.6$ Hz, 1H), 2.84 (dd, $^2J_{\text{HH}} = 18.4$ Hz and $^3J_{\text{HH}} = 4.4$ Hz, 1H), 2.28-2.15 (m, 2H), 1.89-1.80 (m, 2H), 1.72-1.62 (m, 3H), 1.40-1.17 (m, 3H). ^{13}C NMR (CDCl_3): δ 177.8, 176.3, 135.0, 133.4, 132.8, 129.3, 127.8, 127.7, 126.6, 126.5, 126.3, 124.7, 52.2, 45.8, 37.1, 29.0, 28.8, 25.89, 25.87, 25.0. Anal. Calcd for $\text{C}_{20}\text{H}_{21}\text{NO}_2$: C, 78.15; H, 6.89. Found: C, 77.96; H, 6.69.

Table 2, entry 13 (8h). Colorless viscous oil. 95% yield. The ee was determined on a Daicel Chiralpak AD-H column with hexane : isopropanol = 95 : 5, flow = 0.5

mL/min. Retention time: 37.8 min [(*R*)-enantiomer], 90.3 min [(*S*)-enantiomer]. 92% ee. $[\alpha]_D^{20} +17.4$ (c 0.91, CHCl₃). The absolute configuration was assigned by analogy with Table 2, entry 8.

¹H NMR (CDCl₃): δ 7.22-7.15 (m, 3H), 7.00-6.95 (m, 1H), 4.15 (dd, ³*J*_{HH} = 9.7 Hz and 4.8 Hz, 1H), 4.07 (tt, ³*J*_{HH} = 12.4 Hz and 3.8 Hz, 1H), 3.14 (dd, ²*J*_{HH} = 18.4 Hz and ³*J*_{HH} = 9.7 Hz, 1H), 2.61 (dd, ²*J*_{HH} = 18.4 Hz and ³*J*_{HH} = 4.8 Hz, 1H), 2.38 (s, 3H), 2.27-2.16 (m, 2H), 1.89-1.80 (m, 2H), 1.71-1.61 (m, 3H), 1.40-1.18 (m, 3H). ¹³C NMR (CDCl₃): δ 178.3, 176.3, 136.7, 136.3, 131.1, 127.8, 126.8, 126.4, 52.1, 42.8, 37.0, 28.9, 28.8, 25.91, 25.89, 25.1, 19.8. Anal. Calcd for C₁₇H₂₁NO₂: C, 75.25; H, 7.80. Found: C, 75.00; H, 7.76.

IV. X-ray Crystal Structure of [RhCl((*R,R*)-5a)]₂

Data Collection

A yellow prism crystal of C₄₂H₄₀Cl₂Rh₂ having approximate dimensions of 0.15 x 0.10 x 0.05 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3° oscillations that were exposed for 90 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a C-centered monoclinic cell with dimensions:

$$\begin{aligned} a &= 28.551(9) \text{ \AA} \\ b &= 5.731(2) \text{ \AA} \quad \beta = 107.15(2)^\circ \\ c &= 12.165(3) \text{ \AA} \\ V &= 1901.9(1) \text{ \AA}^3 \end{aligned}$$

For Z = 2 and F.W. = 821.50, the calculated density is 1.43 g/cm³. Based on the systematic absences of:

$$hkl: h+k \pm 2n$$

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

$$C2 \text{ (\#5)}$$

The data were collected at a temperature of $-150 \pm 1^\circ \text{C}$ to a maximum 2θ value of 54.9°. A total of 44 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0° in 5.0° step, at χ=45.0° and φ = 0.0°. The exposure rate was 150.0 [sec./°]. A second sweep was performed using ω scans from 0.0 to 160.0° in 5.0° step, at χ=45.0° and φ = 180.0°. The exposure rate was 150.0 [sec./°]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 9385 reflections that were collected, 2401 were unique ($R_{\text{int}} = 0.033$); equivalent reflections were merged.

The linear absorption coefficient, μ , for Mo-K α radiation is 10.3 cm^{-1} . The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods⁷ and expanded using Fourier techniques.⁸ Some non-hydrogen atoms were refined anisotropically, while the rest were refined isotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement⁹ on F was based on 2009 observed reflections ($I > 3.00\sigma(I)$) and 224 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o| = 0.038$$

$$R_w = [\Sigma w (|F_o| - |F_c|)^2 / \Sigma w F_o^2]^{1/2} = 0.063$$

The standard deviation of an observation of unit weight¹⁰ was 0.88. Unit weights were used. Plots of $\Sigma w (|F_o| - |F_c|)^2$ versus $|F_o|$, reflection order in data collection, $\sin \theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 3.01 and $-0.43 \text{ e}^-/\text{\AA}^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber.¹¹ Anomalous dispersion effects were included in F_{calc} ;¹² the values for $\Delta f'$ and $\Delta f''$ were those of Creagh and McAuley.¹³ The values for the mass attenuation coefficients are those of Creagh and Hubbell.¹⁴ All calculations were performed using the CrystalStructure^{15,16} crystallographic software package.

⁷ SIR92: Altomare, A.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A.; Burla, M.; Polidori, G.; Camalli, M. *J. Appl. Cryst.* **1994**, *27*, 435.

⁸ DIRDIF99: Beurskens, P. T.; Admiraal, G.; Beurskens, G.; Bosman, W. P.; de Gelder, R.; Israel, R.; Smits, J. M. M. The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands (1999).

⁹ Least Squares function minimized:

$$\Sigma w (|F_o| - |F_c|)^2 \text{ where } w = \text{Least Squares weights.}$$

¹⁰ Standard deviation of an observation of unit weight:

$$[\Sigma w (|F_o| - |F_c|)^2 / (N_o - N_v)]^{1/2}$$

$$\text{where: } N_o = \text{number of observations, } N_v = \text{number of variables}$$

¹¹ Cromer, D. T.; Waber, J. T. "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

¹² Ibers, J. A.; Hamilton, W. C. *Acta Crystallogr.* **1964**, *17*, 781.

¹³ Creagh, D. C.; McAuley, W. J. "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219–222 (1992).

¹⁴ Creagh, D. C.; Hubbell, J. H. "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200–206 (1992).

¹⁵ CrystalStructure 3.00: Crystal Structure Analysis Package, Rigaku and Rigaku/MSK (2000–2002).

¹⁶ CRYSTALS Issue 10: Watkin, D. J.; Prout, C. K.; Carruthers, J. R.; Betteridge, P. W. Chemical Crystallography Laboratory, Oxford, UK.

Experimental Details

A. Crystal Data

Empirical Formula	$C_{42}H_{40}Cl_2Rh_2$
Formula Weight	821.50
Crystal Color, Habit	yellow, prism
Crystal Dimensions	0.15 X 0.10 X 0.05 mm
Crystal System	monoclinic
Lattice Type	C-centered
Indexing Images	3 oscillations @ 90.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	$a = 28.551(9) \text{ \AA}$ $b = 5.731(2) \text{ \AA}$ $c = 12.165(3) \text{ \AA}$ $\beta = 107.15(2)^\circ$ $V = 1901.9(1) \text{ \AA}^3$
Space Group	C2 (#5)
Z value	2
D _{calc}	1.434 g/cm ³
F ₀₀₀	832.00
$\mu(\text{MoK}\alpha)$	10.33 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoK α ($\lambda = 0.71075 \approx$) graphite monochromated
Detector Aperture	270 mm x 256 mm
Data Images	44 exposures
ω oscillation Range ($\chi=45.0$, $\phi=0.0$)	130.0 - 190.0 $^{\circ}$
Exposure Rate	150.0 sec./ $^{\circ}$
ω oscillation Range ($\chi=45.0$, $\phi=180.0$)	0.0 - 160.0 $^{\circ}$
Exposure Rate	150.0 sec./ $^{\circ}$
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	54.9 $^{\circ}$
No. of Reflections Measured	Total: 9385
Corrections	Unique: 2401 ($R_{\text{int}} = 0.033$) Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F
Function Minimized	$\sum w (F_o - F_c)^2$
Least Squares Weights	1
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>3.00σ(I))	2009
No. Variables	224
Reflection/Parameter Ratio	8.97
Residuals: R (I>3.00σ(I))	0.038
Residuals: Rw (I>3.00σ(I))	0.063
Goodness of Fit Indicator	0.88
Max Shift/Error in Final Cycle	0.00
Maximum peak in Final Diff. Map	3.01 e ⁻ /Å ³
Minimum peak in Final Diff. Map	-0.43 e ⁻ /Å ³

Table 1. Atomic coordinates and Biso/Beq

atom	x	y	z	Beq
Rh(1)	-0.05482(3)	-0.0271(8)	-0.06182(6)	1.17(1)
Cl(1)	-0.01118(9)	-0.1858(7)	0.1236(2)	1.55(5)
C(4)	-0.1029(4)	0.051(2)	-0.2257(9)	1.5(2)
C(1)	-0.1012(4)	0.209(2)	-0.0090(9)	1.4(2)
C(2)	-0.1275(3)	0.010(2)	-0.0577(8)	1.3(2)
C(7)	-0.1592(4)	0.326(2)	-0.187(1)	1.8(2)
C(11)	-0.2150(4)	0.511(3)	0.1121(9)	2.1(3)
C(9)	-0.1288(4)	0.432(2)	0.1377(9)	1.3(2)
C(14)	-0.1179(4)	0.639(2)	0.2007(9)	1.7(2)
C(16)	-0.1401(4)	-0.005(3)	-0.4352(8)	1.5(2)
C(13)	-0.1546(4)	0.779(2)	0.220(1)	2.1(2)
C(21)	-0.1826(4)	-0.137(2)	-0.4763(9)	2.0(2)
C(20)	-0.2208(4)	-0.061(3)	-0.5698(9)	2.2(3)
C(19)	-0.2164(5)	0.149(3)	-0.623(1)	2.5(3)
C(6)	-0.1064(4)	0.380(2)	-0.1099(9)	1.5(2)
C(10)	-0.1789(4)	0.369(2)	0.0950(9)	1.9(2)
C(5)	-0.0765(4)	0.252(2)	-0.1761(9)	1.4(2)
C(15)	-0.1000(4)	-0.087(2)	-0.3272(9)	1.8(2)
C(8)	-0.0887(4)	0.283(2)	0.1144(9)	1.5(2)
C(18)	-0.1729(5)	0.279(2)	-0.584(1)	2.6(3)
C(3)	-0.1502(4)	0.061(2)	-0.1888(9)	1.5(2)
C(12)	-0.2031(4)	0.715(2)	0.174(1)	2.2(2)
C(17)	-0.1362(5)	0.201(2)	-0.490(1)	2.4(2)
H(7)	-0.0847(4)	0.147(2)	0.1609(9)	1.7(2)
H(8)	-0.0590(4)	0.369(2)	0.1340(9)	1.7(2)
H(15)	-0.0687(4)	-0.068(2)	-0.3383(9)	2.1(3)
H(14)	-0.1052(4)	-0.247(2)	-0.3141(9)	2.1(3)
H(2)	-0.1769(4)	-0.034(2)	-0.2303(9)	1.9(2)
H(4)	-0.0982(4)	0.538(2)	-0.0911(9)	1.8(2)
H(18)	-0.2425(5)	0.202(3)	-0.686(1)	2.9(3)
H(19)	-0.2501(4)	-0.150(3)	-0.5961(9)	2.6(4)
H(5)	-0.1837(4)	0.366(2)	-0.152(1)	2.2(2)
H(6)	-0.1668(4)	0.396(2)	-0.261(1)	2.2(2)
H(13)	-0.0846(4)	0.685(2)	0.2311(9)	2.1(2)
H(12)	-0.1465(4)	0.918(2)	0.265(1)	2.7(3)
H(11)	-0.2283(4)	0.811(2)	0.186(1)	2.7(3)
H(20)	-0.1859(4)	-0.280(2)	-0.4403(9)	2.4(3)
H(16)	-0.1074(5)	0.292(2)	-0.461(1)	2.8(3)
H(17)	-0.1686(5)	0.418(2)	-0.622(1)	3.2(3)
H(10)	-0.2484(4)	0.469(3)	0.0802(9)	2.5(4)
H(9)	-0.1877(4)	0.227(2)	0.0541(9)	2.2(2)
H(3)	-0.0545(4)	0.330(2)	-0.2090(9)	1.7(2)
H(1)	-0.1423(3)	-0.092(2)	-0.0161(8)	1.6(3)

$$B_{eq} = 8/3 \pi^2 (U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos \gamma + 2U_{13}(aa^*cc^*)\cos \beta + 2U_{23}(bb^*cc^*)\cos \alpha)$$

Table 2. Anisotropic Displacement Parameters

atom	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Rh(1)	0.0124(3)	0.0144(3)	0.0171(3)	0.0000(6)	0.0039(2)	-0.0008(6)
Cl(1)	0.014(1)	0.022(1)	0.022(1)	-0.000(1)	0.0046(9)	0.005(1)
C(4)	0.014(4)	0.021(5)	0.021(5)	0.004(4)	0.003(4)	0.002(4)
C(1)	0.013(4)	0.019(5)	0.022(5)	0.002(4)	0.005(4)	-0.001(5)
C(2)	0.013(4)	0.017(9)	0.020(4)	0.003(4)	0.005(3)	0.005(5)
C(11)	0.022(5)	0.04(1)	0.022(5)	0.009(6)	0.006(4)	0.002(6)
C(9)	0.018(4)	0.012(8)	0.020(4)	-0.001(4)	0.006(4)	0.001(4)
C(14)	0.021(5)	0.021(6)	0.024(5)	-0.003(5)	0.006(4)	-0.000(5)
C(16)	0.022(4)	0.019(7)	0.016(4)	0.001(6)	0.008(3)	-0.003(6)
C(13)	0.038(6)	0.022(6)	0.024(6)	0.002(5)	0.015(5)	-0.003(5)
C(21)	0.029(6)	0.026(6)	0.023(5)	0.004(5)	0.009(5)	0.000(5)
C(20)	0.020(5)	0.04(1)	0.023(5)	0.000(6)	0.003(4)	-0.001(6)
C(19)	0.030(6)	0.041(8)	0.020(5)	0.016(6)	0.004(5)	0.001(6)
C(6)	0.019(5)	0.015(5)	0.023(5)	0.002(4)	0.007(4)	0.000(4)
C(10)	0.019(5)	0.030(6)	0.021(5)	-0.001(5)	0.005(4)	-0.004(5)
C(5)	0.015(5)	0.020(6)	0.020(5)	0.000(4)	0.005(4)	0.004(4)
C(15)	0.018(5)	0.027(8)	0.024(5)	0.002(4)	0.005(4)	-0.002(4)
C(8)	0.014(4)	0.020(6)	0.021(5)	0.000(4)	0.003(4)	-0.001(5)
C(18)	0.055(8)	0.024(7)	0.024(6)	0.008(6)	0.015(6)	0.003(5)
C(3)	0.016(5)	0.020(5)	0.024(5)	-0.004(4)	0.007(4)	-0.003(4)
C(12)	0.028(6)	0.035(7)	0.023(5)	0.014(6)	0.012(5)	0.002(5)
C(17)	0.031(6)	0.028(7)	0.030(6)	-0.003(6)	0.007(5)	-0.001(6)

The general temperature factor expression: $\exp(-2\pi^2(a^2U_{11}h^2 + b^2U_{22}k^2 + c^2U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 3. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Rh(1)	Cl(1)	2.410(3)	Rh(1)	C(4)	2.11(1)
Rh(1)	C(1)	2.12(1)	Rh(1)	C(2)	2.101(9)
Rh(1)	C(5)	2.09(1)	C(4)	C(5)	1.41(2)
C(4)	C(15)	1.49(2)	C(4)	C(3)	1.54(1)
C(1)	C(2)	1.40(2)	C(1)	C(6)	1.54(2)
C(1)	C(8)	1.50(1)	C(2)	C(3)	1.56(1)
C(7)	C(6)	1.55(1)	C(7)	C(3)	1.54(2)
C(11)	C(10)	1.38(2)	C(11)	C(12)	1.38(2)
C(9)	C(14)	1.40(2)	C(9)	C(10)	1.42(1)
C(9)	C(8)	1.52(1)	C(14)	C(13)	1.40(2)
C(16)	C(21)	1.39(2)	C(16)	C(15)	1.54(1)
C(16)	C(17)	1.38(2)	C(13)	C(12)	1.38(2)
C(21)	C(20)	1.39(2)	C(20)	C(19)	1.39(2)
C(19)	C(18)	1.41(2)	C(6)	C(5)	1.52(1)
C(18)	C(17)	1.38(2)			

Table 4. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
Rh(1)	Cl(1)	2.410(3)	Rh(1)	C(4)	2.11(1)
Rh(1)	C(1)	2.12(1)	Rh(1)	C(2)	2.101(9)
Rh(1)	C(5)	2.09(1)	C(2)	H(1)	0.95(2)
C(7)	H(5)	0.95(2)	C(7)	H(6)	0.95(2)
C(11)	H(10)	0.95(2)	C(14)	H(13)	0.95(2)
C(13)	H(12)	0.95(2)	C(21)	H(20)	0.95(2)
C(20)	H(19)	0.95(2)	C(19)	H(18)	0.95(2)
C(6)	H(4)	0.95(2)	C(10)	H(9)	0.95(2)
C(5)	H(3)	0.95(2)	C(15)	H(15)	0.95(2)
C(15)	H(14)	0.95(2)	C(8)	H(7)	0.95(2)
C(8)	H(8)	0.95(1)	C(18)	H(17)	0.95(2)
C(3)	H(2)	0.95(2)	C(12)	H(11)	0.95(2)
C(17)	H(16)	0.95(2)			

Table 5. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
Cl(1)	Rh(1)	C(4)	167.9(4)	Cl(1)	Rh(1)	C(1)	98.9(3)
C(4)	Rh(1)	C(1)	81.9(4)	Cl(1)	Rh(1)	C(2)	104.9(3)
C(4)	Rh(1)	C(2)	68.2(4)	C(1)	Rh(1)	C(2)	38.7(4)
Cl(1)	Rh(1)	C(5)	151.8(3)	C(4)	Rh(1)	C(5)	39.2(4)
C(1)	Rh(1)	C(5)	67.9(4)	C(2)	Rh(1)	C(5)	80.7(4)
Rh(1)	C(4)	C(5)	69.7(6)	Rh(1)	C(4)	C(15)	119.4(7)
C(5)	C(4)	C(15)	129.5(1)	Rh(1)	C(4)	C(3)	96.5(7)
C(5)	C(4)	C(3)	104.5(9)	C(15)	C(4)	C(3)	121.7(9)
Rh(1)	C(1)	C(2)	69.8(6)	Rh(1)	C(1)	C(6)	94.9(6)
C(2)	C(1)	C(6)	105.5(9)	Rh(1)	C(1)	C(8)	119.8(7)
C(2)	C(1)	C(8)	126.4(1)	C(6)	C(1)	C(8)	124.0(1)
Rh(1)	C(2)	C(1)	71.5(6)	Rh(1)	C(2)	C(3)	96.3(6)
C(1)	C(2)	C(3)	106.8(1)	C(6)	C(7)	C(3)	93.8(9)
C(10)	C(11)	C(12)	120.6(1)	C(14)	C(9)	C(10)	117.0(1)
C(14)	C(9)	C(8)	121.5(9)	C(10)	C(9)	C(8)	121.5(9)
C(9)	C(14)	C(13)	121.6(1)	C(21)	C(16)	C(15)	119.5(1)
C(21)	C(16)	C(17)	118.6(1)	C(15)	C(16)	C(17)	121.8(1)
C(14)	C(13)	C(12)	119.6(1)	C(16)	C(21)	C(20)	121.0(1)
C(21)	C(20)	C(19)	119.6(1)	C(20)	C(19)	C(18)	119.5(1)
C(1)	C(6)	C(7)	101.7(9)	C(1)	C(6)	C(5)	100.4(9)
C(7)	C(6)	C(5)	100.6(8)	C(11)	C(10)	C(9)	121.1(1)
Rh(1)	C(5)	C(4)	71.1(6)	Rh(1)	C(5)	C(6)	96.8(7)
C(4)	C(5)	C(6)	107.8(9)	C(4)	C(15)	C(16)	109.7(1)
C(1)	C(8)	C(9)	112.1(8)	C(19)	C(18)	C(17)	119.4(1)
C(4)	C(3)	C(2)	98.9(8)	C(4)	C(3)	C(7)	101.7(9)
C(2)	C(3)	C(7)	100.8(9)	C(11)	C(12)	C(13)	120.1(1)
C(16)	C(17)	C(18)	121.9(1)				

Table 6. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
Cl(1)	Rh(1)	C(4)	167.9(4)	Cl(1)	Rh(1)	C(1)	98.9(3)
C(4)	Rh(1)	C(1)	81.9(4)	Cl(1)	Rh(1)	C(2)	104.9(3)
C(4)	Rh(1)	C(2)	68.2(4)	C(1)	Rh(1)	C(2)	38.7(4)
Cl(1)	Rh(1)	C(5)	151.8(3)	C(4)	Rh(1)	C(5)	39.2(4)
C(1)	Rh(1)	C(5)	67.9(4)	C(2)	Rh(1)	C(5)	80.7(4)
Rh(1)	C(4)	C(5)	69.7(6)	Rh(1)	C(4)	C(15)	119.4(7)
Rh(1)	C(4)	C(3)	96.5(7)	Rh(1)	C(1)	C(2)	69.8(6)
Rh(1)	C(1)	C(6)	94.9(6)	Rh(1)	C(1)	C(8)	119.8(7)
Rh(1)	C(2)	C(1)	71.5(6)	Rh(1)	C(2)	C(3)	96.3(6)
Rh(1)	C(2)	H(1)	123.2(1)	C(1)	C(2)	H(1)	123.1(1)
C(3)	C(2)	H(1)	122.9(1)	C(6)	C(7)	H(5)	113.3(1)
C(3)	C(7)	H(5)	113.2(1)	C(6)	C(7)	H(6)	113.4(1)
C(3)	C(7)	H(6)	113.0(1)	H(5)	C(7)	H(6)	109.5(2)
C(10)	C(11)	H(10)	119.9(2)	C(12)	C(11)	H(10)	119.5(2)
C(9)	C(14)	H(13)	119.0(1)	C(13)	C(14)	H(13)	119.4(1)
C(14)	C(13)	H(12)	120.5(2)	C(12)	C(13)	H(12)	119.9(2)
C(16)	C(21)	H(20)	119.9(2)	C(20)	C(21)	H(20)	119.0(2)
C(21)	C(20)	H(19)	120.6(2)	C(19)	C(20)	H(19)	119.9(2)
C(20)	C(19)	H(18)	119.8(2)	C(18)	C(19)	H(18)	120.8(2)
C(1)	C(6)	H(4)	117.1(1)	C(7)	C(6)	H(4)	117.3(1)
C(5)	C(6)	H(4)	116.8(1)	C(11)	C(10)	H(9)	119.5(1)
C(9)	C(10)	H(9)	119.4(1)	Rh(1)	C(5)	C(4)	71.1(6)
Rh(1)	C(5)	C(6)	96.8(7)	Rh(1)	C(5)	H(3)	122.5(1)
C(4)	C(5)	H(3)	122.7(1)	C(6)	C(5)	H(3)	122.7(1)
C(4)	C(15)	H(15)	110.1(1)	C(16)	C(15)	H(15)	109.8(1)
C(4)	C(15)	H(14)	108.9(1)	C(16)	C(15)	H(14)	108.9(1)
H(15)	C(15)	H(14)	109.5(1)	C(1)	C(8)	H(7)	108.5(1)
C(9)	C(8)	H(7)	108.5(1)	C(1)	C(8)	H(8)	109.2(1)
C(9)	C(8)	H(8)	109.0(1)	H(7)	C(8)	H(8)	109.5(1)
C(19)	C(18)	H(17)	120.5(2)	C(17)	C(18)	H(17)	120.1(2)
C(4)	C(3)	H(2)	117.3(1)	C(2)	C(3)	H(2)	117.6(1)
C(7)	C(3)	H(2)	117.4(1)	C(11)	C(12)	H(11)	119.9(2)
C(13)	C(12)	H(11)	120.0(2)	C(16)	C(17)	H(16)	118.7(2)
C(18)	C(17)	H(16)	119.4(2)				

Table 7. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
Rh(1)	C(4)	C(5)	Rh(1)	0.0(2)	Rh(1)	C(4)	C(5)	C(6)	-91.2(1)
C(15)	C(4)	C(5)	Rh(1)	-111.8(2)	C(15)	C(4)	C(5)	C(6)	156.9(2)
C(3)	C(4)	C(5)	Rh(1)	91.7(1)	C(3)	C(4)	C(5)	C(6)	0.5(2)
Rh(1)	C(4)	C(15)	C(16)	177.3(9)	C(5)	C(4)	C(15)	C(16)	-95.7(2)
C(3)	C(4)	C(15)	C(16)	57.2(2)	Rh(1)	C(4)	C(3)	C(2)	1.0(1)
Rh(1)	C(4)	C(3)	C(7)	104.1(1)	C(5)	C(4)	C(3)	C(2)	-69.7(2)
C(5)	C(4)	C(3)	C(7)	33.4(2)	C(15)	C(4)	C(3)	C(2)	131.6(2)
C(15)	C(4)	C(3)	C(7)	-125.3(2)	Rh(1)	C(1)	C(2)	Rh(1)	0.0(2)
Rh(1)	C(1)	C(2)	C(3)	-91.2(1)	C(6)	C(1)	C(2)	Rh(1)	89.6(1)
C(6)	C(1)	C(2)	C(3)	-1.6(2)	C(8)	C(1)	C(2)	Rh(1)	-112.8(2)
C(8)	C(1)	C(2)	C(3)	156.0(1)	Rh(1)	C(1)	C(6)	C(7)	105.3(1)
Rh(1)	C(1)	C(6)	C(5)	2.1(1)	C(2)	C(1)	C(6)	C(7)	34.9(2)
C(2)	C(1)	C(6)	C(5)	-68.3(2)	C(8)	C(1)	C(6)	C(7)	-123.4(2)
C(8)	C(1)	C(6)	C(5)	133.4(2)	Rh(1)	C(1)	C(8)	C(9)	-174.2(8)
C(2)	C(1)	C(8)	C(9)	-88.2(2)	C(6)	C(1)	C(8)	C(9)	65.5(2)
Rh(1)	C(2)	C(3)	C(4)	-1.0(1)	Rh(1)	C(2)	C(3)	C(7)	-104.8(1)
C(1)	C(2)	C(3)	C(4)	71.5(2)	C(1)	C(2)	C(3)	C(7)	-32.3(2)
C(3)	C(7)	C(6)	C(1)	-52.0(1)	C(3)	C(7)	C(6)	C(5)	51.1(1)
C(6)	C(7)	C(3)	C(4)	-51.4(1)	C(6)	C(7)	C(3)	C(2)	50.2(1)
C(12)	C(11)	C(10)	C(9)	-1.9(3)	C(10)	C(11)	C(12)	C(13)	-0.1(3)
C(10)	C(9)	C(14)	C(13)	-0.3(2)	C(8)	C(9)	C(14)	C(13)	178.7(1)
C(14)	C(9)	C(10)	C(11)	2.1(2)	C(8)	C(9)	C(10)	C(11)	-176.9(1)
C(14)	C(9)	C(8)	C(1)	-133.2(2)	C(10)	C(9)	C(8)	C(1)	45.8(2)
C(9)	C(14)	C(13)	C(12)	-1.6(2)	C(15)	C(16)	C(21)	C(20)	176.3(1)
C(17)	C(16)	C(21)	C(20)	-0.7(3)	C(21)	C(16)	C(15)	C(4)	-101.6(2)
C(17)	C(16)	C(15)	C(4)	75.3(2)	C(21)	C(16)	C(17)	C(18)	-0.7(3)
C(15)	C(16)	C(17)	C(18)	-177.6(2)	C(14)	C(13)	C(12)	C(11)	1.8(3)
C(16)	C(21)	C(20)	C(19)	0.2(3)	C(21)	C(20)	C(19)	C(18)	1.7(3)
C(20)	C(19)	C(18)	C(17)	-3.0(3)	C(1)	C(6)	C(5)	Rh(1)	-2.1(1)
C(1)	C(6)	C(5)	C(4)	70.2(2)	C(7)	C(6)	C(5)	Rh(1)	-106.3(1)
C(7)	C(6)	C(5)	C(4)	-34.0(2)	C(19)	C(18)	C(17)	C(16)	2.5(3)

Table 8. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	ADC	atom	atom	distance	ADC
RH(1)	CL(1)	2.41(5)	(1)	RH(1)	CL(1)	2.40(5)	(2)
RH(1)	C(4)	2.11(5)	(1)	RH(1)	C(1)	2.12(3)	(1)
RH(1)	C(2)	2.10(2)	(1)	RH(1)	C(7)	3.55(9)	(1)
RH(1)	C(6)	2.73(4)	(1)	RH(1)	C(5)	2.09(3)	(1)
RH(1)	C(15)	3.12(8)	(1)	RH(1)	C(8)	3.15(5)	(1)
RH(1)	C(3)	2.75(8)	(1)	CL(1)	CL(1)	3.253(5)	(2)
CL(1)	C(4)	3.41(1)	(2)	CL(1)	C(1)	3.45(1)	(1)
CL(1)	C(2)	3.58(1)	(1)	CL(1)	C(14)	3.58(1)	(1,1,0,-1)
CL(1)	C(5)	3.47(1)	(2)	CL(1)	C(15)	3.45(1)	(2)
CL(1)	C(8)	3.46(1)	(1)	C(4)	C(1)	2.77(2)	(1)
C(4)	C(2)	2.36(1)	(1)	C(4)	C(7)	2.40(2)	(1)
C(4)	C(16)	2.48(1)	(1)	C(4)	C(21)	3.40(2)	(1)
C(4)	C(6)	2.37(2)	(1)	C(4)	C(5)	1.41(2)	(1)
C(4)	C(15)	1.49(2)	(1)	C(4)	C(3)	1.54(1)	(1)
C(4)	C(17)	3.19(2)	(1)	C(1)	C(2)	1.40(2)	(1)
C(1)	C(7)	2.40(2)	(1)	C(1)	C(9)	2.50(1)	(1)
C(1)	C(6)	1.54(2)	(1)	C(1)	C(10)	3.00(2)	(1)
C(1)	C(5)	2.35(1)	(1)	C(1)	C(8)	1.50(1)	(1)
C(1)	C(3)	2.38(2)	(1)	C(2)	C(7)	2.40(2)	(1)
C(2)	C(9)	3.40(2)	(1)	C(2)	C(6)	2.34(2)	(1)
C(2)	C(10)	3.38(2)	(1)	C(2)	C(5)	2.71(2)	(1)
C(2)	C(8)	2.59(2)	(1)	C(2)	C(3)	1.56(1)	(1)
C(7)	C(6)	1.55(1)	(1)	C(7)	C(5)	2.37(2)	(1)
C(7)	C(3)	1.54(2)	(1)	C(11)	C(9)	2.43(2)	(1)
C(11)	C(14)	2.76(2)	(1)	C(11)	C(13)	2.39(2)	(1)
C(11)	C(10)	1.38(2)	(1)	C(11)	C(12)	1.38(2)	(1)
C(9)	C(14)	1.40(2)	(1)	C(9)	C(13)	2.44(2)	(1)
C(9)	C(6)	3.27(1)	(1)	C(9)	C(10)	1.42(1)	(1)
C(9)	C(8)	1.52(1)	(1)	C(9)	C(12)	2.81(2)	(1)
C(14)	C(13)	1.40(2)	(1)	C(14)	C(10)	2.40(2)	(1)
C(14)	C(8)	2.54(2)	(1)	C(14)	C(12)	2.40(2)	(1)
C(16)	C(21)	1.39(2)	(1)	C(16)	C(20)	2.42(1)	(1)
C(16)	C(19)	2.80(2)	(1)	C(16)	C(5)	3.47(2)	(1)
C(16)	C(15)	1.54(1)	(1)	C(16)	C(18)	2.41(2)	(1)
C(16)	C(3)	3.12(1)	(1)	C(16)	C(17)	1.38(2)	(1)
C(13)	C(10)	2.77(2)	(1)	C(13)	C(12)	1.38(2)	(1)
C(21)	C(20)	1.39(2)	(1)	C(21)	C(19)	2.41(2)	(1)

Table 8. Distances beyond the asymmetric unit out to 3.60 Å (continued)

atom	atom	distance	ADC	atom	atom	distance	ADC
C(21)	C(15)	2.53(2)	(1)	C(21)	C(18)	2.77(2)	(1)
C(21)	C(3)	3.53(2)	(1)	C(21)	C(17)	2.38(2)	(1)
C(20)	C(19)	1.39(2)	(1)	C(20)	C(18)	2.42(2)	(1)
C(20)	C(12)	3.54(2)	(1,1,0,-1,-1)	C(20)	C(17)	2.77(2)	(1)
C(19)	C(18)	1.41(2)	(1)	C(19)	C(12)	3.60(2)	(1,1,0,-1,-1)
C(19)	C(17)	2.40(2)	(1)	C(6)	C(5)	1.52(1)	(1)
C(6)	C(8)	2.68(1)	(1)	C(6)	C(3)	2.26(2)	(1)
C(10)	C(8)	2.56(1)	(1)	C(10)	C(12)	2.39(2)	(1)
C(5)	C(15)	2.62(2)	(1)	C(5)	C(3)	2.34(1)	(1)
C(15)	C(3)	2.65(2)	(1)	C(15)	C(17)	2.55(2)	(1)
C(18)	C(17)	1.38(2)	(1)				

*ADC (S, L, TX, TY, TZ) represents:

S: Symmetry operation number. -S is inversion of operation S.

(1) X,Y,Z

(2) -X,Y,-Z

L: Lattice translation.

(1) 0, 0, 0

(2) 1/2, 1/2, 0

TX, TY, TZ: Unit cell translation along the x, y, and z directions.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	ADC	atom	atom	distance	ADC
RH(1)	CL(1)	2.41(5)	(1)	RH(1)	CL(1)	2.40(5)	(2)
RH(1)	C(4)	2.11(5)	(1)	RH(1)	C(1)	2.12(3)	(1)
RH(1)	C(2)	2.10(2)	(1)	RH(1)	C(7)	3.55(9)	(1)
RH(1)	C(6)	2.73(4)	(1)	RH(1)	C(5)	2.09(3)	(1)
RH(1)	C(15)	3.12(8)	(1)	RH(1)	C(8)	3.15(5)	(1)
RH(1)	C(3)	2.75(8)	(1)	CL(1)	H(7)	2.97(4)	(1)
CL(1)	H(8)	2.91(4)	(1,1,0,-1)	CL(1)	H(8)	3.48(4)	(1)
CL(1)	H(15)	3.0(2)	(2)	CL(1)	H(14)	3.5(3)	(2)
CL(1)	H(4)	3.4(1)	(1,1,0,-1)	CL(1)	H(13)	2.87(9)	(1,1,0,-1)
CL(1)	H(3)	3.3(1)	(2,1,0,-1)	CL(1)	H(3)	3.5(1)	(2)
C(4)	H(15)	2.02(4)	(1)	C(4)	H(14)	2.01(3)	(1)
C(4)	H(2)	2.15(3)	(1)	C(4)	H(4)	3.35(5)	(1,1,0,-1)
C(4)	H(4)	3.22(5)	(1)	C(4)	H(5)	3.26(7)	(1)
C(4)	H(6)	2.64(4)	(1)	C(4)	H(20)	3.5(1)	(1)
C(4)	H(16)	3.15(6)	(1)	C(4)	H(3)	2.08(3)	(1)
C(4)	H(1)	3.18(7)	(1)	C(1)	H(7)	2.01(3)	(1)
C(1)	H(8)	2.02(5)	(1)	C(1)	H(2)	3.2(1)	(1)
C(1)	H(4)	2.15(4)	(1)	C(1)	H(5)	2.64(8)	(1)
C(1)	H(6)	3.3(1)	(1)	C(1)	H(9)	2.79(6)	(1)
C(1)	H(3)	3.18(8)	(1)	C(1)	H(1)	2.08(3)	(1)
C(2)	H(7)	2.69(7)	(1)	C(2)	H(8)	3.3(1)	(1)
C(2)	H(2)	2.17(6)	(1)	C(2)	H(4)	2.89(6)	(1,1,0,-1)
C(2)	H(4)	3.20(6)	(1)	C(2)	H(5)	2.64(5)	(1)
C(2)	H(6)	3.26(7)	(1)	C(2)	H(10)	3.5(1)	(2,2,-1,-1)
C(2)	H(9)	2.78(6)	(1)	C(2)	H(1)	0.95(2)	(1)
C(7)	H(14)	3.49(8)	(1,1,0,1)	C(7)	H(2)	2.15(4)	(1)
C(7)	H(4)	2.16(5)	(1)	C(7)	H(18)	3.5(3)	(2,2,-1,0,-1)
C(7)	H(19)	3.1(3)	(2,2,-1,0,-1)	C(7)	H(5)	0.95(2)	(1)
C(7)	H(6)	0.95(2)	(1)	C(7)	H(11)	3.2(2)	(2,2,-1,-1)
C(7)	H(9)	3.31(7)	(1)	C(7)	H(3)	3.08(5)	(1)
C(7)	H(1)	3.12(6)	(1)	C(11)	H(18)	3.3(2)	(1,1,0,0,1)
C(11)	H(12)	3.25(9)	(1)	C(11)	H(11)	2.03(4)	(1)
C(11)	H(17)	3.2(3)	(1,1,0,0,1)	C(11)	H(10)	0.95(2)	(1)
C(11)	H(10)	3.47(2)	(2,2,-1)	C(11)	H(9)	2.02(4)	(1)
C(11)	H(9)	3.17(4)	(2,2,-1)	C(9)	H(7)	2.03(3)	(1)
C(9)	H(8)	2.04(3)	(1)	C(9)	H(4)	3.21(7)	(1)
C(9)	H(5)	3.4(1)	(1)	C(9)	H(13)	2.04(4)	(1)
C(9)	H(12)	3.44(6)	(1,1,0,-1)	C(9)	H(12)	3.29(6)	(1)
C(9)	H(17)	3.4(2)	(1,1,0,0,1)	C(9)	H(10)	3.29(7)	(1)
C(9)	H(9)	2.06(4)	(1)	C(9)	H(1)	3.50(6)	(1)
C(9)	H(1)	3.26(6)	(1,1,0,1)	C(14)	H(7)	3.06(5)	(1)
C(14)	H(7)	3.14(5)	(1,1,0,1)	C(14)	H(8)	2.59(5)	(1)

Table 9. Distances beyond the asymmetric unit out to 3.60 Å (continued)

atom	atom	distance	ADC	atom	atom	distance	ADC
C(14)	H(13)	0.95(2)	(1)	C(14)	H(12)	2.05(4)	(1)
C(14)	H(11)	3.26(6)	(1)	C(14)	H(17)	3.2(3)	(1,1,0,0,1)
C(14)	H(9)	3.26(8)	(1)	C(14)	H(1)	2.95(9)	(1,1,0,1)
C(16)	H(15)	2.06(5)	(1)	C(16)	H(14)	2.05(5)	(1)
C(16)	H(2)	2.98(7)	(1)	C(16)	H(19)	3.3(1)	(1)
C(16)	H(6)	3.37(7)	(1)	C(16)	H(20)	2.04(4)	(1)
C(16)	H(16)	2.01(5)	(1)	C(16)	H(17)	3.26(7)	(1)
C(13)	H(7)	3.13(8)	(1,1,0,1)	C(13)	H(13)	2.04(4)	(1)
C(13)	H(12)	0.95(2)	(1)	C(13)	H(11)	2.03(4)	(1)
C(13)	H(17)	2.9(2)	(1,1,0,0,1)	C(13)	H(10)	3.2(1)	(1)
C(13)	H(9)	3.23(7)	(1,1,0,1)	C(13)	H(1)	3.1(1)	(1,1,0,1)
C(21)	H(15)	3.2(1)	(1)	C(21)	H(14)	2.57(9)	(1)
C(21)	H(2)	3.01(5)	(1)	C(21)	H(18)	3.3(1)	(1)
C(21)	H(18)	3.4(1)	(2,2,-1,-1,-1)	C(21)	H(19)	2.05(6)	(1)
C(21)	H(20)	0.95(2)	(1)	C(21)	H(16)	3.23(6)	(1)
C(21)	H(17)	3.20(7)	(1,1,0,-1)	C(20)	H(18)	2.04(4)	(1)
C(20)	H(19)	0.95(2)	(1)	C(20)	H(19)	3.36(2)	(2,2,-1,0,-1)
C(20)	H(6)	3.3(1)	(2,2,-1,-1,-1)	C(20)	H(12)	3.3(4)	(1,1,0,-1,-1)
C(20)	H(11)	3.0(2)	(1,1,0,-1,-1)	C(20)	H(20)	2.03(5)	(1)
C(20)	H(20)	3.15(5)	(2,2,-1,0,-1)	C(20)	H(17)	3.48(8)	(1,1,0,-1)
C(20)	H(17)	3.28(8)	(1)	C(19)	H(2)	3.5(1)	(2,2,-1,0,-1)
C(19)	H(18)	0.95(2)	(1)	C(19)	H(19)	2.04(5)	(1)
C(19)	H(19)	3.30(5)	(2,2,-1,0,-1)	C(19)	H(6)	3.5(1)	(2,2,-1,-1,-1)
C(19)	H(12)	3.0(4)	(1,1,0,-1,-1)	C(19)	H(11)	3.0(2)	(1,1,0,-1,-1)
C(19)	H(20)	3.26(7)	(1)	C(19)	H(20)	3.13(7)	(2,2,-1,0,-1)
C(19)	H(16)	3.3(1)	(1)	C(19)	H(17)	2.06(4)	(1)
C(6)	H(7)	3.44(6)	(1)	C(6)	H(8)	2.88(8)	(1)
C(6)	H(14)	3.29(7)	(1,1,0,1)	C(6)	H(2)	3.17(7)	(1)
C(6)	H(4)	0.95(2)	(1)	C(6)	H(5)	2.12(4)	(1)
C(6)	H(6)	2.12(6)	(1)	C(6)	H(9)	3.6(1)	(1)
C(6)	H(3)	2.19(5)	(1)	C(6)	H(1)	3.22(6)	(1)
C(6)	H(1)	3.49(6)	(1,1,0,1)	C(10)	H(7)	2.87(6)	(1)
C(10)	H(8)	3.31(6)	(1)	C(10)	H(5)	2.96(5)	(1)
C(10)	H(13)	3.3(1)	(1)	C(10)	H(12)	3.27(7)	(1,1,0,-1)
C(10)	H(11)	3.25(7)	(1)	C(10)	H(17)	3.4(2)	(1,1,0,0,1)
C(10)	H(10)	2.02(3)	(1)	C(10)	H(10)	3.39(3)	(2,2,-1,-1)
C(10)	H(9)	0.95(2)	(1)	C(10)	H(1)	3.28(6)	(1)
C(5)	H(15)	2.75(4)	(1)	C(5)	H(14)	3.29(6)	(1)
C(5)	H(14)	3.31(6)	(1,1,0,1)	C(5)	H(2)	3.20(6)	(1)
C(5)	H(4)	2.13(4)	(1)	C(5)	H(5)	3.23(6)	(1)
C(5)	H(6)	2.61(6)	(1)	C(5)	H(16)	3.32(8)	(1)
C(5)	H(3)	0.95(2)	(1)	C(15)	H(15)	0.95(2)	(1)

Table 9. Distances beyond the asymmetric unit out to 3.60 Å (continued)

atom	atom	distance	ADC	atom	atom	distance	ADC
C(15)	H(14)	0.95(2)	(1)	C(15)	H(2)	2.80(7)	(1)
C(15)	H(4)	3.58(7)	(1,1,0,-1)	C(15)	H(6)	3.59(7)	(1)
C(15)	H(20)	2.67(7)	(1)	C(15)	H(16)	2.68(5)	(1)
C(15)	H(3)	2.89(6)	(1)	C(8)	H(7)	0.95(2)	(1)
C(8)	H(8)	0.95(1)	(1)	C(8)	H(4)	2.84(5)	(1)
C(8)	H(5)	3.6(2)	(1)	C(8)	H(13)	2.69(5)	(1)
C(8)	H(12)	3.5(1)	(1,1,0,-1)	C(8)	H(9)	2.72(5)	(1)
C(8)	H(1)	2.84(6)	(1)	C(18)	H(18)	2.06(6)	(1)
C(18)	H(19)	3.27(7)	(1)	C(18)	H(19)	3.56(7)	(2,2,-1,0,-1)
C(18)	H(12)	3.0(2)	(1,1,0,-1,-1)	C(18)	H(20)	3.15(7)	(1,1,0,1)
C(18)	H(16)	2.02(6)	(1)	C(18)	H(17)	0.95(2)	(1)
C(3)	H(15)	3.4(1)	(1)	C(3)	H(14)	2.87(6)	(1)
C(3)	H(2)	0.95(2)	(1)	C(3)	H(4)	3.40(6)	(1,1,0,-1)
C(3)	H(4)	3.17(6)	(1)	C(3)	H(5)	2.11(4)	(1)
C(3)	H(6)	2.11(4)	(1)	C(3)	H(20)	3.52(8)	(1)
C(3)	H(10)	3.6(2)	(2,2,-1,-1)	C(3)	H(9)	3.56(9)	(1)
C(3)	H(3)	3.21(5)	(1)	C(3)	H(1)	2.23(4)	(1)
C(12)	H(19)	3.5(3)	(1,1,0,1,1)	C(12)	H(5)	3.28(9)	(2,2,-1)
C(12)	H(13)	3.25(7)	(1)	C(12)	H(12)	2.03(5)	(1)
C(12)	H(11)	0.95(2)	(1)	C(12)	H(17)	2.9(3)	(1,1,0,0,1)
C(12)	H(10)	2.02(5)	(1)	C(12)	H(10)	3.33(5)	(2,2,-1)
C(12)	H(9)	3.25(6)	(1)	C(12)	H(9)	3.36(6)	(1,1,0,1)
C(12)	H(9)	3.51(6)	(2,2,-1)	C(12)	H(1)	3.5(1)	(1,1,0,1)
C(17)	H(15)	2.72(8)	(1)	C(17)	H(14)	3.29(6)	(1)
C(17)	H(18)	3.3(1)	(1)	C(17)	H(6)	3.35(8)	(1)
C(17)	H(12)	3.3(2)	(1,1,0,-1,-1)	C(17)	H(20)	3.24(6)	(1)
C(17)	H(20)	3.42(6)	(1,1,0,1)	C(17)	H(16)	0.95(2)	(1)
C(17)	H(17)	2.03(4)	(1)	C(17)	H(3)	3.6(2)	(1)

*ADC (S, L, TX, TY, TZ) represents:

S: Symmetry operation number. -S is inversion of operation S.

(1) X,Y,Z

(2) -X,Y,-Z

L: Lattice translation.

(1) 0, 0, 0

(2) 1/2, 1/2, 0

TX, TY, TZ: Unit cell translation along the x, y, and z directions.