Rhodium-Catalyzed Asymmetric Synthesis of Indanones: Development of a New “Axially-Chiral” Bisphosphine Ligand

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Supporting Information

I. General

All air- and moisture-sensitive manipulations were carried out with standard Schlenk techniques under nitrogen or in a glove box under argon.

THF was either distilled over benzophenone ketyl under nitrogen or purified by passing through a neutral alumina column under nitrogen. Et₂O was purified by passing through a neutral alumina column under nitrogen. CH₂Cl₂ was distilled over CaH₂ under nitrogen. DMF was distilled over CaH₂ under vacuum. DME was distilled over benzophenone ketyl under nitrogen.

1-Phenyl-2-propyn-1-ol (Wako Chemicals), 2-propyn-1-ol (Nacalai Tesque), 3,4-dihydro-2*H*-pyran (Wako Chemicals), chloro(ethyl)dimethylsilane (Fluoro Chem.), methyl trifluoromethanesulfonate (Kanto Chemicals), tetrabutylammonium fluoride (Aldrich; 1.0 M solution in THF), iodine (Nacalai Tesque), *p*-toluenesulfonic acid (Nacalai Tesque; monohydrate), pyridinium chlorochromate (Aldrich), Cu powder (Acros), LiAlH₄ (Wako Chemicals), *n*-BuLi (Kanto Chemicals; 1.58 M solution in hexane), and *t*-BuLi (Kanto Chemicals; 1.45 M solution in pentane) were used as received.

1-Phenyl-3-triethylsilyl-2-propyn-1-ol (**1a**),¹ 1-phenyl-3-trimethylsilyl-2-propyn-1-ol (**1c**),¹ (*R*)-2-diphenylphosphinyl-2'-methoxy-1,1'-binaphthyl ((*R*)-**4**),² and

¹ Shintani, R.; Okamoto, K.; Hayashi, T. *J. Am. Chem. Soc.* **2005**, *127*, 2872.

² Uozumi, Y.; Tanahashi, A.; Lee, S.-Y.; Hayashi, T. *J. Org. Chem.* **1993**, *58*, 1945.

[Rh(cod)₂]BF₄³ were synthesized following the literature procedures. (*R*)- and (*S*)-1-phenyl-2-propyn-1-ol were obtained by enzymatic kinetic resolution of the racemate, following the literature procedure.⁴

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

II. Synthesis of Substrates

The yields have not been optimized.

1-Phenyl-3-dimethyl(ethyl)silyl-2-propyn-1-ol (1b)

Two drops of HCl (12 M aqueous) were added to a mixture of 1-phenyl-2-propyn-1-ol (2.43 mL, 20.0 mmol) and 3,4-dihydro-2*H*-pyran (2.01 mL, 22.0 mmol), and this was stirred for 2 h at room temperature. The reaction was quenched with Na₂CO₃ and the solid was removed by filtration with Et₂O. The volatiles were removed under vacuum to afford a pale yellow oil. This oil was dissolved in THF (60 mL) and *n*-BuLi (12.3 mL, 19.4 mmol; 1.58 M solution in hexane) was added to it at –40 °C. After stirring for 30 min at –40 °C, chloro(ethyl)dimethylsilane (2.74 mL, 19.5 mmol) was added, and the resulting mixture was stirred for 30 min at –40 °C and for 2 h at room temperature. The reaction was quenched with water and extracted with hexane. The organic layer was dried over MgSO₄, filtered, and concentrated under vacuum to afford a pale yellow oil. This oil was dissolved in MeOH (80 mL) and *p*-toluenesulfonic acid (95.1 mg, 0.500 mmol; monohydrate) was then added to it. The resulting solution was stirred for 12 h at room temperature and the reaction was quenched with NaHCO₃. After removal of MeOH under vacuum, the residue was poured into NaHCO₃ (aqueous, saturated) and extracted with Et₂O. The organic layer was dried over MgSO₄, filtered, and concentrated under vacuum. The residue was chromatographed on silica gel with Et₂O/hexane =

³ Schenck, T. G.; Downes, J. M.; Milne, C. R. C.; MacKenzie, P. B.; Boucher, H.; Whelan, J.; Bosnich, B. *Inorg. Chem.* **1985**, *24*, 2334.

⁴ Raminelli, C.; Comasseto, J. V.; Andrade, L. H.; Porto, A. L. M. *Tetrahedron: Asymmetry* **2004**, *15*, 3117.

1/3 to afford compound **1b** as a pale yellow oil (3.59 g, 16.4 mmol; 85% yield).

^1H NMR (CDCl_3): δ 7.55 (d, $^3J_{\text{HH}} = 7.4$ Hz, 2H), 7.38 (t, $^3J_{\text{HH}} = 6.9$ Hz, 2H), 7.33 (t, $^3J_{\text{HH}} = 7.2$ Hz, 1H), 5.46 (d, $^3J_{\text{HH}} = 6.3$ Hz, 1H), 2.16 (d, $^3J_{\text{HH}} = 6.3$ Hz, 1H), 1.01 (t, $^3J_{\text{HH}} = 7.8$ Hz, 3H), 0.63 (q, $^3J_{\text{HH}} = 7.8$ Hz, 2H), 0.17 (s, 6H). ^{13}C NMR (CDCl_3): δ 140.4, 128.6, 128.4, 126.7, 105.3, 90.8, 65.0, 7.9, 7.3, -2.4. Anal. Calcd for $\text{C}_{13}\text{H}_{18}\text{OSi}$: C, 71.50; H, 8.31. Found: C, 71.42; H, 8.22.

(*R*)-**1b** (98% ee) and (*S*)-**1b** (98% ee) were synthesized in the same way starting from (*S*)-1-phenyl-2-propyn-1-ol and (*R*)-1-phenyl-2-propyn-1-ol, respectively. (*R*)-**1b**: $[\alpha]_{\text{D}}^{20} +21.2$ (*c* 1.00, CHCl_3). (*S*)-**1b**: $[\alpha]_{\text{D}}^{20} -21.5$ (*c* 1.05, CHCl_3).

3-Dimethyl(ethyl)silyl-2-propynal (CAS 3111-95-3)

A solution of 3-dimethyl(ethyl)silyl-2-propyn-1-ol (2.85 g, 20.0 mmol; prepared in the same way as **1b**, starting from 2-propyn-1-ol) in CH_2Cl_2 (25 mL) was added to a mixture of pyridinium chlorochromate (6.47 g, 30.0 mmol) and NaOAc (492 mg, 6.00 mmol) in CH_2Cl_2 (25 mL), and the resulting mixture was stirred for 3 h at room temperature. The reaction mixture was diluted with Et_2O (50 mL) and filtered through celite and then through a pad of florisil. The solvent was removed under vacuum at 0 °C, and the residue was chromatographed on silica gel with Et_2O /pentane = 1/4 to afford 3-dimethyl(ethyl)silyl-2-propynal as a colorless oil (2.05 g, 14.6 mmol; 73% yield).

^1H NMR (CDCl_3): δ 9.16 (s, 1H), 1.00 (t, $^3J_{\text{HH}} = 7.7$ Hz, 3H), 0.67 (q, $^3J_{\text{HH}} = 7.9$ Hz, 2H), 0.21 (s, 6H). ^{13}C NMR (CDCl_3): δ 176.6, 102.6, 102.4, 7.2, 7.0, -3.1.

1-(*p*-Tolyl)-3-dimethyl(ethyl)silyl-2-propyn-1-ol (**1d**)

p-Tolylmagnesium bromide (4.48 mL, 3.60 mmol; 0.80 M solution in THF) was added dropwise over 1 h to a solution of 3-dimethyl(ethyl)silyl-2-propynal (421 mg, 3.00 mmol) in THF (5.0 mL) at -20 °C. The mixture was stirred for 1 h at -20 °C and

for 2 h at room temperature, and the reaction was quenched with HCl (10% aqueous). After extraction with Et₂O, the organic layer was dried over MgSO₄, filtered, and concentrated under vacuum. The residue was chromatographed on silica gel with Et₂O/hexane = 1/3 to afford compound **1d** as a colorless oil (467 mg, 2.01 mmol; 67% yield).

¹H NMR (CDCl₃): δ 7.44 (d, ³J_{HH} = 8.1 Hz, 2H), 7.19 (d, ³J_{HH} = 8.1 Hz, 2H), 5.42 (d, ³J_{HH} = 6.3 Hz, 1H), 2.36 (s, 3H), 2.06 (d, ³J_{HH} = 6.5 Hz, 1H), 1.00 (t, ³J_{HH} = 7.9 Hz, 3H), 0.62 (q, ³J_{HH} = 7.9 Hz, 2H), 0.17 (s, 6H). ¹³C NMR (CDCl₃): δ 138.2, 137.6, 129.3, 126.7, 105.5, 90.5, 64.9, 21.1, 7.9, 7.3, -2.4. Anal. Calcd for C₁₄H₂₀OSi: C, 72.36; H, 8.67. Found: C, 72.43; H, 8.62.

1-(*p*-Anisyl)-3-dimethyl(ethyl)silyl-2-propyn-1-ol (**1e**)

This was synthesized from *p*-anisylmagnesium bromide, following the procedure for compound **1d**. Pale yellow oil, 82% yield.

¹H NMR (CDCl₃): δ 7.47 (d, ³J_{HH} = 8.8 Hz, 2H), 6.91 (d, ³J_{HH} = 8.7 Hz, 2H), 5.41 (d, ³J_{HH} = 6.2 Hz, 1H), 3.82 (s, 3H), 2.05 (d, ³J_{HH} = 6.2 Hz, 1H), 1.01 (t, ³J_{HH} = 7.8 Hz, 3H), 0.63 (q, ³J_{HH} = 7.8 Hz, 2H), 0.17 (s, 6H). ¹³C NMR (CDCl₃): δ 159.7, 132.7, 128.2, 113.9, 105.6, 90.5, 64.6, 55.3, 7.9, 7.3, -2.4. Anal. Calcd for C₁₄H₂₀O₂Si: C, 67.70; H, 8.12. Found: C, 67.85; H, 8.07.

1-(*m*-Tolyl)-3-dimethyl(ethyl)silyl-2-propyn-1-ol (**1f**)

This was synthesized from *m*-tolylmagnesium bromide, following the procedure for compound **1d**. Colorless oil, 84% yield.

¹H NMR (CDCl₃): δ 7.37 (s, 1H), 7.35 (d, ³J_{HH} = 7.6 Hz, 1H), 7.27 (t, ³J_{HH} = 7.5 Hz, 1H), 7.14 (d, ³J_{HH} = 7.4 Hz, 1H), 5.42 (d, ³J_{HH} = 6.2 Hz, 1H), 2.37 (s, 3H), 2.09 (d, ³J_{HH} = 6.2 Hz, 1H), 1.01 (t, ³J_{HH} = 8.0 Hz, 3H), 0.63 (q, ³J_{HH} = 7.9 Hz, 2H), 0.17 (s, 6H). ¹³C

NMR (CDCl₃): δ 140.3, 138.3, 129.1, 128.5, 127.5, 123.8, 105.5, 90.7, 65.1, 21.4, 7.9, 7.2, -2.4. Anal. Calcd for C₁₄H₂₀OSi: C, 72.36; H, 8.67. Found: C, 72.07; H, 8.66.

1-(3,4-Dimethylphenyl)-3-dimethyl(ethyl)silyl-2-propyn-1-ol (1g)

This was synthesized from 3,4-dimethylphenylmagnesium bromide, following the procedure for compound **1d**. Colorless oil, 78% yield.

¹H NMR (CDCl₃): δ 7.33-7.25 (m, 2H), 7.14 (d, ³J_{HH} = 7.7 Hz, 1H), 5.39 (d, ³J_{HH} = 6.1 Hz, 1H), 2.28 (s, 3H), 2.26 (s, 3H), 2.10 (d, ³J_{HH} = 6.2 Hz, 1H), 1.01 (t, ³J_{HH} = 8.0 Hz, 3H), 0.63 (q, ³J_{HH} = 7.8 Hz, 2H), 0.17 (s, 6H). ¹³C NMR (CDCl₃): δ 137.9, 136.71, 136.69, 129.7, 128.0, 124.1, 105.8, 90.2, 64.8, 19.7, 19.4, 7.9, 7.2, -2.4. HRMS (ESI) calcd for C₁₅H₂₂OSiNa (M+Na⁺) 269.1332, found 269.1333.

1-(3,4-Methylenedioxyphenyl)-3-dimethyl(ethyl)silyl-2-propyn-1-ol (1h)

This was synthesized from 3,4-methylenedioxyphenylmagnesium bromide, following the procedure for compound **1h**. Pale yellow oil, 68% yield.

¹H NMR (CDCl₃): δ 7.05 (s, 1H), 7.01 (d, ³J_{HH} = 8.1 Hz, 1H), 6.79 (d, ³J_{HH} = 7.9 Hz, 1H), 5.97 (s, 2H), 5.36 (d, ³J_{HH} = 5.7 Hz, 1H), 2.14 (d, ³J_{HH} = 5.5 Hz, 1H), 1.00 (t, ³J_{HH} = 7.8 Hz, 3H), 0.63 (q, ³J_{HH} = 7.9 Hz, 2H), 0.17 (s, 6H). ¹³C NMR (CDCl₃): δ 147.7, 147.5, 134.5, 120.4, 108.0, 107.4, 105.4, 101.1, 90.4, 64.6, 7.8, 7.2, -2.5. HRMS (ESI) calcd for C₁₄H₁₈O₃SiNa (M+Na⁺) 285.0917, found 285.0918.

III. Synthesis of Ligand (*R,R*)-3

t-BuLi (10.3 mL, 15.0 mmol; 1.45 M solution in pentane) was added to a solution of (*R*)-4 (1.45 g, 2.99 mmol) in THF (150 mL) at -96°C and the mixture was stirred for 2 min. A solution of iodine (2.70 g, 10.6 mmol) in THF (10 mL) was then added to it, and the reaction mixture was warmed to room temperature over 10 min. After addition of Na_2SO_3 (10% aqueous), this was extracted with EtOAc and the organic layer was washed with NaHCO_3 (aqueous, saturated), and then with NaCl (aqueous, saturated). The organic layer was dried over MgSO_4 , filtered, and concentrated under vacuum. The residue was chromatographed on silica gel with $\text{C}_6\text{H}_6/\text{EtOAc} = 1/1$ to afford (*R*)-5 as a brown solid (931 mg, 1.53 mmol; 51% yield).

^1H NMR (CDCl_3): δ 8.80 (d, $^4J_{\text{HH}} = 2.5$ Hz, 1H), 7.75 (d, $^3J_{\text{HH}} = 8.2$ Hz, 1H), 7.59-7.52 (m, 3H), 7.48 (t, $^3J_{\text{HH}} = 7.5$ Hz, 1H), 7.46 (d, $^3J_{\text{HH}} = 9.2$ Hz, 1H), 7.38 (td, $^3J_{\text{HH}} = 7.5$ Hz and $^4J_{\text{HH}} = 1.4$ Hz, 1H), 7.27-7.20 (m, 4H), 7.15 (t, $^3J_{\text{HH}} = 7.8$ Hz, 1H), 7.06-6.97 (m, 3H), 6.93 (td, $^3J_{\text{HH}} = 7.5$ Hz and $^4J_{\text{HH}} = 1.4$ Hz, 1H), 6.84 (d, $^3J_{\text{HH}} = 9.2$ Hz, 1H), 6.75 (d, $^3J_{\text{HH}} = 8.3$ Hz, 1H), 6.73 (td, $^3J_{\text{HH}} = 8.0$ Hz and $^4J_{\text{HH}} = 3.2$ Hz, 2H), 3.67 (s, 3H). ^{13}C NMR (CDCl_3): δ 154.2, 143.1 (d, $J_{\text{CP}} = 9.3$ Hz), 142.3 (d, $J_{\text{CP}} = 9.8$ Hz), 135.5 (d, $J_{\text{CP}} = 2.1$ Hz), 134.2, 133.9 (d, $J_{\text{CP}} = 105.1$ Hz), 133.2 (d, $J_{\text{CP}} = 107.8$ Hz), 132.2 (d, $J_{\text{CP}} = 10.9$ Hz), 131.8 (d, $J_{\text{CP}} = 9.8$ Hz), 131.2, 131.11, 131.09 (d, $J_{\text{CP}} = 10.3$ Hz), 131.09, 130.8 (d, $J_{\text{CP}} = 107.0$ Hz), 128.7, 128.3, 127.7 (d, $J_{\text{CP}} = 12.9$ Hz), 127.5, 127.3 (d, $J_{\text{CP}} = 1.0$ Hz), 127.1, 127.0, 126.7, 126.5 (d, $J_{\text{CP}} = 12.4$ Hz), 125.1, 123.5, 119.1 (d, $J_{\text{CP}} = 5.1$ Hz), 111.7, 94.6 (d, $J_{\text{CP}} = 8.3$ Hz), 55.3. $^{31}\text{P}\{^1\text{H}\}$ NMR (CDCl_3): δ 33.7 (s). $[\alpha]_{\text{D}}^{20} +227$ (*c* 0.56, CHCl_3). Anal. Calcd for $\text{C}_{33}\text{H}_{24}\text{IO}_2\text{P}$: C, 64.93; H, 3.96. Found: C, 64.92; H, 4.12.

A mixture of (*R*)-5 (545 mg, 0.893 mmol) and copper powder (210 mg, 3.31 mmol) in DMF (7.0 mL) was refluxed for 40 h. After removing the solvent under vacuum, the residue was diluted with CHCl₃ and filtered through celite. The solvent was removed under vacuum and the residue was chromatographed on silica gel with EtOAc to afford (*R,R*)-S1 as a pale yellow solid (357 mg, 0.369 mmol; 83% yield).

¹H NMR (CDCl₃): δ 7.61 (dd, ²*J*_{PH} = 11.8 Hz and ³*J*_{HH} = 7.2 Hz, 4H), 7.50 (d, ³*J*_{HH} = 9.2 Hz, 2H), 7.41 (t, *J* = 7.5 Hz, 4H), 7.36 (d, ⁴*J*_{HH} = 3.2 Hz, 2H), 7.33-7.28 (m, 4H), 7.22 (t, ³*J*_{HH} = 7.7 Hz, 2H), 7.20-7.12 (m, 12H), 7.06-7.02 (m, 4H), 6.75-6.72 (m, 4H), 6.55 (td, ³*J*_{HH} = 7.8 Hz and ⁴*J*_{HH} = 2.9 Hz, 4H), 3.79 (s, 6H). ¹³C NMR (CDCl₃): δ 154.4, 142.2 (dd, *J*_{CP} = 8.6 and 2.3 Hz), 139.6 (d, *J*_{CP} = 9.8 Hz), 137.0 (d, *J*_{CP} = 102.5 Hz), 135.2, 134.8 (d, *J*_{CP} = 104.0 Hz), 133.0 (d, *J*_{CP} = 2.1 Hz), 132.6 (d, *J*_{CP} = 9.4 Hz), 132.0 (d, *J*_{CP} = 11.9 Hz), 131.49 (d, *J*_{CP} = 102.5 Hz), 131.46 (d, *J*_{CP} = 9.9 Hz), 131.3 (d, *J*_{CP} = 10.4 Hz), 130.6, 130.1 (d, *J*_{CP} = 2.3 Hz), 128.9, 128.3, 127.9, 127.53, 127.50 (d, *J*_{CP} = 11.9 Hz), 127.0, 126.8, 126.7, 126.6, 126.1, 125.7 (d, *J*_{CP} = 12.9 Hz), 123.3, 120.9 (d, *J*_{CP} = 5.1 Hz), 111.5, 55.4. ³¹P{¹H} NMR (CDCl₃): δ 28.4 (s). [α]_D²⁰ +242 (*c* 0.59, CHCl₃). Anal. Calcd for C₇₂H₅₄O₄P₂ ((*R,R*)-S1•C₆H₆): C, 82.74; H, 5.21. Found: C, 87.70; H, 5.22.

Methyl trifluoromethanesulfonate (294 μL, 2.60 mmol) was added to a solution of (*R,R*)-S1 (418 mg, 0.432 mmol) in DME (26 mL) at room temperature and the mixture was stirred for 3 h at 60 °C. After cooling to 0 °C, LiAlH₄ (246 mg, 6.48 mmol) was added to it and the resulting mixture was stirred for 30 min at 60 °C. The reaction was then quenched by successive addition of water (0.25 mL), NaOH (0.25 mL; 15% aqueous), and water (0.75 mL) at room temperature. This was diluted with C₆H₆ and filtered through celite. The resulting solution was dried over MgSO₄, filtered,

and concentrated under vacuum. The residue was chromatographed on silica gel with C₆H₆ to afford (*R,R*)-**3** as a pale yellow solid (360 mg, 0.385 mmol; 89% yield).

¹H NMR (CDCl₃): δ 7.44-7.36 (m, 6H), 7.31 (bs, 4H), 7.28-7.18 (m, 6H), 7.12-7.06 (m, 8H), 7.03 (t, ³J_{HH} = 7.6 Hz, 4H), 6.97 (d, ³J_{HH} = 9.1 Hz, 2H), 6.76 (d, ³J_{HH} = 8.6 Hz, 2H), 6.72 (bs, 4H), 6.54 (t, ³J_{HH} = 7.3 Hz, 2H), 6.36 (t, ³J_{HH} = 7.5 Hz, 4H), 3.69 (s, 6H). ¹³C NMR (CDCl₃): δ 154.5, 143.4 (t, J_{CP} = 19.3 Hz), 139.0 (t, J_{CP} = 1.6 Hz), 138.1 (t, J_{CP} = 2.6 Hz), 135.8 (t, J_{CP} = 9.4 Hz), 135.0, 134.8 (t, J_{CP} = 11.1 Hz), 134.5 (d, J_{CP} = 8.8 Hz), 133.6, 133.3 (t, J_{CP} = 12.1 Hz), 133.2, 131.4 (t, J_{CP} = 4.9 Hz), 129.7, 128.6, 128.3, 127.7, 127.6, 127.4 (t, J_{CP} = 3.8 Hz), 127.2, 126.6, 126.4 (t, J_{CP} = 3.4 Hz), 126.1, 125.7, 125.6, 125.5, 122.9, 120.8, 111.8, 55.3. ³¹P{¹H} NMR (CDCl₃): δ 1.8 (s). [α]_D²⁰ +488 (c 0.58, CHCl₃). Anal. Calcd for C₆₆H₄₈O₂P₂: C, 84.78; H, 5.17. Found: C, 84.60; H, 5.45.

IV. Catalytic Reactions

General Procedure for Table 1.

A solution of [Rh(cod)₂]BF₄ (4.1 mg, 10 μmol Rh) and ligand (11.0 μmol) in THF (2.5 mL) was stirred for 20 min at 60 °C. Propargyl alcohol **1** (0.20 mmol) and KOH (50 μL, 30 μmol; 0.6 M aqueous) were then added to it. After stirring for 24 h at 60 °C, the reaction mixture was directly passed through a pad of silica gel with Et₂O and the solvent was removed under vacuum. The residue was purified by silica gel preparative TLC with Et₂O/hexane to afford indanone **2**.

Entry 5. (CAS 849518-58-7) Pale yellow oil. 57% yield. The ee was determined on a Daicel Chiralcel OD-H column with hexane : 2-propanol = 95 : 5, flow = 1.0 mL/min. Retention times: 4.8 min [(*R*)-enantiomer], 6.1 min [(*S*)-enantiomer]. 74% ee. [α]_D²⁰ −117 (c 0.74, CHCl₃). The absolute configuration was assigned by analogy with entry 8.

¹H NMR (CDCl₃): δ 7.73 (d, ³J_{HH} = 7.7 Hz, 1H), 7.53 (t, ³J_{HH} = 7.2 Hz, 1H), 7.36 (d, ³J_{HH} = 7.8 Hz, 1H), 7.27 (t, ³J_{HH} = 7.5 Hz, 1H), 2.91 (d, ³J_{HH} = 10.0 Hz, 1H), 2.87 (dd,

$^2J_{\text{HH}} = 18.1$ Hz and $^3J_{\text{HH}} = 8.5$ Hz, 1H), 2.62 (d, $^2J_{\text{HH}} = 18.2$ Hz, 1H), 0.89 (t, $^3J_{\text{HH}} = 7.8$ Hz, 9H), 0.58 (q, $^3J_{\text{HH}} = 8.0$ Hz, 6H). ^{13}C NMR (CDCl_3): δ 207.2, 159.0, 136.2, 134.3, 125.73, 125.68, 124.0, 39.4, 25.3, 7.3, 2.3.

Entry 8. Pale yellow oil. 57% yield. The ee was determined on a Daicel Chiralpak AD-H column with hexane : 2-propanol = 100 : 1, flow = 0.5 mL/min. Retention times: 17.7 min [(*R*)-enantiomer], 18.4 min [(*S*)-enantiomer]. 99% ee. $[\alpha]_{\text{D}}^{20} -155$ (*c* 0.96, CHCl_3). The absolute configuration was determined to be (*S*) by converting it to 1-indanol (see below).

^1H NMR (CDCl_3): δ 7.74 (d, $^3J_{\text{HH}} = 7.5$ Hz, 1H), 7.54 (t, $^3J_{\text{HH}} = 7.5$ Hz, 1H), 7.37 (d, $^3J_{\text{HH}} = 7.8$ Hz, 1H), 7.28 (t, $^3J_{\text{HH}} = 7.7$ Hz, 1H), 2.87 (dd, $^2J_{\text{HH}} = 18.9$ Hz and $^3J_{\text{HH}} = 8.6$ Hz, 1H), 2.81 (d, $^3J_{\text{HH}} = 8.8$ Hz, 1H), 2.58 (dd, $^2J_{\text{HH}} = 18.9$ Hz and $^3J_{\text{HH}} = 1.6$ Hz, 1H), 0.91 (t, $^3J_{\text{HH}} = 7.9$ Hz, 3H), 0.54 (q, $^3J_{\text{HH}} = 7.9$ Hz, 2H), -0.01 (s, 3H), -0.04 (s, 3H). ^{13}C NMR (CDCl_3): δ 207.2, 158.8, 136.2, 134.4, 125.8, 125.7, 124.0, 39.6, 27.6, 7.2, 5.5, -5.1, -5.5. Anal. Calcd for $\text{C}_{13}\text{H}_{18}\text{OSi}$: C, 71.50; H, 8.31. Found: C, 71.77; H, 8.52.

LiAlH_4 (18 mg, 0.47 mmol) was added to a solution of **2b** (102 mg, 0.47 mmol) in Et_2O (3.0 mL) at 0 °C and the mixture was stirred for 30 min at 0 °C. The reaction was quenched with water, and this was passed through a pad of silica gel with Et_2O . After removing the solvent under vacuum, the residue was purified by silica gel preparative TLC with Et_2O /hexane = 1/3 to afford *cis*-**S2** as a pale yellow oil (45 mg, 0.20 mmol; 43% yield) and *trans*-**S2** as a pale yellow oil (14 mg, 64 μmol ; 13% yield).

cis-**S2**: $[\alpha]_{\text{D}}^{20} +42.4$ (*c* 1.64, CHCl_3). ^1H NMR (CDCl_3): δ 7.37 (d, $^3J_{\text{HH}} = 7.0$ Hz, 1H), 7.23-7.20 (m, 1H), 7.18-7.15 (m, 2H), 5.28 (m, 1H), 2.62 (ddd, $^2J_{\text{HH}} = 13.4$ Hz and $^3J_{\text{HH}} = 9.2$ and 7.5 Hz, 1H), 2.52 (dd, $^3J_{\text{HH}} = 8.9$ and 6.6 Hz, 1H), 1.84 (ddd, $^2J_{\text{HH}} = 13.4$ Hz and $^3J_{\text{HH}} = 6.5$ and 4.9 Hz, 1H), 1.64 (d, $^3J_{\text{HH}} = 5.8$ Hz, 1H), 0.96 (t, $^3J_{\text{HH}} = 7.8$ Hz, 3H)

0.65-0.59 (m, 2H), 0.05 (s, 3H), 0.03 (s, 3H). ^{13}C NMR (CDCl_3): δ 146.0, 144.8, 128.3, 125.6, 124.3, 124.1, 76.6, 37.6, 30.7, 7.3, 6.1, -4.6, -4.9. HRMS (ESI) calcd for $\text{C}_{13}\text{H}_{20}\text{OSiNa}$ ($\text{M}+\text{Na}^+$) 243.1176, found 243.1172.

trans-**S2**: $[\alpha]_{\text{D}}^{20} +44.1$ (c 0.65, CHCl_3). ^1H NMR (CDCl_3): δ 7.38 (d, $^3J_{\text{HH}} = 7.3$ Hz, 1H), 7.21 (t, $^3J_{\text{HH}} = 7.3$ Hz, 1H), 7.16 (t, $^3J_{\text{HH}} = 7.3$ Hz, 1H), 7.11 (d, $^3J_{\text{HH}} = 7.5$ Hz, 1H), 5.21 (bs, 1H), 2.66 (dd, $^3J_{\text{HH}} = 9.8$ and 3.4 Hz, 1H), 2.49 (ddd, $^2J_{\text{HH}} = 13.1$ Hz and $^3J_{\text{HH}} = 7.2$ and 3.3 Hz, 1H), 2.13 (ddd, $^2J_{\text{HH}} = 13.0$ Hz and $^3J_{\text{HH}} = 9.5$ and 6.9 Hz, 1H), 1.68 (bs, 1H), 0.92 (t, $^3J_{\text{HH}} = 7.9$ Hz, 3H), 0.54 (q, $^3J_{\text{HH}} = 8.0$ Hz, 2H), -0.04 (s, 3H), -0.05 (s, 3H). ^{13}C NMR (CDCl_3): δ 145.6, 144.1, 128.1, 125.4, 124.1, 124.0, 76.3, 38.8, 30.7, 7.3, 5.8, -4.9, -5.1. HRMS (ESI) calcd for $\text{C}_{13}\text{H}_{20}\text{OSiNa}$ ($\text{M}+\text{Na}^+$) 243.1176, found 243.1173.

TBAF (1.0 mL, 1.0 mmol; 1.0 M solution in THF) was added to a solution of *cis*-**S2** (45 mg, 0.20 mmol) in THF (1.0 mL) at 0 °C and the mixture was stirred for 3 h at room temperature. The reaction was quenched with water and extracted with Et_2O . The organic layer was dried over MgSO_4 , filtered, and concentrated under vacuum. The residue was purified by silica gel preparative TLC with Et_2O /hexane = 1/2 to afford 1-indanol **S3** (CAS 6351-10-6, 697-64-3 for (*R*)) as a white solid (21 mg, 0.15 mmol; 76% yield). $[\alpha]_{\text{D}}^{20} -26.7$ (c 1.03, CHCl_3).

^1H NMR (CDCl_3): δ 7.42 (d, $^3J_{\text{HH}} = 6.0$ Hz, 1H), 7.29-7.22 (m, 3H), 5.25 (t, $^3J_{\text{HH}} = 6.0$ Hz, 1H), 3.06 (ddd, $^2J_{\text{HH}} = 16.0$ Hz and $^3J_{\text{HH}} = 8.5$ and 4.7 Hz, 1H), 2.82 (ddd, $^2J_{\text{HH}} = 15.6$ Hz and $^3J_{\text{HH}} = 8.0$ and 6.8 Hz, 1H), 2.49 (dddd, $^2J_{\text{HH}} = 13.2$ Hz and $^3J_{\text{HH}} = 8.3$, 6.8 , and 4.9 Hz, 1H), 1.95 (dddd, $^2J_{\text{HH}} = 13.6$ Hz and $^3J_{\text{HH}} = 8.5$, 6.6 , and 5.2 Hz, 1H), 1.73 (bs, 1H).

The absolute configuration of **S3** was determined to be (*R*) by comparison of the optical rotation with the literature value ($[\alpha]_{\text{D}}^{20} +34.4$ (c 1.97, CHCl_3) for (*S*)).⁵ This assignment leads to the determination of the absolute configuration of indanone **2b** as (*S*).

⁵ Hüchel, W.; Mossner, F. *Ann.* **1960**, 637, 57.

Entry 9. (CAS 849518-57-6) Pale yellow oil. 44% yield. The ee was determined on a Daicel Chiralpak AD-H column with hexane : 2-propanol = 98 : 2, flow = 0.5 mL/min. Retention times: 18.1 min [(*R*)-enantiomer], 20.2 min [(*S*)-enantiomer]. 92% ee. $[\alpha]_D^{20}$ -155 (*c* 0.64, CHCl₃). The absolute configuration was assigned by analogy with entry 8.

¹H NMR (CDCl₃): δ 7.74 (d, ³*J*_{HH} = 7.7 Hz, 1H), 7.54 (t, ³*J*_{HH} = 7.6 Hz, 1H), 7.37 (d, ³*J*_{HH} = 7.8 Hz, 1H), 7.28 (t, ³*J*_{HH} = 7.7 Hz, 1H), 2.87 (dd, ²*J*_{HH} = 19.4 Hz and ³*J*_{HH} = 8.5 Hz, 1H), 2.79-2.76 (m, 1H), 2.57 (dd, ²*J*_{HH} = 19.3 Hz and ³*J*_{HH} = 2.0 Hz, 1H), 0.02 (s, 9H). ¹³C NMR (CDCl₃): δ 207.1, 158.6, 136.0, 134.3, 125.8, 125.6, 123.9, 39.5, 28.7, -3.2.

Entry 10. Pale yellow oil. 60% yield. The ee was determined on a Daicel Chiralpak AD-H column with hexane : 2-propanol = 100 : 1, flow = 0.5 mL/min. Retention times: 23.8 min [(*R*)-enantiomer], 29.0 min [(*S*)-enantiomer]. 93% ee. $[\alpha]_D^{20}$ -133 (*c* 0.93, CHCl₃). The absolute configuration was assigned by analogy with entry 8.

¹H NMR (CDCl₃): δ 7.61 (d, ³*J*_{HH} = 7.8 Hz, 1H), 7.13 (s, 1H), 7.08 (d, ³*J*_{HH} = 7.8 Hz, 1H), 2.84 (dd, ²*J*_{HH} = 19.1 Hz and ³*J*_{HH} = 8.4 Hz, 1H), 2.73 (d, ³*J*_{HH} = 9.2 Hz, 1H), 2.54 (dd, ²*J*_{HH} = 19.3 Hz and ³*J*_{HH} = 2.1 Hz, 1H), 2.41 (s, 3H), 0.90 (t, ³*J*_{HH} = 7.9 Hz, 3H), 0.53 (q, ³*J*_{HH} = 7.8 Hz, 2H), -0.02 (s, 3H), -0.05 (s, 3H). ¹³C NMR (CDCl₃): δ 206.6, 159.3, 145.5, 133.9, 127.2, 126.0, 123.8, 39.7, 27.3, 22.2, 7.2, 5.5, -5.1, -5.5. Anal. Calcd for C₁₄H₂₀OSi: C, 72.36; H, 8.67. Found: C, 72.21; H, 8.64.

Entry 11. Pale yellow oil. 57% yield. The ee was determined on a Daicel Chiralpak AD-H column with hexane : 2-propanol = 100 : 1, flow = 1.0 mL/min. Retention times: 25.9 min [(*R*)-enantiomer], 34.5 min [(*S*)-enantiomer]. 94% ee. $[\alpha]_{\text{D}}^{20}$ -87.8 (*c* 1.31, CHCl₃). The absolute configuration was assigned by analogy with entry 8.

¹H NMR (CDCl₃): δ 7.66 (d, ³*J*_{HH} = 8.5 Hz, 1H), 6.81 (dd, ³*J*_{HH} = 8.5 Hz and ⁴*J*_{HH} = 2.2 Hz, 1H), 6.77 (d, ⁴*J*_{HH} = 1.8 Hz, 1H), 3.86 (s, 3H), 2.84 (dd, ²*J*_{HH} = 19.1 Hz and ³*J*_{HH} = 8.5 Hz, 1H), 2.74 (d, ³*J*_{HH} = 8.5 Hz, 1H), 2.54 (dd, ²*J*_{HH} = 19.2 Hz and ³*J*_{HH} = 2.0 Hz, 1H), 0.91 (t, ³*J*_{HH} = 7.9 Hz, 3H), 0.54 (q, ³*J*_{HH} = 7.8 Hz, 2H), -0.01 (s, 3H), -0.04 (s, 3H). ¹³C NMR (CDCl₃): δ 205.3, 165.0, 161.6, 129.6, 125.6, 113.5, 109.1, 55.5, 39.7, 27.6, 7.2, 5.5, -5.1, -5.5. Anal. Calcd for C₁₄H₂₀O₂Si: C, 67.70; H, 8.12. Found: C, 67.89; H, 8.34.

Entry 12. Pale yellow oil. 56% yield (>20/1 regioselectivity). The ee was determined on a Daicel Chiralpak AD-H column with hexane : 2-propanol = 100 : 1, flow = 0.5 mL/min. Retention times: 21.1 min [(*S*)-enantiomer], 24.0 min [(*R*)-enantiomer]. 92% ee. $[\alpha]_{\text{D}}^{20}$ -129 (*c* 0.92, CHCl₃). The absolute configuration was assigned by analogy with entry 8.

¹H NMR (CDCl₃): δ 7.53 (s, 1H), 7.35 (dd, ³*J*_{HH} = 7.9 Hz and ⁴*J*_{HH} = 1.6 Hz, 1H), 7.24 (d, ³*J*_{HH} = 7.9 Hz, 1H), 2.85 (dd, ²*J*_{HH} = 19.3 Hz and ³*J*_{HH} = 8.4 Hz, 1H), 2.75 (d, ³*J*_{HH} = 8.5 Hz, 1H), 2.56 (dd, ²*J*_{HH} = 19.3 Hz and ³*J*_{HH} = 1.9 Hz, 1H), 2.38 (s, 3H), 0.91 (t, ³*J*_{HH} = 7.9 Hz, 3H), 0.53 (q, ³*J*_{HH} = 7.9 Hz, 2H), -0.02 (s, 3H), -0.05 (s, 3H). ¹³C NMR (CDCl₃): δ 207.3, 156.1, 136.3, 135.7, 135.6, 125.3, 123.9, 39.8, 27.0, 20.9, 7.2, 5.5, -5.1, -5.5. Anal. Calcd for C₁₄H₂₀OSi: C, 72.36; H, 8.67. Found: C, 72.30; H, 8.61.

Entry 13. Pale yellow oil. 50% yield (>20/1 regioselectivity). The ee was determined on a Daicel Chiralcel OD-H column with hexane : 2-propanol = 98 : 2, flow = 1.0 mL/min. Retention times: 6.9 min [(*R*)-enantiomer], 8.5 min [(*S*)-enantiomer]. 95% ee. $[\alpha]_D^{20}$ -132 (*c* 0.97, CHCl₃). The absolute configuration was assigned by analogy with entry 8.

¹H NMR (CDCl₃): δ 7.49 (s, 1H), 7.11 (s, 1H), 2.82 (dd, ²*J*_{HH} = 19.3 Hz and ³*J*_{HH} = 8.4 Hz, 1H), 2.71 (d, ³*J*_{HH} = 8.7 Hz, 1H), 2.53 (dd, ²*J*_{HH} = 19.3 Hz and ³*J*_{HH} = 2.1 Hz, 1H), 2.33 (s, 3H), 2.28 (s, 3H), 0.91 (t, ³*J*_{HH} = 7.9 Hz, 3H), 0.53 (q, ³*J*_{HH} = 7.8 Hz, 2H), -0.02 (s, 3H), -0.05 (s, 3H). ¹³C NMR (CDCl₃): δ 206.9, 156.9, 144.5, 134.7, 134.4, 126.4, 124.3, 39.7, 26.9, 20.9, 19.6, 7.2, 5.6, -5.1, -5.5. HRMS (ESI) calcd for C₁₅H₂₃OSi (M+H⁺) 247.1513, found 247.1515.

Entry 14. Pale yellow oil. 55% yield (10/1 regioselectivity). The ee was determined on a Daicel Chiralpak AS column with hexane : 2-propanol = 98 : 2, flow = 1.0 mL/min. Retention times: 39.2 min [(*S*)-enantiomer], 62.6 min [(*R*)-enantiomer]. 96% ee. $[\alpha]_D^{20}$ -5.9 (*c* 0.96, CHCl₃). The absolute configuration was assigned by analogy with entry 8.

¹H NMR (CDCl₃): δ 7.37 (d, ³*J*_{HH} = 8.1 Hz, 1H), 6.80 (d, ³*J*_{HH} = 7.9 Hz, 1H), 6.05 (d, ²*J*_{HH} = 18.3 Hz, 1H), 6.04 (d, ²*J*_{HH} = 18.3 Hz, 1H), 2.86 (dd, ²*J*_{HH} = 18.8 Hz and ³*J*_{HH} = 8.7 Hz, 1H), 2.79 (dd, ³*J*_{HH} = 8.7 and 1.5 Hz, 1H), 2.58 (dd, ²*J*_{HH} = 18.9 Hz and ³*J*_{HH} = 1.7 Hz, 1H), 0.90 (t, ³*J*_{HH} = 8.0 Hz, 3H), 0.55 (q, ³*J*_{HH} = 7.8 Hz, 2H), 0.02 (s, 3H), -0.01 (s, 3H). ¹³C NMR (CDCl₃): δ 204.6, 152.3, 143.0, 138.4, 132.3, 119.5, 107.4, 101.5, 40.1, 24.0, 7.2, 5.8, -4.8, -5.2. HRMS (ESI) calcd for C₁₄H₁₉O₃Si (M+H⁺) 263.1098, found 263.1104.

V. X-ray Crystal Structure of [Rh((*R,R*)-**3**)(MeCN)₂]PF₆

Data Collection

A deep red dichloromethane solution of [Rh((*R,R*)-**3**)(MeCN)₂]PF₆ was prepared. Crystals suitable for X-ray analysis were obtained by layering of Et₂O at room temperature.

A red prism crystal of C₇₀H₅₆O₂F₆N₂P₃Rh having approximate dimensions of 0.45 x 0.30 x 0.30 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-K α radiation.

Indexing was performed from 6 oscillations that were exposed for 150 seconds. The crystal-to-detector distance was 127.40 mm.

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

$$a = 11.64(3) \text{ \AA}$$

$$b = 21.09(3) \text{ \AA} \quad \beta = 94.94(8)^\circ$$

$$c = 11.95(2) \text{ \AA}$$

$$V = 2921(8) \text{ \AA}^3$$

For $Z = 2$ and F.W. = 1267.04, the calculated density is 1.44 g/cm³. Based on the systematic absences of:

$$0k0: k \pm 2n$$

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

$$P2_1 \text{ (#4)}$$

The data were collected at a temperature of -150 ± 1 °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 $\chi=45.0^\circ$ and $\phi = 30.0^\circ$. The exposure rate was 30.0 [sec./°]. A second sweep was performed using ω scans from 0.0 to

160.0° in 5.0° step, at $\chi=45.0^\circ$ and $\phi = 180.0^\circ$. The exposure rate was 30.0 [sec./°]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 26569 reflections that were collected, 12810 were unique ($R_{\text{int}} = 0.164$).

The linear absorption coefficient, μ , for Mo-K α radiation is 4.4 cm⁻¹. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods⁶ and expanded using Fourier techniques.⁷ The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement⁸ on F^2 was based on 12810 observed reflections and 758 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma | |Fo| - |Fc| | / \Sigma |Fo| = 0.084$$

$$wR2 = [\Sigma (w (Fo^2 - Fc^2)^2) / \Sigma w(Fo^2)^2]^{1/2} = 0.232$$

The standard deviation of an observation of unit weight⁹ was 0.85. A Sheldrick weighting scheme was used. The maximum and minimum peaks on the final difference Fourier map corresponded to 1.41 and -0.74 e⁻/Å³, respectively.

Neutral atom scattering factors were taken from Cromer and Waber.¹⁰ Anomalous dispersion effects were included in F_{calc} ;¹¹ the values for $\Delta f'$ and $\Delta f''$

⁶ SIR92: Altomare, A.; Cascarano, G.; Giacovazzo, C.; Guagliardi, A.; Burla, M.; Polidori, G.; Camalli, M. *J. Appl. Cryst.* **1994**, 27, 435.

⁷ 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).

⁸ Least Squares function minimized: (SHELXL97)

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

⁹ Standard deviation of an observation of unit weight:

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

where: N_o = number of observations, N_v = number of variables

were those of Creagh and McAuley.¹² The values for the mass attenuation coefficients are those of Creagh and Hubbell.¹³ All calculations were performed using the CrystalStructure^{14,15} crystallographic software package except for refinement, which was performed using SHELXL-97.¹⁶

The crystal structure has been deposited at the Cambridge Crystallographic Data Centre (deposition number: CCDC 284625). The data can be obtained free of charge via the Internet at www.ccdc.cam.ac.uk/conts/retrieving.html.

¹⁰ Cromer, D. T.; Waber, J. T. "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

¹¹ Ibers, J. A.; Hamilton, W. C. *Acta Crystallogr.* **1964**, *17*, 781.

¹² Creagh, D. C.; McAuley, W. J. "International Tables for Crystallography", Vol C, (Wilson, A. J. C., ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219–222 (1992).

¹³ Creagh, D. C.; Hubbell, J. H. "International Tables for Crystallography", Vol C, (Wilson, A. J. C., ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200–206 (1992).

¹⁴ CrystalStructure 3.6.0: Crystal Structure Analysis Package, Rigaku and Rigaku/MSO (2000-2004). 9009 New Trails Dr. The Woodlands TX 77381 USA.

¹⁵ CRYSTALS Issue 10: Watkin, D. J.; Prout, C. K.; Carruthers, J. R.; Betteridge, P. W. Chemical Crystallography Laboratory, Oxford, UK. (1996).

¹⁶ SHELXL97: Sheldrick, G. M. (1997).

Experimental Details

A. Crystal Data

Empirical Formula	$C_{70}H_{56}O_2F_6N_2P_3Rh$
Formula Weight	1267.04
Crystal Color, Habit	red, prism
Crystal Dimensions	0.45 X 0.30 X 0.30 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	6 oscillations @ 150.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	$a = 11.64(3) \text{ \AA}$ $b = 21.09(3) \text{ \AA}$ $c = 11.95(2) \text{ \AA}$ $\beta = 94.94(8)^\circ$ $V = 2921(8) \text{ \AA}^3$
Space Group	$P2_1$ (#4)
Z value	2
D _{calc}	1.440 g/cm ³
F ₀₀₀	1300.00
$\mu(\text{MoK}\alpha)$	4.42 cm ⁻¹

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoK α ($\lambda = 0.71075$ Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	44 exposures
ω oscillation Range ($\chi=45.0$, $\phi=30.0$)	130.0 - 190.0°
Exposure Rate	30.0 sec./°
ω oscillation Range ($\chi=45.0$, $\phi=180.0$)	0.0 - 160.0°
Exposure Rate	30.0 sec./°
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{\max}$	54.9°
No. of Reflections Measured	Total: 26569
Corrections	Unique: 26540 ($R_{\text{int}} = 0.164$) Lorentz-polarization

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	$\Sigma w (F_o^2 - F_c^2)^2$
Least Squares Weights	$w = 1 / [\sigma^2(F_o^2) + (0.1000 \cdot P)^2 + 0.0000 \cdot P]$ where $P = (\text{Max}(F_o^2, 0) + 2F_c^2) / 3$
$2\theta_{\text{max}}$ cutoff	54.9°
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	12810
No. Variables	758
Reflection/Parameter Ratio	16.90
Residuals: R (All reflections)	0.201
Residuals: R1 ($I > 2.00\sigma(I)$)	0.084
Residuals: wR2 (All reflections)	0.232
Goodness of Fit Indicator	0.847
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	1.41 e ⁻ /Å ³
Minimum peak in Final Diff. Map	-0.74 e ⁻ /Å ³