

Supporting information for

**Cobalt(II)-Catalyzed Asymmetric Hydrosilylation of Simple Ketones
Using Dipyridylphosphine Ligands in Air**

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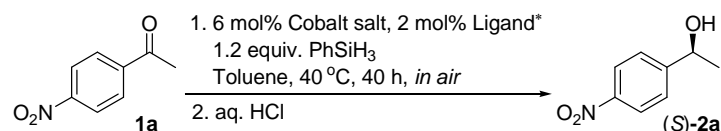
General

^1H NMR and ^{13}C NMR spectra were recorded in CDCl_3 or $(\text{CD}_3)_2\text{CO}$ on Bruker advance 400 (400 MHz) or Bruker advance 500 (500 MHz) at room temperature. Chemical shifts (δ) are given in ppm and are referenced to residual solvent peaks. IR absorption spectra (FT = diffuse reflectance spectroscopy) were recorded for samples loaded as neat films on KBr plates by using a Bruker TENSOR27 and only noteworthy absorptions (in cm^{-1}) are listed. Low resolution mass spectra were obtained with an Agilent Technologies 5975. Enantiomeric excesses of the asymmetric hydrosilylation products were determined by chiral GC or HPLC. Gas chromatographic analyses were conducted on a Fuli 9790 with an FID detector. HPLC analyses were performed using an Agilent 1200 with a UV detector. Optical rotations were measured on a Perkin-Elmer Model 341 polarimeter in a 10 cm cell. Optically pure diphosphine ligands, including (*S*)-P-Phos, (*S*)-Xyl-P-Phos, (*S*)-BINAP, (*S*)-MeO-BIPHEP, (*S*)-(*R*)-Josiphos, (*S,S*)-Me-Duphos and (*S,S*)-DIOP, were purchased from Strem or Aldrich. (*S*)-Tol-P-Phos was prepared according to previous reported procedure.¹ 4 Å Molecular sieves were purchased from Across and activated under 300 °C prior to use. Cobalt salts, phenylsilane, and ketone substrates were purchased from Aldrich, Alfa aesar, Acros or TCI and used as received without further purification unless otherwise stated.

A Typical Procedure of Asymmetric Hydrosilylation in Air (Table 1, entry 6)

$\text{Co}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (11.3 mg, 4.5×10^{-2} mmol), (*S*)-P-Phos (9.7 mg, 1.5×10^{-2} mmol) and 4 Å MS (100 mg) were weighted under air and placed in a 25 mL round-bottomed flask equipped with a magnetic stirring bar. Toluene (1.8 mL) was added and the mixture was stirred at 60 °C for 20 min until a pink-colored solution was observed. Then phenylsilane (112 μL , 0.90 mmol) and 4'-nitroacetophenone (**1a**, 124 mg, 0.75 mmol) were sequentially added under vigorous stirring, and the flask was stoppered. The reaction was monitored by TLC. Upon completion, the reaction mixture was treated with 10% HCl (0.5 mL) and organic product was extracted with ether (3 \times 10mL). The combined extract was washed with water, dried with anhydrous sodium sulfate, and concentrated in vacuo to give the crude product. The residue was purified by column chromatography on silica gel with ethyl acetate/petroleum ether (1 : 4) as eluent to give the desirable (*S*)-1-(4-nitrophenyl)ethanol [(*S*)-**2a**] (117 mg, 0.70 mmol) in 93% yield. The enantiomeric excess of the product (*S*)-**2a** was determined by chiral GC analysis to be 93% (Column, Chirasil-DEX CB 25 m \times 0.25 mm, Varian, carrier gas, N_2).

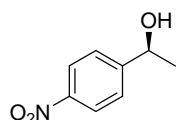
Table S1. Effects of cobalt salts and ligands on the Co(II)-catalyzed asymmetric hydrosilylation of 4'-nitroacetophenone **1a** in air.^a



| Entry | Ligand | [Co]/additive ^b | Yield (%) | ee (%) ^c |
|-------|------------------|---|-----------|---------------------|
| 1 | (S)-P-Phos | CoF ₂ /4 Å MS | 93 | 91 |
| 2 | (S)-P-Phos | Co(OAc) ₂ /4 Å MS | 99 | 93 |
| 3 | (S)-P-Phos | CoCl ₂ /4 Å MS | 1 | 86 |
| 4 | (S)-P-Phos | CoBr ₂ /4 Å MS | 1 | 69 |
| 5 | (S)-P-Phos | CoI ₂ /4 Å MS | 6 | 56 |
| 6 | (S)-P-Phos | Co(acac) ₂ /4 Å MS | 3 | 53 |
| 7 | (S)-P-Phos | Co(NO ₃) ₂ •6H ₂ O/4 Å MS | 4 | 70 |
| 8 | (S)-(R)-Josiphos | Co(OAc) ₂ •4H ₂ O/4 Å MS | 1 | 44 |
| 9 | (S,S)-Me-Duphos | Co(OAc) ₂ •4H ₂ O/4 Å MS | – | – |
| 10 | (S,S)-DIOP | Co(OAc) ₂ •4H ₂ O/4 Å MS | – | – |

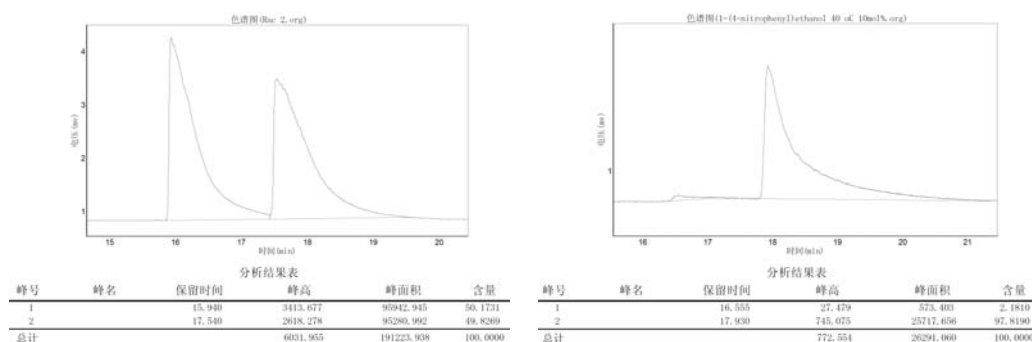
^a Reaction conditions: 24.8 mg substrate, substrate concentration = 0.15 M in toluene. ^b 30 mg of 4 Å MS was added. ^c The ee values were determined by chiral GC analysis. The absolute configuration was determined by comparing the retention times with the known data.

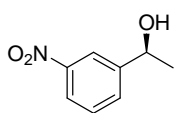
Analytical Data and GC or HPLC Spectra for 2a–2m



(S)-1-(4-Nitrophenyl)ethanol (2a): ¹H NMR (400 MHz, CDCl₃): δ 1.52 (d, *J* = 6.4 Hz, 3H), 2.06 (d, *J* = 2.8 Hz, 1H), 5.02 (q, *J* = 2.8 Hz, 1H), 7.54 (d, *J* = 8.4 Hz, 2H), 8.20 (d, *J* = 8.4 Hz, 2H). ¹³C NMR (100 MHz, CDCl₃): δ 25.5, 69.5, 123.8, 126.2, 147.2, 153.3. IR (thin film): ν_{max} (cm⁻¹) = 3362, 3080, 2970, 2931, 1720, 1685, 1604, 1510, 1346, 1089, 855. MS (EI) *m/z* (%): 167 (M⁺, 2.2), 152 (100).

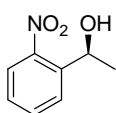
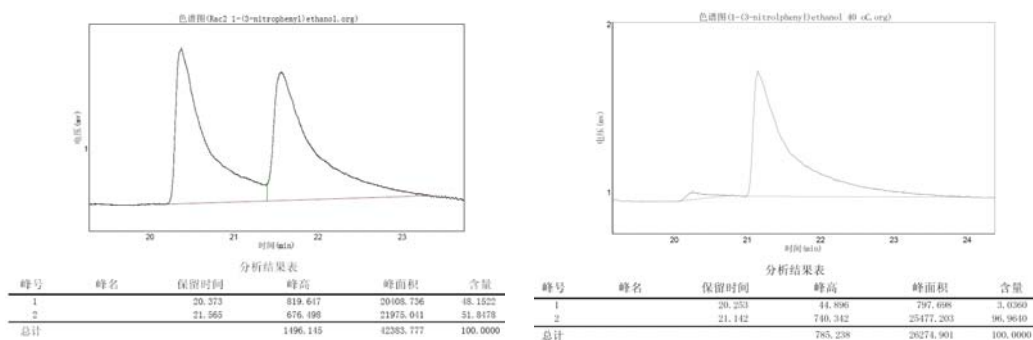
The conversion and ee value were determined by Capillary GC, Chirasil-DEX CB column; 170 °C; isothermal; *t_R* (**1a**) = 6.5 min; *t_R* (*R*) = 15.94 min; *t_R* (*S*) = 17.54 min.² Chromatograms are illustrated below for a 96% ee sample:





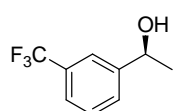
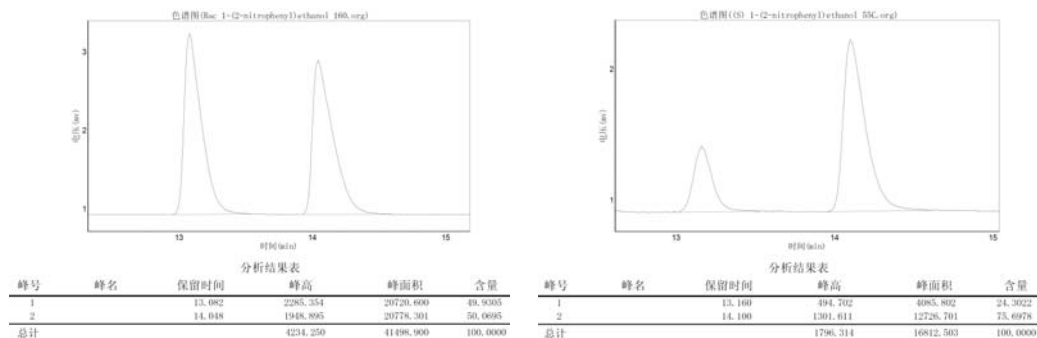
(S)-1-(3-Nitrophenyl)ethanol (2d): The general method was employed using the following quantities: $\text{Co}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (3.8 mg, 1.5×10^{-2} mmol), (*S*)-Xyl-P-Phos (11.4 mg, 1.5×10^{-2} mmol), 4 Å MS (50 mg), PhSiH_3 (22 μL , 0.18 mmol), Toluene (1.0 mL), 3'-nitroacetophenone (**1d**, 24.8 mg, 0.15 mmol). ^1H NMR (400 MHz, CDCl_3): δ 1.55 (d, $J = 5.2$ Hz, 3H), 1.95 (br, 1H), 5.03 (q, $J = 5.2$ Hz, 1H), 7.53 (t, $J = 6.4$ Hz, 1H), 7.72 (d, $J = 6.4$ Hz, 1H), 8.13 (d, $J = 6.4$ Hz, 1H), 8.26 (s, 1H). ^{13}C NMR (125 MHz, $(\text{CD}_3)_2\text{CO}$): δ 25.9, 68.8, 120.6, 122.2, 130.1, 132.5, 149.0, 150.4. IR (thin film): ν_{max} (cm^{-1}) = 3385, 3092, 2977, 2930, 1616, 1582, 1530, 1480, 1350, 1069, 809, 689. MS (EI) m/z (%): 167 (M^+ , 9.1), 152 (100), 121 (88.9).

The conversion and ee value were determined by Capillary GC, Chirasil-DEX CB column; 160 °C; isothermal; t_{R} (**1d**) = 7.56 min; t_{R} (*R*) = 20.37 min; t_{R} (*S*) = 21.57 min.³ Chromatograms are illustrated below for a 94% ee sample:



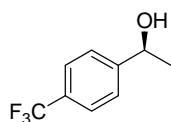
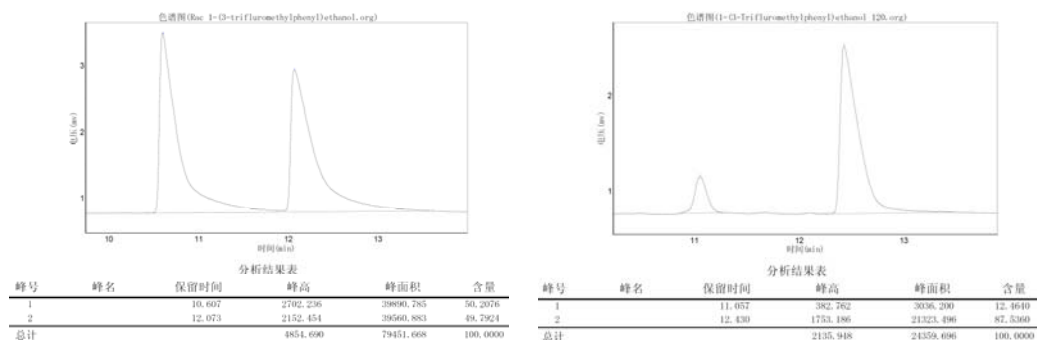
(S)-1-(2-Nitrophenyl)ethanol (2e): The general method was employed using the following quantities: $\text{Co}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (3.8 mg, 1.5×10^{-2} mmol), (*S*)-Xyl-P-Phos (11.4 mg, 1.5×10^{-2} mmol), 4 Å MS (50 mg), PhSiH_3 (22 μL , 0.18 mmol), Toluene (1.0 mL), 2'-nitroacetophenone (**1e**, 24.8 mg, 0.15 mmol). ^1H NMR (400 MHz, CDCl_3): δ 1.57 (d, $J = 6.8$ Hz, 3H), 2.40 (br, 1H), 5.41 (q, $J = 6.0$ Hz, 1H), 7.42 (t, $J = 7.2$ Hz, 1H), 7.65 (t, $J = 7.2$ Hz, 1H), 7.83–7.91 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3): δ 24.3, 65.6, 124.3, 127.6, 128.2, 133.7, 141.1, 147.8. IR (thin film): ν_{max} (cm^{-1}) = 3384, 2980, 2933, 1610, 1578, 1523, 1445, 1349, 1106, 747, 707. MS (EI) m/z (%): 167 (M^+ , 7.7), 152 (30.8), 104 (55.4).

The conversion and ee value were determined by Capillary GC, CP-Cyclodextrin-B-236-M-19 column; 160 °C; isothermal; t_{R} (**1e**) = 8.68 min; t_{R} (*R*) = 13.08 min; t_{R} (*S*) = 14.05 min.^{4,5} Chromatograms are illustrated below for a 51% ee sample:



(S)-1-(3-Trifluoromethylphenyl)ethanol (2f): The general method was employed using the following quantities: $\text{Co}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (3.8 mg, 1.5×10^{-2} mmol), (*S*)-Xyl-P-Phos (11.4 mg, 1.5×10^{-2} mmol), 4 Å MS (50 mg), PhSiH_3 (22 μL , 0.18 mmol), Toluene (1.2 mL), 3'-(trifluoromethyl)acetophenone (**1f**, 28.3 mg, 0.15 mmol). ^1H NMR (400 MHz, CDCl_3): δ 1.52 (d, $J = 5.2$ Hz, 3H), 1.87 (br, 1H), 4.97 (q, $J = 5.2$ Hz, 1H), 7.45–7.65 (m, 4H). ^{13}C NMR (125 MHz, CDCl_3): δ 25.5, 70.0, 122.3, 122.4, 124.3, 124.4, 128.9, 129.1, 146.8. IR (thin film): ν_{max} (cm^{-1}) = 3368, 2978, 2932, 1618, 1453, 1126, 1073, 828. MS (EI) m/z (%): 190 (M^+ , 14.2), 175 (75.3), 127 (79.2), 105 (58.3).

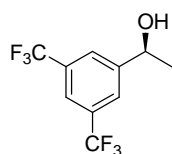
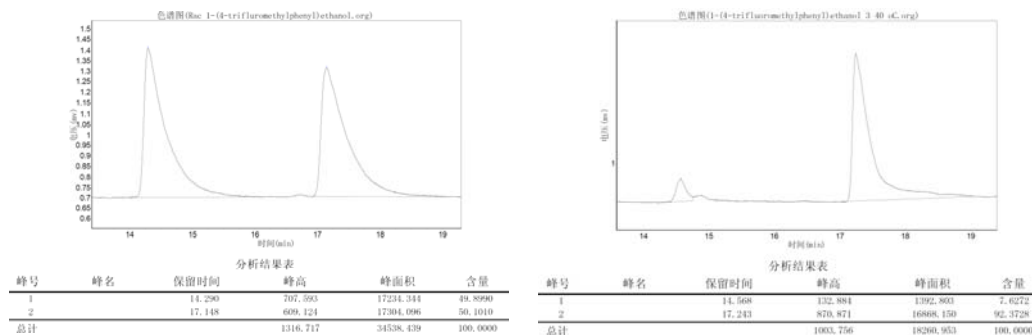
The conversion and ee value were determined by Capillary GC, Chirasil-DEX CB column; 120 °C; isothermal; t_{R} (**1f**) = 3.49 min; t_{R} (*R*) = 10.61 min; t_{R} (*S*) = 12.07 min.² Chromatograms are illustrated below for a 75% ee sample:



(S)-1-(4-Trifluoromethylphenyl)ethanol (2g): The general method was employed using the following quantities: $\text{Co}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (3.8 mg, 1.5×10^{-2} mmol), (*S*)-Xyl-P-Phos (11.4 mg, 1.5×10^{-2} mmol), 4 Å MS (50 mg), PhSiH_3 (22 μL , 0.18 mmol), Toluene (1.2 mL), 4'-(trifluoromethyl)acetophenone (**1g**, 28.3 mg, 0.15 mmol). ^1H NMR (400 MHz, CDCl_3): δ 1.51 (d, $J = 5.2$ Hz, 3H), 1.84 (br, 1H), 4.98 (q, $J = 5.2$ Hz, 1H), 7.50 (d, $J = 6.4$ Hz, 2H), 7.61 (d, $J = 6.8$ Hz, 2H). ^{13}C NMR (125 MHz,

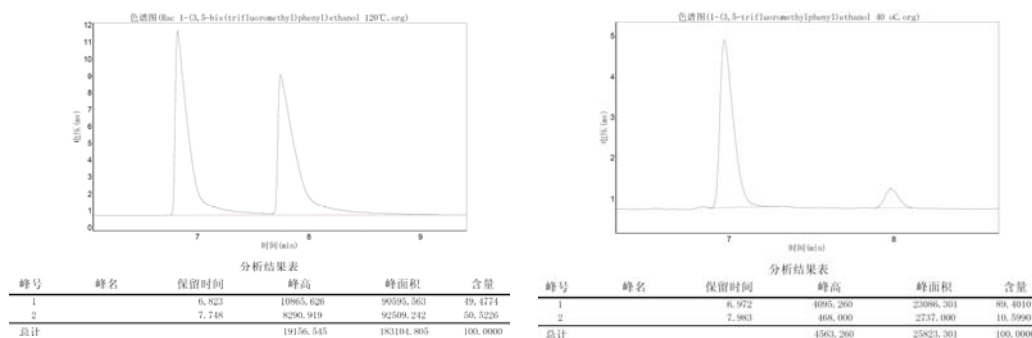
CDCl₃): δ 25.5, 70.0, 122.9, 125.6 ($J_{C,F} = 4.4$ Hz), 125.8, 129.8, 149.8. IR (thin film): ν_{\max} (cm⁻¹) = 3355, 2978, 1621, 1165, 1125, 1069, 1016, 738. MS (EI) m/z (%): 190 (M⁺, 13.7), 175 (100), 173 (80.4), 127 (84.3).

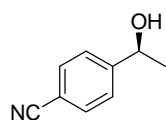
The conversion and ee value were determined by Capillary GC, Chirasil-DEX CB column; 120 °C; isothermal; t_R (**1g**) = 4.86 min; t_R (*R*) = 14.29 min; t_R (*S*) = 17.15 min.² Chromatograms are illustrated below for an 85% ee sample:



(S)-1-[3,5-Bis(trifluoromethyl)phenyl]ethanol (2h): The general method was employed using the following quantities: Co(OAc)₂•4H₂O (2.4 mg, 1.2×10^{-2} mmol), (*S*)-Xyl-P-Phos (3.0 mg, 4.0×10^{-3} mmol), 4 Å MS (50 mg), PhSiH₃ (22 μ L, 0.18 mmol), Toluene (1.2 mL), 3',5'-bis(trifluoromethyl)acetophenone (**1h**, 38.4 mg, 0.15 mmol). ¹H NMR (400 MHz, CDCl₃): δ 1.55 (d, $J = 5.6$ Hz, 3H), 1.98 (d, $J = 3.2$ Hz, 1H), 5.05 (q, $J = 3.2$ Hz, 1H), 7.79 (s, 1H), 7.84 (s, 2H). ¹³C NMR (125 MHz, CDCl₃): δ 25.7, 69.4, 121.5 (sep, $J_{C,F} = 4.4$ Hz), 123.5 (q, $J_{C,F} = 271.8$ Hz), 125.8, 131.9 (q, $J_{C,F} = 40.8$ Hz), 148.4. IR (thin film): ν_{\max} (cm⁻¹) = 3175, 2980, 2938, 1623, 1456, 1374, 1338, 1120, 842, 705. MS (EI) m/z (%): 243 (28.8), 240 (47.6), 132 (100).

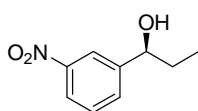
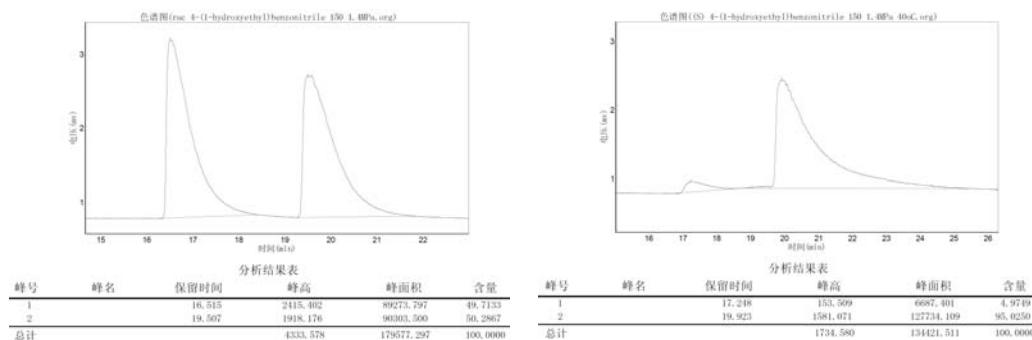
The conversion and ee value were determined by Capillary GC, Chirasil-DEX CB column; 120 °C; isothermal; t_R (**1h**) = 2.48 min; t_R (*S*) = 6.82 min; t_R (*R*) = 7.75 min.⁶ Chromatograms are illustrated below for a 79% ee sample:





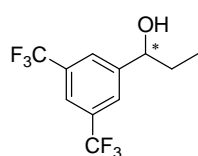
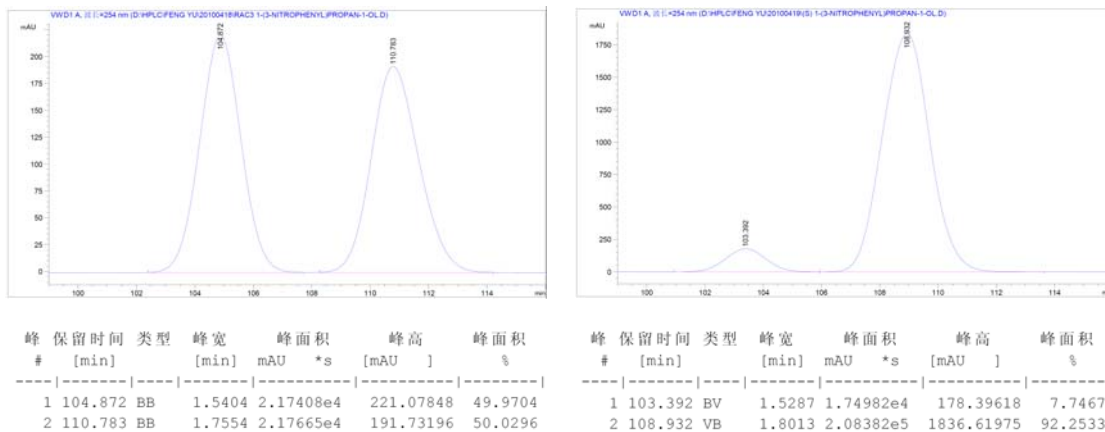
(S)-1-(4-Nitrophenyl)ethanol (2i): The general method was employed using the following quantities: $\text{Co}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (7.5 mg, 3.0×10^{-2} mmol), (S)-Xyl-P-Phos (22.7 mg, 3.0×10^{-2} mmol), 4 Å MS (75 mg), PhSiH_3 (45 μL , 0.36 mmol), Toluene (1.2 mL), 4'-nitroacetophenone (**1i**, 43.6 mg, 0.30 mmol). ^1H NMR (400 MHz, CDCl_3): δ 1.47 (d, $J = 6.8$ Hz, 3H), 2.37 (br, 1H), 4.94 (q, $J = 6.8$ Hz, 1H), 7.47 (d, $J = 8.0$ Hz, 2H), 7.61 (d, $J = 8.4$ Hz, 2H). ^{13}C NMR (100 MHz, CDCl_3): δ 25.4, 69.6, 110.9, 119.0, 126.2, 132.4, 151.3. IR (thin film): ν_{max} (cm^{-1}) = 3418, 2976, 2930, 2230, 1610, 1504, 1453, 1091, 839. MS (EI) m/z (%): 147 (M^+ , 16.7), 132 (100), 104 (88.9).

The conversion and ee value were determined by Capillary GC, Chirasil-DEX CB column; 150 °C; isothermal; t_{R} (**1i**) = 5.86 min; t_{R} (*R*) = 16.52 min; t_{R} (*S*) = 19.51 min.⁷ Chromatograms are illustrated below for a 90% ee sample:



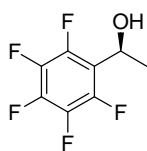
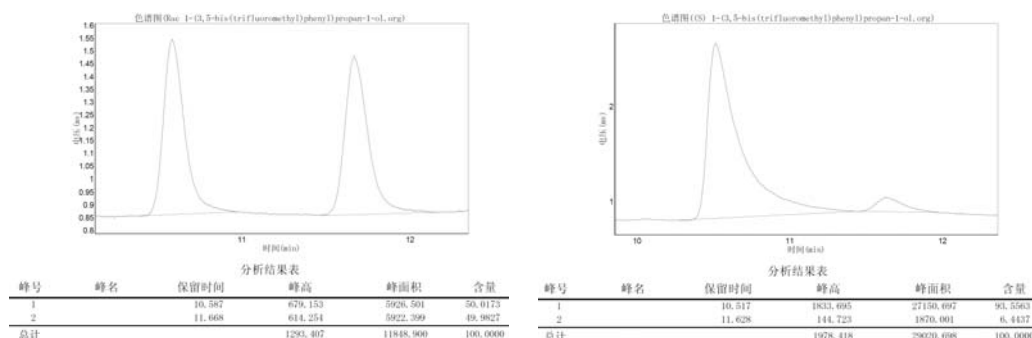
(S)-1-(3-Nitrophenyl)propan-1-ol (2j): The general method was employed using the following quantities: $\text{Co}(\text{OAc})_2 \cdot 4\text{H}_2\text{O}$ (3.8 mg, 1.5×10^{-2} mmol), (S)-Xyl-P-Phos (11.4 mg, 1.5×10^{-2} mmol), 4 Å MS (50 mg), PhSiH_3 (22 μL , 0.18 mmol), Toluene (1.0 mL), 1-(3-nitrophenyl)propan-1-one (**1j**, 26.9 mg, 0.15 mmol). ^1H NMR (400 MHz, CDCl_3): δ 0.94 (t, $J = 7.2$ Hz, 3H), 1.77–1.86 (m, 2H), 2.09 (br, 1H), 4.75 (t, $J = 6.4$ Hz, 1H), 7.52 (t, $J = 7.6$ Hz, 1H), 7.68 (d, $J = 7.6$ Hz, 1H), 8.11–8.23 (m, 2H). ^{13}C NMR (100 MHz, CDCl_3): δ 9.9, 32.2, 74.9, 121.0, 122.5, 129.4, 132.2, 146.8, 148.4. IR (thin film): ν_{max} (cm^{-1}) = 3363, 3074, 2968, 1697, 1593, 1538, 1462, 1352, 1152, 806, 698. MS (EI) m/z (%): 181 (M^+ , 7.7), 152 (100), 105 (38.4).

The ee value was determined by chiral HPLC analysis with a 25 cm \times 4.6 mm Daicel Chiralpak AD-H column (eluent, 2-propanol/hexane 1 : 99; flow rate: 0.6 mL/min; detection: 254 nm light); t_{R} (*R*) = 104.87 min; t_{R} (*S*) = 110.78 min.⁸ Chromatograms are illustrated below for an 85% ee sample:



(-)-1-[3,5-Bis(trifluoromethyl)phenyl]propan-1-ol (2k)⁹: The general method was employed using the following quantities: Co(OAc)₂•4H₂O (2.4 mg, 1.2 × 10⁻² mmol), (*S*)-Xyl-P-Phos (3.0 mg, 4.0 × 10⁻³ mmol), 4 Å MS (50 mg), PhSiH₃ (22 μL, 0.18 mmol), Toluene (1.2 mL), 1-[3,5-bis(trifluoromethyl)phenyl]propan-1-one (**1k**, 40.6 mg, 0.15 mmol). ¹H NMR (400 MHz, CDCl₃): δ 0.96 (t, *J* = 7.2 Hz, 3H), 1.77–1.84 (m, 2H), 2.07 (d, *J* = 2.0 Hz, 1H), 4.77 (t, *J* = 4.8 Hz, 1H), 7.79 (s, 2H), 7.81 (s, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 9.7, 32.2, 74.6, 121.5 (sep, *J*_{C,F} = 4.1 Hz), 123.5 (q, *J*_{C,F} = 270.7 Hz), 126.3, 131.8 (q, *J*_{C,F} = 33.4 Hz), 147.1. IR (thin film): ν_{max} (cm⁻¹) = 3200, 2981, 2920, 1619, 1463, 1289, 1164, 1118, 861, 705. MS (EI) *m/z* (%): 253 (M⁺, 13.6), 243 (45.5), 165 (72.7).

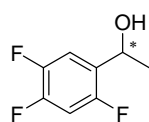
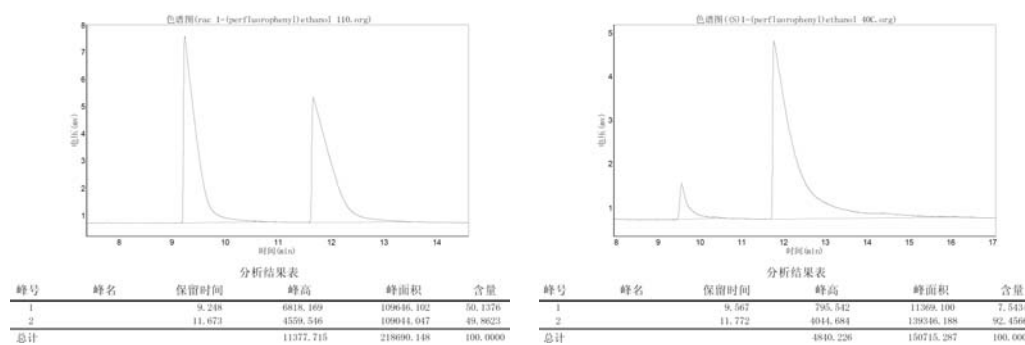
The conversion and ee value were determined by Capillary GC, Chirasil-DEX CB column; 120 °C; isothermal; *t*_R (**1k**) = 2.97 min; *t*_R (major) = 10.59 min; *t*_R (minor) = 11.67 min. [α]_D²⁰ = -22.6° (*c* = 1.0, CHCl₃). Chromatograms are illustrated below for an 87% ee sample:



(S)-1-(Pentafluorophenyl)ethanol (2l): The general method was employed using the following quantities: Co(OAc)₂•4H₂O (3.8 mg, 1.5 × 10⁻² mmol), (*S*)-Xyl-P-Phos (11.4 mg, 1.5 × 10⁻² mmol), PhSiH₃ (22 μL, 0.18 mmol),

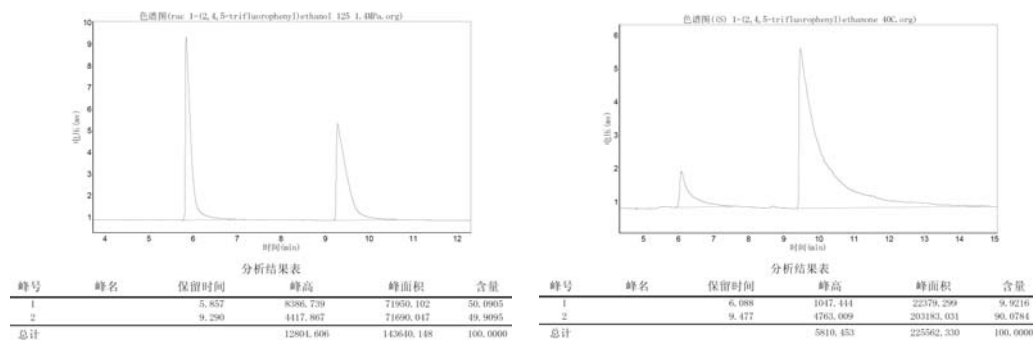
Toluene (1.0 mL), 2',3',4',5',6'-pentafluoroacetophenone (**11**, 31.6 mg, 0.15 mmol). ¹H NMR (400 MHz, CDCl₃): δ 1.45 (d, *J* = 6.8 Hz, 3H), 2.41 (br, 1H), 5.11 (q, *J* = 6.4 Hz, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 23.3, 62.4, 118.1, 137.7, 140.6, 144.8. IR (thin film): *v*_{max} (cm⁻¹) = 3332, 3074, 2983, 2937, 1652, 1586, 1505, 1352, 1267, 1168. MS (EI) *m/z* (%): 212 (M⁺, 10.2), 197 (100).

The conversion and ee value were determined by Capillary GC, Chirasil-DEX CB column; 110 °C; isothermal; *t*_R (**11**) = 2.82 min; *t*_R (*R*) = 9.25 min; *t*_R (*S*) = 11.67 min.¹⁰ Chromatograms are illustrated below for an 85% ee sample:



(-)-1-(2,4,5-Trifluorophenyl)ethanol (**2m**)¹¹: The general method was employed using the following quantities: Co(OAc)₂•4H₂O (3.8 mg, 1.5 × 10⁻² mmol), (*S*)-Xyl-P-Phos (11.4 mg, 1.5 × 10⁻² mmol), PhSiH₃ (22 µL, 0.18 mmol), Toluene (1.0 mL), 2',4',5'-trifluoroacetophenone (**1m**, 26.2 mg, 0.15 mmol). ¹H NMR (400 MHz, CDCl₃): δ 1.45 (d, *J* = 6.8 Hz, 3H), 2.41 (br, 1H), 5.11 (q, *J* = 6.4 Hz, 1H), 6.84–6.90 (m, 1H), 7.27–7.33 (m, 1H). ¹³C NMR (100 MHz, CDCl₃): δ 24.2, 63.7 (d, *J*_{C,F} = 2.0 Hz), 105.5 (dd, *J*_{C,F} = 28.7, 21.6 Hz), 114.7 (ddd, *J*_{C,F} = 19.6, 5.8, 1.4 Hz), 129.4 (td, *J*_{C,F} = 15.9, 4.7 Hz), 147.1 (ddd, *J*_{C,F} = 242.9, 13.5, 4.2 Hz), 149.1 (ddd, *J*_{C,F} = 248.7, 14.5, 12.3 Hz), 154.3 (ddd, *J*_{C,F} = 243.3, 9.8, 3.2 Hz). IR (thin film): *v*_{max} (cm⁻¹) = 3353, 2981, 1632, 1510, 1427, 1192, 1154, 1121, 835. MS (EI) *m/z* (%): 175 (M⁺, 71.4), 159 (92.9), 158 (88.9), 137 (14.3).

The conversion and ee value were determined by Capillary GC, Chirasil-DEX CB column; 125 °C; isothermal; *t*_R (**1m**) = 1.98 min; *t*_R (minor) = 5.86 min; *t*_R (major) = 9.29 min. [α]_D²⁰ = -29.9° (*c* = 0.5, CHCl₃). Chromatograms are illustrated below for an 80% ee sample:



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^1H NMR and ^{13}C NMR spectra of the alcohol products

