Chiral phosphoric acid catalyzed enantioselective sulfamination of amino–alkenes

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Supporting Information

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**General Methods.** All commercially available reagents were used without further purification. Toluene, tetrahydrofuran, and ethyl ether were distilled from sodium-benzophenone. CH$_3$CN and CH$_2$Cl$_2$ were distilled from CaH$_2$. CHCl$_3$ was distilled from P$_2$O$_5$. CH$_3$CCl$_3$ was dried by CaCl$_2$ and used directly. Column chromatography was performed on silica gel (200-300 mesh). $^1$H NMR spectra were recorded on a 400 MHz NMR spectrometer and $^{13}$C NMR spectra were recorded on a 100 MHz NMR spectrometer. IR spectra were recorded on a FT-IR spectrometer. Melting points were uncorrected.

Compounds 1a-c, 1j were prepared from commercially available alcohols by Mitsunobu reaction with 4-nitrobenzenesulfonylamide.$^1$ Compounds 1d, 1e, 1h, and 1i were prepared by Wittig reaction of the corresponding aldehydes and {4-[(tert-butyldiphenylsilyl)oxy]butyl}triphenylphosphonium iodide,$^2$ desilylation with TBAF, and Mitsunobu reaction with 4-nitrobenzenesulfonylamide.$^1$ Compounds 1f and 1g were prepared by Wittig reaction of 4-oxobutyl acetate and the above phosphonium salt,$^2$ followed by deacetylation with K$_2$CO$_3$ in MeOH, or desilylation with TBAF and subsequent Mitsunobu reaction with 4-nitrobenzenesulfonylamide.$^1$ Compounds 1k and 1l were prepared by Johnson-Claisen rearrangement,$^{3-5}$ reduction with LiAlH$_4$, and Mitsunobu reaction 4-nitrobenzenesulfonylamide.$^1$ Phosphoric acids 3a and 3b were prepared according to the reported procedure.$^6$ Phosphoric acid 3c was prepared according to the reported procedure and recrystallized from dichloromethane/hexane.$^7$

Representative procedure for asymmetric sulfamination (Table 2, entry 1).

To a stirred solution of alkene 1a (0.102 g, 0.30 mmol) and chiral phosphoric acid 3c (0.023 g, 0.030 mmol) in CHCl₃ (15.0 mL) was added PhSOMe (2) (0.051 g, 0.36 mmol) at 35 °C. Upon stirring at 35 °C for 72 h, the reaction mixture was quenched with Et₃N (0.6 mL), concentrated, and purified by column chromatography (silica gel, eluent: petroleum ether/ethyl acetate/dichloromethane = 50:1:0 to 20:1:1) to give pyrrolidine 4a as yellow solid (0.108 g, 80%).

(R)-1-(4-nitrophenylsulfonyl)-2-[(R)-1-(phenylthio)hexyl]pyrrolidine (Table 2, entry 1)

4a

Yellow solid; mp. 133-135 °C; [α]D²⁰ = +205.1 (c 1.00, CHCl₃) (86% ee); IR (film) 1522, 1159 cm⁻¹; ¹H NMR (400 MHz, CDCl₃) δ 8.18 (d, J = 8.8 Hz, 2H), 7.60-7.49 (m, 4H), 7.46-7.33 (m, 3H), 3.89 (dt, J = 11.6, 3.2 Hz, 1H) 3.58-3.44 (m, 2H), 3.25-3.16 (m, 1H), 2.05-1.92 (m, 1H), 1.90-1.65 (m, 4H), 1.58-1.45 (m, 1H), 1.45-1.20 (m, 6H), 0.93 (t, J = 6.8 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 150.2, 142.1, 135.2, 132.2, 129.4, 128.9, 127.3, 124.3, 62.5, 52.1, 51.4, 31.9, 27.7, 27.4, 26.8, 24.6, 22.8, 14.3; Anal. Calcd for C₂₂H₂₈N₂O₄S₂: C, 58.90; H, 6.29; N, 6.24; Found: C, 58.74; H, 6.35; N, 6.15.


(R)-1-(4-nitrophenylsulfonyl)-2-[(R)-1-(phenylthio)ethyl]pyrrolidine (Table 2, entry 2)

4b

White solid; mp. 139-141 °C; [α]D²⁰ = +208.5 (c 1.04, CHCl₃) (78% ee); IR (film) 1530,
$^{1351}$ cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.20 (d, $J = 8.8$ Hz, 2H), 7.62 (d, $J = 8.8$ Hz, 2H), 7.56-7.49 (m, 2H), 7.45-7.33 (m, 3H), 4.12-4.02 (m, 1H), 3.57-3.46 (m, 2H), 3.22-3.12 (m, 1H), 2.05-1.94 (m, 1H), 1.83-1.71 (m, 1H), 1.70-1.57 (m, 1H), 1.47-1.33 (m, 1H), 1.30 (d, $J = 7.2$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 150.2, 142.1, 134.6, 132.3, 129.4, 128.9, 127.5, 124.3, 62.1, 51.1, 45.8, 26.0, 24.7, 13.3; HRMS (ESI) Calcd for C$_{18}$H$_{21}$N$_2$O$_4$S$_2$ (M+H): 393.0937; Found: 393.0932.

(R)-1-(4-nitrophenylsulfonyl)-2-[(R)-1-(phenylthio)propyl]pyrrolidine (Table 2, entry 3)

![Image of (R)-1-(4-nitrophenylsulfonyl)-2-[(R)-1-(phenylthio)propyl]pyrrolidine](4c)

Pale yellow solid; mp. 148-149 °C; $[\alpha]_D^{20} = +211.1$ (c 0.95, CHCl$_3$) (84% ee); IR (film) 1534, 1350 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.18 (d, $J = 8.4$ Hz, 2H), 7.63-7.48 (m, 4H), 7.45-7.32 (m, 3H), 3.84-3.74 (m, 1H), 3.59-3.44 (m, 2H), 3.27-3.15 (m, 1H), 2.07-1.88 (m, 2H), 1.83-1.62 (m, 2H), 1.42-1.24 (m, 2H), 1.20 (t, $J = 7.2$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 150.2, 142.1, 134.6, 132.3, 129.4, 128.9, 127.2, 124.3, 62.6, 54.3, 51.3, 26.8, 24.5, 20.8, 12.9; HRMS (ESI) Calcd for C$_{19}$H$_{23}$N$_2$O$_4$S$_2$ (M+H): 407.1094; Found: 407.1094.

(R)-1-(4-nitrophenylsulfonyl)-2-[(R)-1-(phenylthio)decyl]pyrrolidine (Table 2, entry 4)

![Image of (R)-1-(4-nitrophenylsulfonyl)-2-[(R)-1-(phenylthio)decyl]pyrrolidine](4d)

White solid; mp. 92-94 °C; $[\alpha]_D^{20} = +177.8$ (c 1.02, CHCl$_3$) (85% ee); IR (film) 1519, 1350, 1161 cm$^{-1}$; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.17 (d, $J = 8.8$ Hz, 2H), 7.60-7.50 (m, 4H), 7.45-7.33 (m, 3H), 3.95-3.83 (m, 1H), 3.56-3.44 (m, 2H), 3.27-3.16 (m, 1H), 2.05-1.91 (m, 1H), 1.90-1.64 (m, 4H), 1.58-1.20 (m, 15H), 0.89 (t, $J = 6.8$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl$_3$) $\delta$ 150.2, 142.1, 134.6, 132.2, 129.4, 128.9, 127.3, 124.3, 62.5, 52.1, 51.4, 32.1, 29.7, 29.5, 28.0, 27.5, 26.7, 24.5, 22.9, 14.3; HRMS (ESI) Calcd for C$_{26}$H$_{37}$N$_2$O$_4$S$_2$ (M+H): 505.2189; Found: 505.2185.

(R)-1-(4-nitrophenylsulfonyl)-2-[(R)-3-phenyl-1-(phenylthio)propyl]pyrrolidine (Table 2, entry 3)
entry 5)

Yellow solid; mp. 140-142 °C; \([\alpha]_D^{20} = +179.0 \ (c \ 1.04, \ \text{CHCl}_3) \ (83\% \ ee); \ \text{IR (film)} 1530, 1351, 1164 \text{ cm}^{-1}; \ ^1\text{H NMR} (400 MHz, \text{CDCl}_3) \delta 8.17 \ (d, \ J = 8.8 \text{ Hz}, 2H), 7.58-7.49 \ (m, 4H), 7.45-7.37 \ (m, 3H), 7.33 \ (t, \ J = 7.2 \text{ Hz}, 2H), 7.29-7.20 \ (m, 3H), 3.92 \ (dt, \ J = 11.6, 3.2 \text{ Hz}, 1H), 3.58-3.43 \ (m, 2H), 3.25-3.08 \ (m, 2H), 2.87-2.75 \ (m, 1H), 2.27-2.13 \ (m, 1H), 2.07-1.91 \ (m, 1H), 1.80-1.67 \ (m, 2H), 1.66-1.52 \ (m, 1H), 1.40-1.24 \ (m, 1H); \ ^{13}\text{C NMR} (100 MHz, \text{CDCl}_3) \delta 150.2, 142.1, 141.7, 134.9, 132.2, 129.4, 128.9, 128.73, 128.67, 127.4, 126.3, 124.3, 62.5, 51.8, 51.3, 34.2, 29.6, 26.8, 24.5; \ \text{HRMS (ESI) Calcd for C}_{25}\text{H}_{27}\text{N}_2\text{O}_4\text{S}_2 (M+H): 483.1407; \ \text{Found: 483.1405.}

(R)-2-[(R)-4-(tert-butyldiphenylsilyloxy)-1-(phenylthio)butyl]-1-(4-nitrophenylsulfonyl)pyrrolidine (Table 2, entry 6)

White solid; mp. 102-104 °C; \([\alpha]_D^{20} = +133.2 \ (c \ 1.03, \ \text{CHCl}_3) \ (83\% \ ee); \ \text{IR (film) 1559, 1165 \text{ cm}^{-1}; \ ^1\text{H NMR} (400 MHz, \text{CDCl}_3) \delta 8.17 \ (d, \ J = 8.8 \text{ Hz}, 2H), 7.74-7.66 \ (m, 4H), 7.60-7.50 \ (m, 4H), 7.48-7.34 \ (m, 9H), 3.92-3.83 \ (m, 1H), 3.78 \ (t, \ J = 6.0 \text{ Hz}, 2H) 3.57-3.44 \ (m, 2H), 3.26-3.14 \ (m, 1H), 2.13-1.92 \ (m, 3H), 1.83-1.62 \ (m, 3H), 1.45-1.20 \ (m, 2H), 1.08 \ (s, 9H); \ ^{13}\text{C NMR} (100 MHz, \text{CDCl}_3) \delta 150.2, 142.2, 141.7, 135.8, 135.81, 135.1, 134.2, 132.3, 129.8, 129.4, 128.9, 127.9, 127.4, 124.3, 63.8, 62.5, 52.3, 51.3, 31.3, 27.1, 26.7, 24.5, 24.2, 19.5; \ \text{HRMS (ESI) Calcd for C}_{36}\text{H}_{43}\text{N}_2\text{O}_5\text{S}_2\text{Si(M+H): 675.2377; Found: 675.2360.}

(R)-4-[(R)-1-(4-nitrophenylsulfonyl)pyrrolidin-2-yl]-4-(phenylthio)butyl acetate (Table 2, entry 7)

Pale yellow solid; mp. 106-108 °C; \([\alpha]_D^{20} = +177.5 \ (c \ 1.05, \ \text{CHCl}_3) \ (80\% \ ee); \ \text{IR (film) }
1734, 1530, 1350 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.18 (d, \(J = 8.8\) Hz, 2H), 7.59-7.51 (m, 4H), 7.46-7.36 (m, 3H), 4.17 (t, \(J = 6.0\) Hz, 2H), 3.94-3.85 (m, 1H), 3.60-3.44 (m, 2H), 3.25-3.15 (m, 1H), 2.19-2.06 (m, 1H), 2.09 (s, 3H), 2.05-1.68 (m, 5H), 1.44-1.29 (m, 2H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 171.4, 150.2, 141.8, 134.7, 132.4, 129.4, 128.9, 127.5, 124.3, 64.2, 62.4, 51.8, 51.4, 27.1, 26.7, 24.4, 24.1, 21.2; HRMS (ESI) Calcd for C\(_{22}\)H\(_{27}\)N\(_2\)O\(_4\)S\(_2\) (M+H): 479.1305; Found: 479.1300.

(R)-2-[(R)-3-methyl-1-(phenylthio)butyl]-1-(4-nitrophenylsulfonyl)pyrrolidine (Table 2, entry 8)

Pale yellow solid; mp. 99-102 °C; \([\alpha]_D^{20} = +231.6\) (c 1.05, CHCl\(_3\)) (85% ee); IR (film) 1531, 1351, 1165 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.17 (d, \(J = 8.8\) Hz, 2H), 7.58-7.51 (m, 4H), 7.45-7.34 (m, 3H), 4.00 (dt, \(J = 11.6, 3.2\) Hz, 1H), 3.57-3.43 (m, 2H), 3.24-3.14 (m, 1H), 2.10-1.94 (m, 2H), 1.81-1.65 (m, 2H), 1.62-1.53 (m, 1H), 1.39-1.25 (m, 2H), 1.05 (d, \(J = 6.8\) Hz, 3H), 1.02 (d, \(J = 6.8\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 150.2, 142.1, 135.1, 132.3, 129.4, 128.9, 127.3, 124.3, 62.4, 51.8, 51.4, 27.1, 26.7, 24.4, 24.1, 21.2; HRMS (ESI) Calcd for C\(_{22}\)H\(_{27}\)N\(_2\)O\(_4\)S\(_2\) (M+H): 479.1305; Found: 479.1300.

(R)-2-[(R)-2,2-dimethyl-1-(phenylthio)propyl]-1-(4-nitrophenylsulfonyl)pyrrolidine (Table 2, entry 9)

Yellow solid; mp. 105-107 °C; \([\alpha]_D^{20} = +218.9\) (c 0.78, CHCl\(_3\)) (85% ee); IR (film) 1529, 1350, 1163 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.20 (d, \(J = 8.8\) Hz, 2H), 7.60 (d, \(J = 8.0\) Hz, 2H), 7.51 (d, \(J = 6.8\) Hz, 2H), 7.42-7.28 (m, 3H), 3.95-3.83 (m, 1H), 3.57-3.44 (m, 1H), 3.32-3.13 (m, 2H), 2.04-1.91 (m, 1H), 1.88-1.75 (m, 1H), 1.69-1.55 (m, 1H), 1.37-1.17 (m, 1H), 1.26 (s, 9H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 150.1, 143.0, 137.0, 132.6, 129.3, 128.9, 127.2, 124.2, 65.4, 63.4, 50.3, 35.9, 30.7, 29.7, 24.2; HRMS (ESI) Calcd for C\(_{21}\)H\(_{27}\)N\(_2\)O\(_4\)S\(_2\) (M+H): 435.1407; Found: 435.1405.
(R)-1-(4-nitrophenylsulfonyl)-2-[(R)-1-(phenylthio)propyl]piperidine (Table 2, entry 10)

Yellow oil; \([\alpha]_D^{20} = +72.9 \text{ (c 1.01, CHCl}_3\text{) (71% ee)}; \quad \text{IR (film) 1529, 1348, 1188 cm}^{-1};
\text{H NMR (400 MHz, CDCl}_3\text{) } \delta 8.29 (d, J = 8.8 \text{ Hz, 2H}), 8.02 (d, J = 8.8 \text{ Hz, 2H}), 7.38-7.32 (m, 2H), 7.31-7.20 (m, 3H), 4.13-4.03 (m, 1H), 3.74 (dd, J = 14.8, 4.4 Hz, 1H), 3.63-3.53 (m, 1H), 3.08-2.95 (m, 1H), 1.85-1.30 (m, 8H), 1.05 (t, J = 7.2 \text{ Hz, 3H}); \quad \text{13C NMR (100 MHz, CDCl}_3\text{) } \delta 149.9, 147.1, 135.5, 132.5, 129.2, 128.7, 127.3, 124.3, 55.9, 51.3, 42.0, 25.7, 24.3, 23.8, 18.8, 9.8; \quad \text{HRMS (ESI) Calcd for C}_{20}H_{25}N_2O_4S_2 (M+H): 421.1250; Found: 421.1249.}

(2R,3S)-1-(4-nitrophenylsulfonyl)-2-phenyl-3-(phenylthio)piperidine (Table 2, entry 11)

Yellow syrup; \([\alpha]_D^{20} = -45.3 \text{ (c 0.76, CHCl}_3\text{) (44% ee)}; \quad \text{IR (film) 1529, 1349, 1162 cm}^{-1};
\text{H NMR (400 MHz, CDCl}_3\text{) } \delta 8.28 (d, J = 8.8 \text{ Hz, 2H}), 8.04 (d, J = 8.8 \text{ Hz, 2H}), 7.47-7.16 (m, 10H), 5.39 (s, 1H), 3.99-3.81 (m, 2H), 3.38-3.22 (m, 1H), 1.95-1.76 (m, 3H), 1.54-1.42 (m, 1H); \quad \text{13C NMR (100 MHz, CDCl}_3\text{) } \delta 150.0, 146.6, 138.2, 134.8, 132.1, 129.6, 129.1, 128.9, 127.9, 127.8, 126.9, 124.1, 60.8, 49.4, 42.5, 24.1, 20.1; \quad \text{HRMS (ESI) Calcd for C}_{33}H_{28}O_6S_2 (M+H): 455.1094; Found: 455.1091.}

(2R,3S)-2-(naphthalen-1-yl)-1-(4-nitrophenylsulfonyl)-3-(phenylthio)piperidine (Table 2, entry 12)

Pale yellow solid; mp. 160-162 °C; \([\alpha]_D^{20} = +4.1 \text{ (c 0.93, CHCl}_3\text{) (55% ee)}; \quad \text{IR (film) 1528, 1348, 1164 cm}^{-1};
\text{H NMR (400 MHz, CDCl}_3\text{) } \delta 8.07 (d, J = 8.4 \text{ Hz, 2H}), 7.80-7.72 (m, 3H), 7.66 (d, J = 8.0 \text{ Hz, 1H}), 7.61-7.52 (m, 2H), 7.46-7.33 (m, 4H), 7.33-7.20 (m, 3H),...
7.14 (t, J = 7.6 Hz, 1H), 5.98 (s, 1H), 4.15-4.03 (m, 1H), 3.86-3.70 (m, 2H), 2.24-2.07 (m, 1H), 1.92-1.62 (m, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 149.7, 145.9, 134.9, 134.8, 134.1, 133.7, 130.5, 129.6, 129.3, 128.8, 128.7, 128.5, 127.0, 126.0, 125.0, 124.6, 123.8, 122.7, 58.4, 50.3, 44.5, 24.2, 20.4; HRMS (ESI) Calcd for C\(_{27}\)H\(_{24}\)N\(_2\)O\(_4\)S\(_2\)(M+Na): 527.1070; Found: 527.1060.

The determination of the absolute configuration of pyrrolidine 4c (Scheme 2)

![Scheme 2](image)

To a stirred mixture of pyrrolidine 4c (0.315 g, 0.77 mmol), K\(_2\)CO\(_3\) (0.428 g, 3.10 mmol), CH\(_3\)CN (14.7 mL), and DMSO (0.3 mL) was added PhSH (0.342 g, 3.10 mmol) at 50 °C. Upon stirring at 50 °C for 7 h, the reaction mixture was quenched with saturated aqueous NH\(_4\)Cl solution, concentrated to remove CH\(_3\)CN, extracted with CH\(_2\)Cl\(_2\) (3×50 mL), washed with brine, dried over MgSO\(_4\), filtered, concentrated, and purified by column chromatography (silica gel, eluent: petroleum ether/ethyl acetate = 10:1 to 5:1 to 2:1) to afford pyrrolidine 6c as yellow oil (0.134 g, 79%).

(R)-2-[(R)-1-(phenylthio)propyl]pyrrolidine (6c) (Scheme 2). \([\alpha]_D^{20} = +17.8\ (c\ 1.03,\ \text{CHCl}_3);\) IR (film) 1583, 1479, 1438 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 7.44 (d, \(J = 7.2\) Hz, 2H), 7.26 (t, \(J = 7.6\) Hz, 2H), 7.23-7.17 (m, 1H), 3.22-3.14 (m, 1H), 3.07-2.93 (m, 2H), 2.91-2.82 (m, 1H), 2.01 (br s, 1H), 1.92-1.67 (m, 4H), 1.62-1.48 (m, 2H), 1.07 (t, \(J = 7.6\) Hz, 3H); \(^{13}\)C NMR (100 MHz, CDCl\(_3\)) \(\delta\) 136.2, 132.1, 129.0, 126.8, 61.8, 57.7, 46.6, 29.7, 26.0, 25.7, 11.8; HRMS (ESI) Calcd for C\(_{13}\)H\(_{21}\)NS(M+H): 222.1311; Found: 222.1308.

A solution of pyrrolidine 6c (0.134 g, 0.61 mmol), DMAP (0.037 g, 0.31 mmol), and (Boc)_2O (0.266 g, 1.22 mmol) in THF (5 mL) was stirred at room temperature for 12 h, concentrated, and purified by column chromatography (silica gel, eluent: petroleum ether/ethyl acetate = 20:1) to afford pyrrolidine 8c as colorless oil (0.151 g, 77%). \[\alpha\]_D^{20} = -31.5 (c 1.10, CHCl_3).


A solution of pyrrolidine 8c (0.170 g, 0.53 mmol) in ethanol (7 mL) was added Raney Ni (1.1 g) at room temperature. Upon stirring at 80 °C for 2 h, the reaction mixture was filtered through a plug of silica gel with ethanol as eluent, concentrated, and purified by column chromatography (silica gel, eluent: petroleum ether/ethyl acetate = 30:1) to afford N-Boc-pyrrolidine 7c along with small amounts of 9c as colorless oil. The mixture was hydrogenated with Pd/C (0.018 g) in ethanol (10 mL) under hydrogen (1 atm) at rt for 24 h to give pyrrolidine 7c as colorless oil (0.050 g, 45% from 8c) after purification by column chromatography (silica gel, eluent: petroleum ether/ethyl acetate = 30:1). \[\alpha\]_D^{20} = +40.2 (c 0.99, CHCl_3) (84% ee) \{lit.\} for S-7c; \[\alpha\]_D^{21} = +45.6 (c 0.46, CHCl_3); IR (film) 1697, 1395, 1173 cm\(^{-1}\); \(^1\)H NMR (400 MHz, CDCl_3) \(\delta\) 3.85-3.64 (m, 1H), 3.50-3.22 (m, 2H), 2.01-1.71 (m, 4H), 1.70-1.57 (m, 1H), 1.45 (s, 9H), 1.39-1.17 (m, 3H), 0.91 (t, \(J = 7.2\) Hz, 3H); \(^1^3\)C NMR (100 MHz, CDCl_3) \(\delta\) 154.9, 79.0, 57.2, 46.6 and 46.2, 37.1 and 36.5, 30.8 and 30.1, 28.8, 23.9 and 23.3, 19.7, 14.3; HRMS (ESI) Calcd for C_{12}H_{23}NNaO_2(M+Na): 236.1621; Found: 236.1617.


The determination of the absolute configuration of piperidine 4j (Scheme 2)
Piperidine 4j was converted to 7j in a manner similar to transformation of pyrrolidine 4c to 7c.

(R)-2-[(R)-1-(phenylthio)propyl]piperidine (Scheme 2)

Pale yellow oil; $[\alpha]_D^{20} = +7.4$ (c 0.91, CHCl₃); IR (film) 3316, 1479, 1438 cm⁻¹; $^1$H NMR (400 MHz, CDCl₃) $\delta$ 7.42 (d, $J = 7.6$ Hz, 2H), 7.30-7.23 (m, 2H), 7.23-7.17 (m, 1H), 3.16-3.07 (m, 1H), 2.94-2.85 (m, 1H), 2.62 (td, $J = 12.0$, 2.8 Hz, 1H), 2.57-2.50 (m, 1H), 2.20 (br s, 1H), 1.88-1.69 (m, 3H), 1.63-1.19 (m, 5H), 1.06 (t, $J = 7.2$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl₃) $\delta$ 136.1, 132.1, 129.0, 126.8, 59.4, 58.3, 47.5, 30.3, 26.5, 25.1, 24.5, 11.6; HRMS (ESI) Calcd for C₁₄H₂₂NS(M+H): 236.1468; Found: 236.1468.

(R)-tert-butyl 2-[(R)-1-(phenylthio)propyl]piperidine-1-carboxylate

Pale yellow oil; $[\alpha]_D^{20} = +54.3$ (c 1.03, CHCl₃); IR (film) 1690, 1142 cm⁻¹; $^1$H NMR (400 MHz, CDCl₃) $\delta$ 7.43 (d, $J = 7.2$ Hz, 2H), 7.30-7.23 (m, 2H), 7.23-7.17 (m, 1H), 4.37-4.24 (m, 1H), 4.04-3.90 (m, 1H), 3.63-3.54 (m, 1H), 2.71-2.57 (m, 1H), 1.82-1.35 (m, 8H), 1.50 (s, 9H), 1.06 (t, $J = 7.6$ Hz, 3H); $^{13}$C NMR (100 MHz, CDCl₃) $\delta$ 155.3, 136.6, 132.7, 129.0, 126.9, 79.7, 52.4, 51.4, 39.4, 28.7, 26.3, 25.5, 24.3, 19.3, 9.4; HRMS (ESI) Calcd for C₁₉H₃₀NO₂S (M+H): 336.1992; Found: 336.1995.
Scheme 2

\[
\text{N} \quad \text{Boc}\]

Colorless oil; \([\alpha]_D^{20} = +22.0 (c\ 1.15, \text{CHCl}_3)\ (70\%\ \text{ee})\ \{\text{lit. for } R\text{-7j};\ [\alpha]_D = -39.8 (c\ 0.60, \text{CHCl}_3)\};\ \text{IR (film) 1692, 1416, 1148 cm}^{-1};\ 1^H\ \text{NMR (400 MHz, CDCl}_3)\ \delta 4.28-4.13\ (m, 1H), 4.02-3.87\ (m, 1H), 2.74\ (t, J = 13.2 Hz, 1H), 1.71-1.49\ (m, 6H), 1.44\ (s, 9H), 1.42-1.17\ (m, 4H), 0.91\ (t, J = 7.6 Hz, 3H);\ 13^C\ \text{NMR (100 MHz, CDCl}_3)\ \delta 155.3, 79.1, 50.3, 38.8, 32.1, 28.7, 25.9, 19.7, 19.2, 14.2;\ \text{HRMS (ESI) Calcd for } C_{13}H_{25}N\text{NaO}_2\ (M+Na): 250.1778;\ \text{Found: 250.1780.}


The determination of the absolute configuration of piperidine 5k (Scheme 2)

\[
\begin{align*}
\text{N} & \quad \text{SPh} \\
\text{Ph} & \quad \text{Ns} \\
\text{5k} & \\
\text{PhSH, K}_2\text{CO}_3 & \\
\text{CH}_3\text{CN/DMSO} & \\
50\ ^\circ\text{C}, 78\% & \\
\rightarrow & \\
\text{N} & \quad \text{SPh} \\
\text{Ph} & \quad \text{Boc} \\
\text{6k} & \\
\text{(Boc)}_2\text{O}, \text{DMAP} & \\
\text{THF, rt, 65\%} & \\
\rightarrow & \\
\text{N} & \quad \text{SPh} \\
\text{Ph} & \quad \text{Boc} \\
\text{7k} & \\
\text{Pd/C, H}_2 & \\
\text{EtOH, rt+} & \\
77\% & \\
\rightarrow & \\
\text{N} & \quad \text{SPh} \\
\text{Ph} & \quad \text{Boc} \\
\text{8k} & \\
\text{Raney Ni} & \\
\text{EtOH, 80\ ^\circ\text{C}} & \\
\rightarrow & \\
\text{N} & \quad \text{SPh} \\
\text{Ph} & \quad \text{Boc} \\
\text{7k} & \\
\text{EtOH, rt} & \\
77\% & \\
\rightarrow & \\
\text{N} & \quad \text{SPh} \\
\text{Ph} & \quad \text{Boc} \\
\text{9k} & \\
\text{Pd/C, H}_2 & \\
\text{EtOH, rt, 77\% from 8k} & \\
77\% & \\
\rightarrow & \\
\text{N} & \quad \text{SPh} \\
\text{Ph} & \quad \text{Boc} \\
\text{7k} & \\
\end{align*}
\]

Piperidine 5k was converted to 7k in a manner similar to transformation of pyrrolidine 4c to 7c.

\((2R,3S)-2\text{-phenyl-3-(phenylthio)piperidine (Scheme 2)}\)

\[
\begin{align*}
\text{N} & \quad \text{SPh} \\
\text{Ph} & \\
\text{6k} & \\
\text{EtOH, 80\ ^\circ\text{C}} & \\
\rightarrow & \\
\text{N} & \quad \text{SPh} \\
\text{Ph} & \\
\end{align*}
\]

Colorless oil; \([\alpha]_D^{20} = -28.0 (c\ 1.03, \text{CHCl}_3);\ \text{IR (film) 3325, 1474, 1438 cm}^{-1};\ 1^H\ \text{NMR (400 MHz, CDCl}_3)\ \delta 7.35\ (d, J = 7.2 Hz, 2H), 7.30-7.19\ (m, 3H), 7.17-7.09\ (m, 5H), 3.52\ (d,
J = 10.0 Hz, 1H), 3.20 (td, J = 10.4, 3.6 Hz, 1H), 3.13-3.05 (m, 1H), 2.74 (td, J = 11.6, 3.2 Hz, 1H), 2.28-2.18 (m, 1H), 1.82-1.48 (m, 4H); 13C NMR (100 MHz, CDCl3) δ 142.5, 134.6, 133.0, 128.7, 128.4, 128.2, 127.9, 126.9, 68.0, 52.0, 47.3, 33.9, 27.3; HRMS (ESI) Calcd for C17H20NS(M+H): 270.1311; Found: 270.1310.

(2R,3S)-tert-butyl 2-phenyl-3-(phenylthio)piperidine-1-carboxylate

8k

Colorless oil; [α]D20 = -23.7 (c 0.97, CHCl3); IR (film) 1692, 1415 cm⁻¹; 1H NMR (400 MHz, CDCl3) δ 7.50 (d, J = 7.6 Hz, 2H), 7.36-7.27 (m, 4H), 7.26-7.15 (m, 4H), 5.51 (s, 1H), 4.20 (dd, J = 13.6, 3.2 Hz, 1H), 4.10 (s, 1H), 2.81 (td, J = 13.2, 3 Hz, 1H), 2.11-1.96 (m, 1H), 1.95-1.80 (m, 2H), 1.55-1.30 (m, 1H), 1.42 (s, 9H); 13C NMR (100 MHz, CDCl3) δ 156.1, 139.5, 135.7, 132.1, 129.3, 128.9, 127.3, 127.0, 126.5, 80.0, 57.1, 47.9, 39.7, 28.5, 24.6, 20.5; HRMS (ESI) Calcd for C22H28NO2S (M+H): 370.1835; Found: 370.1845.

Scheme 2

7k

White solid; mp 71-73 °C; [α]D20 = -43.6 (c 1.06, CHCl3) (42% ee) {lit. for R-7k; [α]D22 = +76.2 (c 1.00, CHCl3)}; IR (film) 1691, 1157 cm⁻¹; 1H NMR (400 MHz, CDCl3) δ 7.34 (t, J = 7.6 Hz, 2H), 7.25-7.18 (m, 3H), 5.42 (s, 1H), 4.05 (d, J = 13.6 Hz, 1H), 2.83-2.70 (m, 1H), 2.36-2.24 (m, 1H), 1.94-1.82 (m, 1H), 1.64-1.35 (m, 4H), 1.46 (s, 9H); 13C NMR (100 MHz, CDCl3) δ 155.9, 140.7, 128.7, 126.7, 126.5, 79.7, 53.5, 40.3, 28.7, 28.3, 25.7, 19.6; HRMS (ESI) Calcd for C16H23NO2 (M+H): 262.1802; Found: 262.1803.

The determination of enantiomeric excess

Table 2, entry 1

![Chemical structure](image)

**HPLC Condition:** **Column:** Chiralpak OD-H, Daicel Chemical Industries, Ltd.; **Eluent:** Hexanes/IPA (95/5); **Flow rate:** 1.0 mL/min; **Detection:** UV256 nm.

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**Racemic**

**Chiral**

![Chromatograms](image)

Table 2, entry 2

![Chemical structure](image)

**HPLC Condition:** **Column:** Chiralpak AD-H, Daicel Chemical Industries, Ltd.; **Eluent:** Hexanes/IPA (90/10); **Flow rate:** 1.0 mL/min; **Detection:** UV256 nm.

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**Racemic**

**Chiral**

![Chromatograms](image)
Table 2, entry 3

4c

**HPLC Condition:**  
**Column:** Chiralpak OD-H, Daicel Chemical Industries, Ltd.;  
**Eluent:** Hexanes/IPA (90/10);  
**Flow rate:** 1.0 mL/min;  
**Detection:** UV252 nm.

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**Racemic**

**Chiral**

Table 2, entry 4

4d

**HPLC Condition:**  
**Column:** Chiralpak IC-H, Daicel Chemical Industries, Ltd.;  
**Eluent:** Hexanes/IPA (95/5);  
**Flow rate:** 1.0 mL/min;  
**Detection:** UV256 nm.

<table>
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**Racemic**

**Chiral**
Table 2, entry 5

HPLC Condition: Column: Chiralpak OD-H, Daicel Chemical Industries, Ltd.; Eluent: Hexanes/IPA (90/10); Flow rate: 1.0 mL/min; Detection: UV252 nm.

Racemic

Chiral

Table 2, entry 6

HPLC Condition: Column: Chiralpak AD-H, Daicel Chemical Industries, Ltd.; Eluent: Hexanes/IPA (95/5); Flow rate: 1.0 mL/min; Detection: UV258 nm.
Table 2, entry 7

**HPLC Condition:** Column: Chiralpak AD-H, Daicel Chemical Industries, Ltd.; Eluent: Hexanes/IPA (90/10); Flow rate: 1.0 mL/min; Detection: UV230 nm.

**Racemic**

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**Chiral**

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Table 2, entry 8

**HPLC Condition:** Column: Chiralpak AD-H, Daicel Chemical Industries, Ltd.; Eluent: Hexanes/IPA (95/5); Flow rate: 1.0 mL/min; Detection: UV258 nm.

**Racemic**

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**Chiral**

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Table 2, entry 9

\[
\text{HPLC Condition: Column: Chiralpak OD-H, Daicel Chemical Industries, Ltd.; Eluent: Hexanes/IPA (95/5); Flow rate: 1.0 mL/min; Detection: UV256 nm.}
\]

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Table 2, entry 10

\[
\text{HPLC Condition: Column: Chiralpak OD-H, Daicel Chemical Industries, Ltd.; Eluent: Hexanes/IPA (90/10); Flow rate: 1.0 mL/min; Detection: UV256 nm.}
\]

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Table 2, entry 11

\[
\begin{align*}
\text{HPLC Condition: Column:} & \quad \text{Chiralpak AD-H, Daicel Chemical Industries, Ltd.;} \\
\text{Eluent:} & \quad \text{Hexanes/IPA (90/10); Flow rate:} \quad 1.0 \text{ mL/min; Detection:} \quad \text{UV256 nm.}
\end{align*}
\]

\[
\begin{array}{|c|c|c|c|c|}
\hline
\text{Index} & \text{Time [Min]} & \text{Area %} & \text{Start [Min]} & \text{End [Min]} \\
\hline
1 & 33.20 & 50.11 & 32.24 & 34.63 \\
\hline
\text{Total} & & & & 100.00 \\
\hline
\end{array}
\]

Table 2, entry 12

\[
\begin{align*}
\text{HPLC Condition: Column:} & \quad \text{Chiralpak AD-H, Daicel Chemical Industries, Ltd.;} \\
\text{Eluent:} & \quad \text{Hexanes/IPA (90/10); Flow rate:} \quad 1.0 \text{ mL/min; Detection:} \quad \text{UV230 nm.}
\end{align*}
\]

\[
\begin{array}{|c|c|c|c|c|}
\hline
\text{Index} & \text{Time [Min]} & \text{Area %} & \text{Start [Min]} & \text{End [Min]} \\
\hline
1 & 33.03 & 27.89 & 32.41 & 35.83 \\
\hline
\text{Total} & & & & 100.00 \\
\hline
\end{array}
\]
**Scheme 2**

**7c**

**GC Condition:**  **Column:** Chiraldex CP-7495, Advanced Separation Technologies Inc.  
Oven: 120 °C;  Carrier: Helium, head pressure: 25 psi;  Detection: FID 250 °C.

![Chromatogram of 7c](image)

**Peak results:**

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<th>Peak</th>
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**Scheme 2**

**7j**

**GC Condition:**  **Column:** Chiraldex B-DM, Advanced Separation Technologies Inc.  
Oven: 120 °C;  Carrier: Helium, head pressure: 10 psi;  Detection: FID 250 °C.

![Chromatogram of 7j](image)

**Peak results:**

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</table>

Electronic Supplementary Material (ESI) for RSC Advances  
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Scheme 2

HPLC Condition: **Column:** Chiralpak AD-H, Daicel Chemical Industries, Ltd.;  
**Eluent:** Hexanes/IPA (99/1);  
**Flow rate:** 0.5 mL/min;  
**Detection:** UV206 nm.

![HPLC Chart]

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Table 2, Entry 1, 4a
Table 2, Entry 1, 4a

Electronic Supplementary Material (ESI) for RSC Advances
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Table 2, Entry 2, 4b
Table 2, Entry 3, 4c

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Table 2, Entry 3, 4c

- 150.15
- 142.09
- 135.16
- 132.19
- 129.36
- 128.84
- 127.25
- 124.27

- 77.95
- 77.23
- 76.91

- 62.59
- 54.26
- 51.33

- 26.76
- 24.50
- 20.77
- 12.86
Table 2, Entry 4, 4d
Table 2, Entry 4, 4d

Electronic Supplementary Material (ESI) for RSC Advances
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Table 2, Entry 5, 4e
Table 2, Entry 6, 4f

**Electronic Supplementary Material (ESI) for RSC Advances**

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Table 2, Entry 7, 4g

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Table 2, Entry 8, 4h
Table 2, Entry 9, 4i
Table 2, Entry 10, 4j

Electronic Supplementary Material (ESI) for RSC Advances
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Table 2, Entry 10, 4j

N
Ns
SPh
Table 2, Entry 11, 5K

NS
Ph

- 150.017
- 146.589
- 138.239
- 134.773
- 132.060
- 129.599
- 129.131
- 128.936
- 127.875
- 127.798
- 126.910
- 124.118

- 77.547
- 77.230
- 76.912

- 60.823

- 49.361
- 42.514

- 24.053
- 20.138
Table 2, Entry 12, 5l

[SPh]

Table 2. Entry 12. 5l

N

1-Naphth
Scheme 2, 6j