

Experimental

General procedures: All non-aqueous reactions were performed using oven dried glassware under an atmosphere of dry nitrogen. Diethyl ether (Et₂O) and tetrahydrofuran (THF) were distilled from sodium benzophenone ketyl prior to use. *N,N*-Diisopropylamine, dichloromethane, 1,2-dichloroethane, 2,6-lutidine, and triethylamine were distilled from calcium hydride prior to use. Toluene was distilled from sodium prior to use. Spectroscopy grade chloroform (with 0.75% ethanol) was used for all optical rotation data. Chromatographic purification of products was accomplished using forced flow chromatography on Baker 7024-R silica gel according to the method of Still¹. NMR spectra were recorded on a General Electric QE Plus operating at 300 and 75 MHz for ¹H and ¹³C, respectively, and are referenced to internal solvent signals. Data for ¹H are reported as follows: chemical shift (δ in ppm), integration, multiplicity (s singlet, d doublet, t triplet, q quartet, dd doublet of doublets, m multiplet) and coupling constant (*J* in Hz). IR spectra were recorded on a Perkin-Elmer 1600 Series spectrometer. Optical rotations were determined on a JASCO DIP-181 polarimeter operating at the sodium D line or the mercury 365 nm line and are reported as follows: $[\alpha]_D^{19}$, or $[\alpha]_{365}^{23}$, concentration (g/100 mL), and solvent. High-resolution mass spectrometry was performed by the Midwest Center for Mass Spectrometry at the University of Nebraska, with partial support by the National Science Foundation, Biology Division (Grant No. DIR9017262).

General Procedure for Enantioselective Mukaiyama Aldol Addition Reaction

To a 2.5 mM solution of **3** (0.11 equiv) in toluene was added Ti(*i*-PrO)₄ (0.05 equiv). The orange solution was stirred for 1 h at 23 °C before adding 3,5-di-*tert*-butylsalicylic acid (0.06 equiv) in toluene. Stirring was continued for an additional hour at 23 °C. The solvent was removed *in vacuo* and the solid orange residue was taken up in Et₂O. After cooling the solution to -78 °C, the aldehyde (1 equiv) and silyl ketene acetal² (1.2 equiv) were added sequentially. The flask was then kept at -10 °C for 4 h before it was quenched by pouring onto a 5% aqueous solution of NaHCO₃. The aqueous solution was extracted with Et₂O, and the combined organic extracts were washed with a saturated aqueous NaCl solution and dried over anhydrous Na₂SO₄. The organic extracts were concentrated *in vacuo*. The residue was taken up in THF and treated with excess Bu₄NF (2-3 equiv). The solution was partitioned between Et₂O and 1M aqueous HCl. The organic layer was washed with a 5% aqueous NaHCO₃ solution and then with a saturated

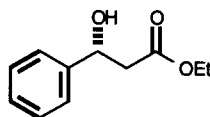
¹ Still, W. C.; Kahn, M.; Mitra, A. *J. Org. Chem.* **1978**, *43*, 2923.

² Prepared according to procedure of Kita, Y.; Segawa, J.; Haruta, J.; Yasuda, H.; Tamura, Y., *J. C. S. Perkin I*, **1982**, 1099.

aqueous NaCl solution. The reaction mixture was then dried over anhydrous Na₂SO₄ and concentrated *in vacuo*. Purification by chromatography on silica gel using 10:1 CH₂Cl₂/hexanes to elute the ligand followed by 10:1 CH₂Cl₂/Et₂O afforded the aldol adduct.

A portion of the aldol adduct was converted to the corresponding (*S*)-MTPA-ester.³ To a solution of the alcohol (0.01 mmol, 1 equiv) and 10 mg DMAP in 1 mL CH₂Cl₂ were added 10 μl triethylamine followed by (*R*)-MTPA-Cl (0.01 mmol, 1.1 equiv). The reaction mixture transferred to a column of silica gel and eluted with 6:1 hexanes/EtOAc. The enantiomeric excess of the product was determined by integration of the ¹H NMR (300 MHz, CDCl₃ or C₆D₆) resonances of the methoxy signals between δ 3.4-3.6 ppm.

Physical Data of β-Hydroxy Ester Products

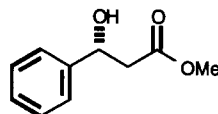


3-Hydroxy-3-phenylpropanoic acid ethyl ester.⁴ [α]_D²⁵ +63.1° (*c* = 2.7, CHCl₃); [α]_D¹⁹ +35.4° (*c* = 1.6, CHCl₃); IR (thin film) ν 3436, 3060, 3025, 2978, 2919, 1719, 1490, 1448, 1396, 1366, 1296, 1260, 1190, 1155, 1079, 1055, 1025, 949, 908, 885, 844, 756, 697 cm⁻¹; ¹H NMR (300 MHz, CDCl₃) δ 1.27 (3H, t, *J* = 7.1), 2.70 (1H, dd, *J* = 16.5, 2.3), 2.78 (1H, dd, *J* = 16.5, 6.1), 3.27 (1H, d, *J* = 3.4), 4.19 (2H, q, *J* = 7.1), 5.14 (1H, m), 7.28-7.40 (5H, m); ¹³C (75 MHz, CDCl₃) δ 14.1, 43.3, 60.9, 70.3, 125.6, 127.8, 128.5, 142.4, 172.4.

(*S*)-MTPA ester data: ¹H NMR (CDCl₃) methoxy resonances at δ 3.52 and 3.42 ppm in ratio of 29.5:1 (93% ee).

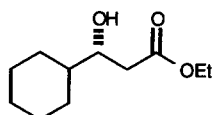
³ Dale, J. A.; Mosher, H. S. *J. Am. Chem. Soc.* **1973**, *95*, 512.

⁴ (a) Maruoka, K.; Hashimoto, S.; Kitagawa, Y.; Yamamoto, H.; Nozaki, H. *J. Am. Chem. Soc.* **1977**, *99*, 7705. (b) Cohen, S. G.; Weinstein, S. Y. *J. Am. Chem. Soc.* **1964**, *86*, 725.



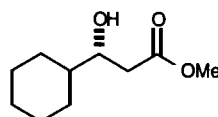
3-Hydroxy-3-phenylpropanoic acid methyl ester.⁵ IR (thin film) ν 3448, 3025, 2943, 1731, 1490, 1431, 1361, 1267, 1196, 1161, 1055, 1026, 985, 908, 879, 756, 697 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) 2.71 (1H, dd, $J = 16.4, 4.3$), 2.78 (1H, dd, $J = 16.4, 6.1$), 3.23 (1H, s), 3.73 (3H, s), 5.14 (1H, m), 7.27-7.40 (5H, m); ^{13}C (75 MHz, CDCl_3) δ 43.1, 51.9, 70.3, 125.6, 127.8, 128.5, 142.4, 172.8.

(*S*)-MTPA ester data: ^1H NMR (CDCl_3) methoxy resonances at δ 3.52 and 3.42 ppm in ratio of 48.0:1 (96% ee).



β -Hydroxycyclohexanepropionic acid, ethyl ester.⁶ $[\alpha]_{\text{D}}^{20} +20.8^\circ$ ($c = 1.7$, CHCl_3); $[\alpha]_{\text{D}}^{19} +27.8^\circ$ ($c = 0.66$, CHCl_3); IR (thin film) ν 3495, 2919, 2849, 1725, 1713, 1443, 1367, 1179, 1032, 891 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 1.29 (3H, t, $J = 7.1$), 1.0-1.5 (5H, m), 1.66-1.94 (6H, m), 2.45 (1H, dd, $J = 16.3, 9.3$), 2.53 (1H, dd, $J = 16.3, 3.0$), 2.84 (1H, s), 3.77-3.82 (1H, m), 4.16 (2H, q, $J = 7.1$); ^{13}C (75 MHz, CDCl_3) δ 14.1, 26.0, 26.1, 26.4, 28.2, 28.7, 38.5, 43.0, 60.6, 72.1, 173.5.

(*S*)-MTPA ester data: ^1H NMR (CDCl_3) methoxy resonances at δ 3.55 and 3.52 ppm in ratio of 31.2:1 (94% ee).



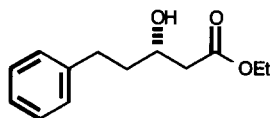
β -Hydroxycyclohexanepropionic acid, methyl ester.⁷ IR (thin film) ν 3460, 2919, 2837, 1725, 1437, 1308, 1279, 1167, 1102, 1038, 991, 885, 838 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 0.97-1.38 (6H, m), 1.63-1.87 (5H, m), 2.41 (1H, dd, $J = 16.3, 9.4$), 2.52 (1H, dd, $J = 16.3, 3.0$), 2.83 (1H, d, $J = 4.0$), 3.70 (3H, s), 3.77 (1H, m); ^{13}C (75 MHz, CDCl_3) δ 26.0, 26.1, 26.4, 28.2, 28.7, 38.3, 43.0, 51.7, 72.1, 173.9.

(*S*)-MTPA ester data: ^1H NMR (CDCl_3) methoxy resonances at δ 3.65 and 3.59 ppm in ratio of 39.1:1 (95% ee).

⁵ Devant, R.; Braun, M. *Chem. Ber.* **1986**, *119*, 2191.

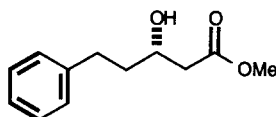
⁶ Bernardi, A.; Colombo, L.; Gennari, C.; Prati, L. *Tetrahedron* **1984**, *40*, 3769.

⁷ Otsubo, K.; Inanaga, J.; Yamaguchi, M. *Tetrahedron Lett.* **1987**, *28*, 4437.



3-Hydroxy-5-phenylpentanoic acid, ethyl ester.⁸ $[\alpha]_{365}^{19}$ -3.14° ($c = 0.88$, CHCl_3); $[\alpha]_{\text{D}}^{19} +1.88^\circ$ ($c = 1.40$, CHCl_3); IR (thin film) ν 3436, 3025, 2966, 2919, 2860, 1725, 1495, 1448, 1372, 1302, 1255, 1184, 1155, 1084, 1025, 931, 743, 696 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 1.27 (3H, t, $J = 7.1$), 1.68-1.79 (1H, m), 1.81-1.91 (1H, m), 2.44 (1H, dd, $J = 16.5, 8.4$), 2.52 (1H, dd, $J = 16.5, 3.7$), 2.65-2.78 (1H, m), 2.80-2.88 (1H, m), 3.10 (1H, s), 4.02 (1H, m), 4.17 (2H, q, $J = 7.1$), 7.16-7.36 (5H, m); ^{13}C (75 MHz, CDCl_3) δ 14.1, 31.7, 38.1, 41.2, 60.7, 67.2, 125.9, 128.4, 128.4, 141.7, 173.0.

(*S*)-MTPA ester data: ^1H NMR (CDCl_3) methoxy resonances at δ 3.57 and 3.54 ppm in ratio of 17.9:1 (89% ee).

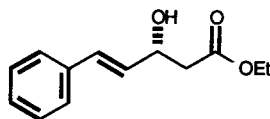


3-Hydroxy-5-phenylpentanoic acid, methyl ester.⁹ IR (thin film) ν 3436, 3013, 2933, 2849, 1731, 1596, 1490, 1455, 1431, 1361, 1302, 1255, 1196, 1173, 1155, 1085, 1055, 997, 932, 873, 750, 697 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 1.73-1.87 (2H, m), 2.45 (1H, dd, $J = 16.5, 8.7$), 2.53 (1H, dd, $J = 16.5, 4.2$), 2.68-2.83 (2H, m), 3.10 (1H, s), 3.71 (3H, s), 4.03 (1H, m), 7.17-7.31 (5H, m); ^{13}C (75 MHz, CDCl_3) δ 31.7, 38.1, 41.1, 51.7, 67.1, 125.8, 128.4, 128.4, 141.6, 173.3.

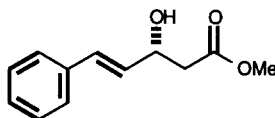
(*S*)-MTPA ester data: ^1H NMR (CDCl_3) methoxy resonances at δ 3.68 and 3.61 ppm in ratio of 34.0:1 (94% ee).

⁸ Kuwajima, I.; Minami, N.; Sato, T. *Tetrahedron Lett.* **1976**, 2253.

⁹ Fujisawa, T.; Fujimura, A.; Sato, T. *Bull. Chem. Soc. Jpn.* **1988**, 61, 1273.



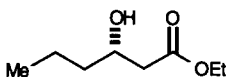
3-Hydroxy-5-phenyl-4-pentenoic acid, ethyl ester.¹⁰ $[\alpha]_{365}^{20} +27.6^\circ$ ($c = 0.94$, CHCl_3); $[\alpha]_{\text{D}}^{19} +13.6^\circ$ ($c = 1.2$, CHCl_3); IR (thin film) ν 3424, 2978, 1954, 1883, 1725, 1713, 1601, 1578, 1496, 1449, 1373, 1155, 1102, 1032, 967, 750, 691 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 1.30 (3H, t, $J = 7.1$), 2.61 (1H, dd, $J = 16.3, 5.9$), 2.69 (1H, dd, $J = 16.3, 4.1$), 3.10 (1H, d, $J = 4.2$), 4.21 (2H, q, $J = 7.1$), 4.75 (1H, m), 6.23 (1H, dd, $J = 16.0, 6.1$), 6.68 (1H, d, $J = 16.0$), 7.25-7.42 (5H, m); ^{13}C (75 MHz, CDCl_3) δ 14.3, 41.8, 60.9, 69.0, 126.6, 127.9, 128.7, 130.2, 130.7, 136.6, 172.2. (*S*)-MTPA ester data: ^1H NMR (CDCl_3) methoxy resonances at δ 3.58 and 3.52 ppm in ratio of 26.0:1 (93% ee).



3-Hydroxy-5-phenyl-4-pentenoic acid, methyl ester.¹¹ IR (thin film) ν 3448, 3025, 2943, 1725, 1490, 1431, 1402, 1355, 1284, 1202, 1161, 1102, 1032, 967, 750, 691 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) 2.61 (1H, dd, $J = 16.1, 3.4$), 2.69 (1H, dd, $J = 16.1, 4.6$), 3.13 (1H, s), 3.73 (3H, s), 4.74 (1H, m), 6.23 (1H, dd, $J = 15.9, 6.2$), 6.66 (1H, d, $J = 15.9$), 7.23-7.4 (5H, m); ^{13}C (75 MHz, CDCl_3) δ 41.3, 51.8, 68.8, 126.5, 127.8, 128.5, 129.9, 130.8, 136.3, 172.5. (*S*)-MTPA ester data: ^1H NMR (CDCl_3) methoxy resonances at δ 3.69 and 3.61 ppm in ratio of 80.0:1 (98% ee).

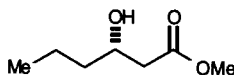
¹⁰ Araki, S.; Ito, H.; Butsugan, Y. *Syn. Commun.* **1988**, 453.

¹¹ Meyer, H. H. *Liebigs Ann. Chem.* **1979**, 484.



3-Hydroxyhexanoic acid, ethyl ester.¹² $[\alpha]_{365}^{25} +10.5^\circ$ ($c = 0.58$, CHCl_3); $[\alpha]_{\text{D}}^{19} +9.46^\circ$ ($c = 0.83$, CHCl_3); IR (thin film) ν 3483, 2955, 2919, 2861, 1725, 1708, 1449, 1367, 1302, 1290, 1179, 1138, 1079, 1014, 955, 850, 720, 685 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 0.93 (3H, t, $J = 7.1$), 1.27 (3H, t, $J = 7.1$), 1.34-1.55 (4H, m), 2.39 (1H, dd, $J = 16.5, 8.9$), 2.50 (1H, dd, $J = 16.5, 3.2$), 2.94 (1H, d, $J = 4.0$), 4.02 (1H, m), 4.17 (2H, q, $J = 7.1$); ^{13}C (75 MHz, CDCl_3) δ 13.9, 14.2, 18.7, 38.6, 41.3, 60.6, 67.7, 173.1.

(*S*)-MTPA ester data: ^1H NMR (C_6D_6) methoxy resonances at δ 3.50 and 3.43 ppm in ratio of 15.4:1 (88% ee).

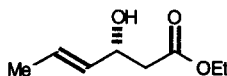


3-Hydroxyhexanoic acid, methyl ester.¹³ IR (thin film) ν 3448, 2943, 2919, 2861, 1731, 1431, 1361, 1302, 1261, 1167, 1119, 1073, 1020, 991, 844 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 0.92 (3H, t, $J = 7.0$), 1.31-1.54 (4H, m), 2.40 (1H, dd, $J = 16.4, 8.9$), 2.51 (1H, dd, $J = 16.4, 3.3$), 2.89 (1H, s), 3.70 (3H, s), 4.01 (1H, m); ^{13}C (75 MHz, CDCl_3) δ 13.9, 18.6, 38.6, 41.1, 51.7, 67.7, 173.5.

(*S*)-MTPA ester data: ^1H NMR (C_6D_6) methoxy resonances at δ 3.27 and 3.21 ppm in ratio of 40.5:1 (95% ee).

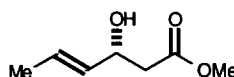
¹² Crump, D. R. *Aus. J. Chem.* **1982**, 1945.

¹³ Devant, R.; Braun, M. *Chem. Ber.* **1986**, 119, 2191.



3-Hydroxy-4-hexenoic acid, ethyl ester.¹⁴ $[\alpha]_{365}^{20} +26.5^\circ$ ($c = 1.00$, CHCl_3); $[\alpha]_{\text{D}}^{19} +11.3^\circ$ ($c = 1.00$, CHCl_3); IR (thin film) ν 3436, 2978, 2919, 1713, 1443, 1366, 1302, 1278, 1249, 1167, 1114, 1091, 1026, 961 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 1.27 (3H, t, $J = 7.1$), 1.69 (3H, dd, $J = 6.3, 0.8$), 2.48 (1H, dd, $J = 16.4, 2.4$), 2.55 (1H, d, $J = 16.4, 4.1$), 2.86 (1H, d, $J = 4.0$), 4.17 (2H, q, $J = 7.1$), 4.48 (1H, m), 5.51 (1H, m), 5.73 (1H, m); ^{13}C (75 MHz, CDCl_3) δ 14.2, 17.7, 41.5, 60.7, 68.9, 127.5, 131.7, 172.4.

(*S*)-MTPA ester data: ^1H NMR (C_6D_6) methoxy resonances at δ 3.50 and 3.44 ppm in ratio of 23.7:1 (92% ee).

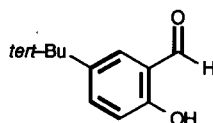


3-Hydroxy-4-hexenoic acid, methyl ester.¹⁵ IR (thin film) ν 3436, 2942, 2919, 1731, 1437, 1355, 1284, 1250, 1167, 1120, 1044, 1014, 967, 879 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) 1.69 (3H, d, $J = 6.3$), 2.53 (2H, m), 2.82 (1H, d, $J = 4.0$), 3.70 (3H, s), 4.48 (1H, m), 5.50 (1H, m), 5.73 (1H, m); ^{13}C (75 MHz, CDCl_3) δ 17.7, 41.3, 51.8, 68.9, 127.6, 131.7, 172.8.

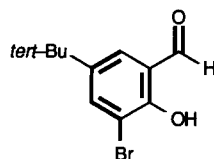
(*S*)-MTPA ester data: ^1H NMR (C_6D_6) methoxy resonances at δ 3.25 and 3.20 ppm in ratio of 80.0:1 (98% ee).

¹⁴ Zibuck, R.; Streiber, J. M. *J. Org. Chem.* **1989**, *54*, 4717.

¹⁵ Chamberlin, R. A.; Dezube, M.; Dussault, P. *Tetrahedron Lett.* **1981**, *22*, 4611.



5-tert-Butylsalicylaldehyde.¹⁶ Formylation of 4-tert-butylphenol was carried out according to the procedure of Jacobsen¹⁷: bp = 109.5 °C (4 mmHg); IR (thin film) ν 3178, 3072, 2955, 2861, 1696, 1655, 1619, 1584, 1478, 1390, 1373, 1361, 1314, 1284, 1261, 1226, 1179, 926, 826, 773, 732, 650, 603 cm^{-1} ; ^1H NMR (300 MHz, C_6D_6) δ 1.09 (9H, s), 6.84 (1H, d, $J = 8.8$), 6.97 (1H, d, $J = 2.5$), 7.10 (1H, dd, $J = 8.8, 2.5$), 9.22 (1H, s), 11.42 (1H, s); ^{13}C NMR (75 MHz, C_6D_6) δ 31.2, 33.9, 117.5, 120.4, 129.8, 134.5, 142.3, 160.1, 196.8.



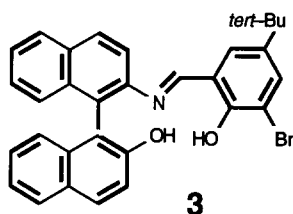
Bromination of 5-tert-Butylsalicylaldehyde.¹⁸ 5-tert-Butylsalicylaldehyde (1.00 g, 5.61 mmol) was dissolved in 15 mL glacial acetic acid. To the solution was added bromine (360 μL , 7.01 mmol) dropwise. The dark yellow solution was allowed to stir for 24 h at 23 °C. The solution was then diluted with 10 mL water to precipitate the product. The crystals were collected by suction filtration and recrystallized from 4:1 ethanol/water to give needles: mp = 81 °C; IR (thin film) ν 3072, 3037, 2955, 2861, 2731, 1661, 1614, 1455, 1414, 1378, 1325, 1261, 1214, 1155, 1114, 1014, 938, 885, 850, 814, 732, 691, 626 cm^{-1} ; ^1H NMR (300 MHz, C_6D_6): δ 0.98 (9H, s), 6.79 (1H, d, $J = 2.3$), 7.65 (1H, d, $J = 2.3$), 8.96 (1H, s), 11.77 (1H, s); ^{13}C NMR (75 MHz, C_6D_6): δ 30.9, 33.9, 111.3, 120.9, 129.3, 137.3, 143.6, 156.5, 196.1; HRMS (EI): calcd for $\text{C}_{11}\text{H}_{13}^{79}\text{BrO}_2$ (M)⁺ 256.0099, found 256.0092.

¹⁶ (a) Kerr, J. M.; Suckling, C. J.; Bamfield, P. *J. Chem. Soc. Perkin Trans. 1*, **1990**, 887. (b) Craig, J. A.; Harlan, E. W.; Snyder, B. S.; Whitener, M. A.; Holm, R. H. *Inorg. Chem.* **1989**, *28*, 2082.

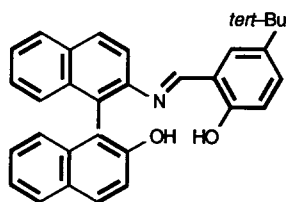
¹⁷ Deng, L.; Jacobsen, E. N. *J. Org. Chem.*, **1992**, *57*, 4320.

¹⁸ Werner, H., *Bull. Soc. Chim. Fr.*, **1886**, *46*, 277.

General Procedure for Schiff Base Formation. (*R*)-2-Amino-2'-hydroxy-1,1'-binaphthyl (0.100 g, 0.350 mmol) and 1.2 eq of the salicylaldehyde (0.420 mmol) were taken up in 5 mL absolute ethanol and heated to reflux for 24 h. The solvent was removed *in vacuo* and the product was isolated by chromatography on silica gel using 6:1 hexane/EtOAc. The orange product was dissolved in 10 mL CH₂Cl₂ and washed with a 5% solution of aqueous NaHCO₃. After drying the organic phase over anhydrous Na₂SO₄, the solvent was removed *in vacuo* and the resulting orange powder was dried under vacuum (2mm Hg) over 8 h.



3: mp = 164 °C; $[\alpha]_D^{19} +22.8^\circ$ ($c = 1.00$, CHCl₃); IR (thin film) ν 3389, 3049, 2955, 2908, 2861, 1608, 1502, 1461, 1425, 1343, 1261, 1208, 1161, 1138, 1067, 973, 950, 926, 873, 808, 744, 714 cm⁻¹; ¹H NMR (300 MHz, DMSO-d₆) δ 1.22 (9H, s), 6.79 (1H, d, $J = 7.7$), 7.10 (1H, d, $J = 8.4$), 7.13 (1H, dd, $J = 8.4, 7.7$), 7.22 (1H, dd, $J = 7.7, 7.0$), 7.31 (1H, dd, 7.7, 7.7), 7.37 (1H, d, 8.9), 7.48 (1H, dd, 7.7, 7.0), 7.54 (1H, s), 7.61 (1H, s), 7.87 (1H, d, $J = 7.7$), 7.90 (1H, d, $J = 9.2$), 7.93 (1H, d, $J = 9.2$), 8.04 (1H, d, $J = 7.7$), 8.16 (1H, d, $J = 8.9$), 9.07 (1H, s), 9.59 (1H, s), 13.21 (1H, s); ¹³C (75 MHz, DMSO-d₆) δ 31.0, 33.9, 109.5, 115.4, 117.8, 118.4, 119.5, 122.5, 123.8, 125.9, 126.1, 126.3, 126.8, 127.9, 128.1, 128.2, 129.0, 129.1, 129.3, 129.5, 132.4, 133.0, 133.1, 133.6, 142.3, 143.1, 152.8, 154.8, 162.8; HRMS (EI): calcd for C₃₁H₂₆⁷⁹BrNO₂ (M)⁺ 523.1147, found 523.1156.



mp = 148 °C; $[\alpha]_D^{19} +83.2^\circ$ ($c = 1.00$, CHCl_3); IR (thin film) ν 3366, 3049, 2943, 2861, 2355, 1619, 1572, 1484, 1461, 1425, 1373, 1355, 1337, 1284, 1261, 1202, 1173, 1138, 1067, 1020, 973, 920, 867, 814, 744 cm^{-1} ; ^1H NMR (300 MHz, CDCl_3) δ 1.22 (9H, s), 6.58 (1H, d, $J = 8.8$), 6.84 (1H, d, $J = 8.0$), 7.12 (1H, dd, $J = 8.0, 7.0$), 7.16 (1H, d, $J = 8.8$), 7.21 (1H, dd, $J = 8.0, 7.0$), 7.29 (1H, d, $J = 8.0$), 7.30 (1H, dd, $J = 8.0, 7.0$), 7.36 (1H, d, $J = 8.8$), 7.47 (1H, dd, $J = 8.0, 7.0$), 7.54 (1H, s), 7.86 (1H, d, $J = 8.0$), 7.91 (2H, d, $J = 8.8$), 8.03 (1H, d, $J = 8.0$), 8.15 (1H, d, $J = 8.8$), 9.07 (1H, s), 9.50 (1H, s), 12.21 (1H, s); ^{13}C (75 MHz, DMSO-d_6) δ 31.2, 33.7, 115.9, 116.0, 117.5, 118.3, 118.5, 122.4, 123.9, 125.6, 126.1, 126.2, 126.6, 127.9, 128.1, 128.1, 128.9, 128.9, 129.1, 129.3, 130.2, 132.3, 133.1, 133.6, 140.9, 143.6, 152.7, 157.9, 162.9; HRMS (EI): calcd for $\text{C}_{31}\text{H}_{27}\text{NO}_2$ (M) $^+$ 445.2042, found 445.2045.