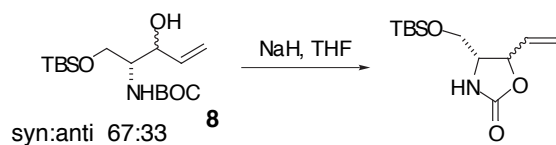
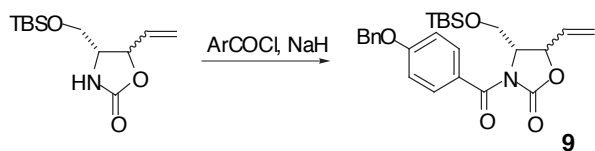


Experimental Section (Supporting Information)

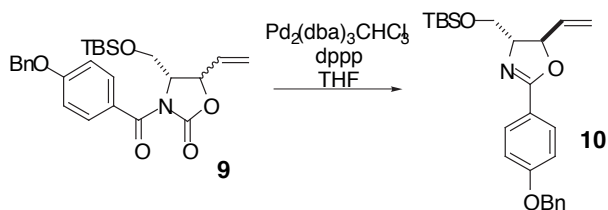


(4R)-4-[(*tert*-Butyldimethylsilyloxy)methyl]-5-vinyl-1,3-oxazolidine-2-one. To a suspension of sodium hydride (1.485 g, 60% in mineral oil, 37 mmol) in THF (20 mL), a solution of the alcohol **8** (6.14 g, 18.56 mmol) in THF (80 mL) was added dropwise at 0 °C and stirred at ambient temperature for 12h. The reaction mixture was cooled and carefully quenched with sat. aq. NH₄Cl solution, diluted with water, and extracted with ethyl acetate (3x100 mL). The organic layer was washed with water then brine, dried over MgSO₄, concentrated and purified by column chromatography over silica gel. Elution with (4:6) ethylacetate-hexane gave the oxazolidinone (4.328 g, 91%) as (2:1) mixture of *trans* and *cis* diastereomers. $[\alpha]_D^{25} = 41.28$ (*c* = 0.86, CHCl₃, 2.5:1 mixture of *trans* and *cis*); ¹H NMR (400 MHz, CDCl₃) (2:1 mixture of *trans* and *cis* isomers): δ 5.96-5.85 (m, 1H, *trans* and *cis*), 5.47 (d, *J* = 17.2 Hz, *cis*), 5.40 (d, *J* = 17.0 Hz, 1H, *trans*), 5.36 (d, *J* = 10.7 Hz, *cis*), 5.30 (d, *J* = 10.2 Hz, 1H, *trans*), 5.08 (t, *J* = 7.6 Hz, *cis*), 4.71 (t, *J* = 5.6 Hz, 1H, *trans*), 3.88 (dt, *J* = 7.8, 4.6 Hz, *cis*), 3.68-3.50 (m, 3H, *trans* and 2H *cis*), 0.88 (s, 9H, *trans*), 0.87 (s, *cis*), 0.06 (s, 6H, *trans*), 0.045 (s, 3H, *cis*), 0.04 (s, 3H, *cis*); ¹³C NMR (100.5 MHz, CDCl₃) (2:1 mixture of *trans* and *cis* isomers): δ 159.70 (*cis*), 159.57 (*trans*), 134.75 (*trans*), 130.82 (*cis*), 120.11 (*cis*), 118.14 (*trans*), 79.56 (*trans*), 79.40 (*cis*), 64.15 (*trans*) 62.15 (*cis*), 59.38 (*trans*), 57.08 (*cis*), 25.81 (*trans* and *cis*), 18.21 (*trans*), 18.13 (*cis*), -5.42 (*trans*), -5.50 (*cis*), -5.52 (*cis*); IR (neat film): 3308, 1757, 837 cm⁻¹. Anal. Calcd for C₁₂H₂₃NO₃Si: C, 55.99; H, 9.01; N, 5.44. Found: C, 56.07; H, 8.84; N, 5.46.



(4R)-3-[4-(Benzyloxy)benzoyl]-4-[(*tert*-Butyldimethylsilyloxy)methyl]-5-vinyl-1,3-oxazolidine-2-one (9**).** Into a suspension of sodium hydride (666 mg, 60% in mineral oil, 16.65 mmol) in THF (20 mL) at 0 °C, the oxazolidinone in THF (1.07 g, 4.16 mmol) was added and stirred at ambient temperature for 2h. In a separate flask 4-benzyloxy benzoic acid (2.09 g, 9.16 mmol) was dissolved in CH₂Cl₂. Into the benzoic acid solution, oxalyl chloride (0.95 mL, 10.82 mmol) and DMF (10 μL) were added and stirred at ambient temperature for 5h. The solution was concentrated and the solvent was evaporated completely under vacuum. The crude acid chloride was dissolved in THF (20 mL) and added to the oxazolidinone-sodium hydride solution at 0 °C. After stirring at

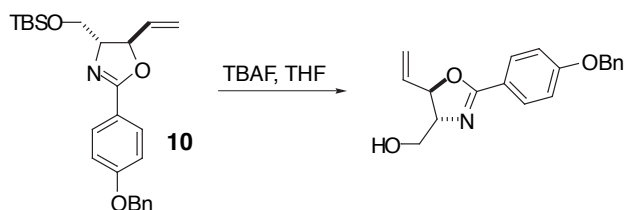
room temperature for 24h it was diluted with water and extracted with ether (3x75 mL). The organic layer was washed with water and brine, dried over anhydrous MgSO₄, and concentrated. Purification by column chromatography over silica gel (1:7 ethyl acetate-hexane) gave the acylated oxazolidinone **9** (1.747 g, 90%) as a 2:1 mixture of *trans* and *cis* diastereomers. $[\alpha]_D^{25} = -34.75$ (c = 1.18, CHCl₃, 1:1 mixture of *trans* and *cis*); ¹H NMR (400 MHz, CDCl₃) (2:1 mixture of *trans* and *cis*): δ 7.7-7.68 (m, 2H, *trans* and *cis*), 7.46-7.31 (m, 5H, *trans* and *cis*), 7.01-6.95 (m, 2H, *trans* and *cis*), 6.18 (ddd, J = 17.9, 10.5, 7.9 Hz, *cis*), 5.94 (ddd, J = 17.1, 10.5, 6.4 Hz, 1H, *trans*), 5.55 (d, J = 17.9 Hz, *cis*), 5.51 (d, J = 10.5 Hz, *cis*), 5.50 (d, J = 17.1 Hz, 1H, *trans*), 5.40 (d, J = 10.4 Hz, 1H, *trans*), 5.14-5.06 (m, 2 H, *trans* and *cis*), 5.08 (t, *cis*) 5.02 (t, J = 6.2 Hz, 1H, *trans*), 4.55 (ddd, J = 7.8, 3.8, 1.6 Hz, *cis*), 4.32 (ddd, J = 6.2, 4.2, 2.3 Hz, 1H, *trans*), 4.21 (dd, J = 11.0, 3.8, Hz, *cis*), 4.09 (dd, J = 11.0, 4.2 Hz, 1H, *trans*), 3.82 (dd, J = 11.0, 1.7 Hz, *cis*), 3.77 (dd, J = 11.0, 2.1 Hz, 1H, *trans*), 0.88 (s, 9H, *trans*), 0.86 (s, *cis*), 0.05 (s, 3H, *trans*), 0.04 (s, *cis*), 0.00 (s, 3H, *trans* and *cis*); ¹³C NMR (100.5 MHz, CDCl₃) (1:1 mixture of *trans* and *cis* isomers): δ 169.28, 169.08, 162.55, 162.21, 153.37, 153.31, 136.42, 136.35, 133.78, 131.94, 131.53, 131.02, 128.78, 128.33, 128.31, 127.71, 125.68, 125.46, 122.21, 119.68, 114.16, 114.06, 78.81, 76.81, 70.22, 70.19, 61.62, 59.98, 59.81, 59.31, 25.91, 25.84, 18.27, 18.10, -5.32, -5.44, -5.54; IR (neat film): 1786, 1676, 1604, 1510, 1253 cm⁻¹. Anal. Calcd for C₂₆H₃₃NO₅Si: C, 66.78; H, 7.11; N, 3.00. Found: C, 66.52; H, 6.83; N, 2.99.



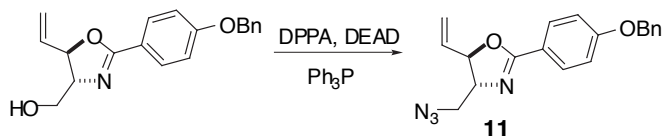
(4*R*,5*R*)-2-[4-(Benzyloxy)phenyl]-4-[(*tert*-Butyldimethylsilyloxy)methyl]-5-vinyl-1,3-

oxazoline (10). Oxazolidinone **9** (1.153 g, 2.47 mmol), Pd₂(dba)₃CHCl₃ (25.5 mg, 0.0247 mmol) and bis(diphenylphosphino)propane (41 mg, 0.099 mmol) were placed in a 50 mL flask and evacuated and flushed with dry N₂ three times. THF (25 mL) was added and the solution was stirred at room temperature for 10 min and at 35 °C for 12h. After cooling to ambient temperature, the solution was passed through a plug of fluorisil and the solvent was evaporated under reduced pressure to afford the *trans*-oxazoline **10** and the corresponding *cis* isomer in a 94:6 ratio. Purification by column chromatography over silica gel with 1:12 ethyl acetate-hexane as eluent afforded pure *trans*-**10** (0.824 g, 79%) as a colorless liquid and *cis*-**10** (0.033 g, 3%). *trans*-**10**: $[\alpha]_D^{25} = -6.1$ (c = 1.23, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 7.93-7.85 (m, 2H), 7.46-7.28 (m, 5H), 7.01-6.93 (m, 2H), 5.93 (ddd, J = 16.9, 10.3, 6.3 Hz, 1H), 5.35 (dt, J = 17.2, 1.4 Hz, 1H), 5.19 (dt, J

= 10.6, 1.2 Hz, 1H), 5.09 (s, 2H), 4.97 (t, J = 6.5 Hz, 1H), 4.01 (dt, J = 6.8, 3.9 Hz, 1H), 3.91 (dd, J = 10.2, 3.9 Hz, 1H), 3.64 (dd, J = 10.1, 6.9 Hz, 1H), 0.85 (s, 9H), 0.06 (s, 3H), 0.024 (s, 3H); ^{13}C NMR (100.5 MHz, CDCl_3): δ 163.71, 161.33, 136.96, 136.51, 130.14, 128.74, 128.22, 127.59, 120.58, 116.39, 114.60, 83.14, 74.11, 70.06, 64.93, 26.02, 18.43, -5.17, -5.20; IR (neat film): 2930, 2805, 1647, 1608, 1512, 1251 cm^{-1} . Anal. Calcd for $\text{C}_{25}\text{H}_{33}\text{NO}_3\text{Si}$: C, 70.88; H, 7.85; N, 3.31. Found: C, 70.86; H, 7.76; N, 3.30. *cis*-**10** Characteristic resonances: ^1H NMR (400 MHz, CDCl_3): δ 6.17 (ddd, J = 17.3, 10.5, 6.7 Hz, 1H), 5.40 (d, J = 17.3 Hz, 1H), 5.29 (d, J = 10.7 Hz, 1H), 5.15 (m, 1H), 5.09 (s, 2H), 4.32 (ddd, J = 10.2, 7.0, 3.5 Hz, 1H), 3.83 (dd, J = 10.5, 3.5 Hz, 1H), 3.69 (dd, J = 10.5, 7.0 Hz, 1H)

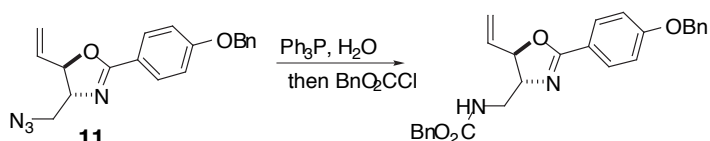


(4*R*,5*R*)-2-[4-(Benzyloxy)phenyl]-4-(hydroxymethyl)-5-vinyl-1,3-oxazoline. To a solution of oxazoline **10** (818 mg, 1.934 mmol) in THF (10 mL) was added TBAF (2.32 mL, 1M solution in THF) and the solution was stirred at room temperature for 2h. The reaction mixture was diluted with sat. aq. NaHCO_3 solution and extracted with ethyl acetate (2x30 mL). The organic layer was washed with brine, dried over MgSO_4 , and concentrated. The residue was dissolved in 1:9 ether-hexane, cooled to 0 °C and the precipitate was filtered to afford the alcohol (495 mg, 83%) as white solid. mp 120-122 °C; $[\alpha]_D^{25} = 19.53$ (c = 1.06, CHCl_3); ^1H NMR (270 MHz, CDCl_3): δ 7.84-7.75 (m, 2H), 7.46-7.28 (m, 5H), 6.95-6.86 (m, 2H), 5.95 (ddd, J = 17.2, 10.2, 7.2 Hz, 1H), 5.39 (dt, J 17, 1.1 Hz, 1H), 5.26 (dt, J = 10.2, 1.1 Hz, 1H), 5.08 (s, 2H), 4.95 (t, J = 7.4 Hz, 1H), 4.06-3.95 (m, 2H), 3.70-3.58 (m, 1H); ^{13}C NMR (100.5 MHz, CDCl_3): δ 164.58, 161.19, 136.56, 136.16, 130.08, 128.76, 128.24, 127.51, 119.69, 117.98, 114.41, 82.09, 73.98, 69.97, 62.51; IR (neat film): 3217, 1647, 1608, 1514, 1251 cm^{-1} . Anal. Calcd for $\text{C}_{19}\text{H}_{19}\text{NO}_3$: C, 73.77; H, 6.19; N, 4.53. Found: C, 73.57; H, 5.97; N, 4.60.



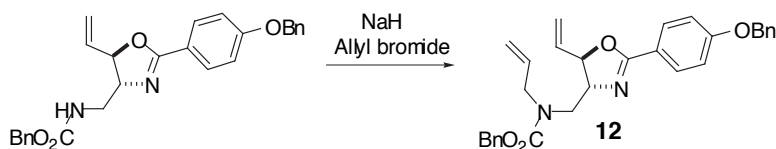
(4*R*,5*R*)-2-[4-(Benzyloxy)phenyl]-4-azidomethyl)-5-vinyl-1,3-oxazoline (11**).** To a solution of the alcohol (450 mg, 1.46 mmol) and triphenylphosphine (572 mg, 2.18 mmol) in dry THF (12 mL) at 0°C, diphenylphosphoryl azide (0.47 mL, 2.18 mmol) was added followed by diethyl

azodicarboxylate (0.344 mL, 2.18 mmol). The reaction mixture was stirred at room temperature for 5h, concentrated, and the residue was chromatographed over silica gel using (1:4) ethyl acetate-hexane as eluent to yield the azide **11** (426 mg, 88 %) as a yellow oil. $[\alpha]_D^{25} = 57.39$ ($c = 0.88$, CHCl_3); $^1\text{H NMR}$ (270 MHz, CDCl_3): δ 7.95-7.85 (m, 2H), 7.48-7.28 (m, 5H), 7.03-6.94 (m, 2H), 5.93 (ddd, $J = 17.2, 10.3, 7.1$ Hz, 1H), 5.39 (dt, $J = 17, 1.1$ Hz, 1H), 5.27 (dt, $J = 10.3, 1.1$ Hz, 1H), 5.10 (s, 2H), 4.84 (t, $J = 7$ Hz, 1H), 4.13 (dt, $J = 6.9, 5.0$ Hz, 1H), 3.57 (dd, $J = 12.5, 5.4$ Hz, 1H), 3.46 (dd, $J = 12.5, 4.5$ Hz, 1H); $^{13}\text{C NMR}$ (100.5 MHz, CDCl_3): δ 164.62, 161.63, 136.41, 135.73, 130.35, 128.75, 128.25, 127.58, 119.93, 117.93, 114.71, 83.4, 72.24, 70.11, 53.90; IR (neat film): 2102, 1643, 1608, 1510, 1251 cm^{-1} .



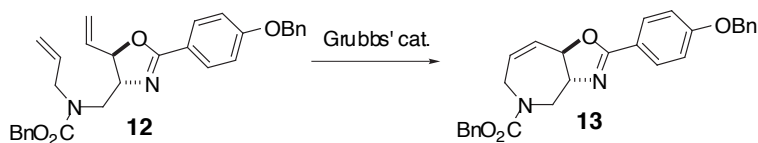
(4*R*,5*R*)-4-(Benzyloxycarbonylaminomethyl)-2-[4-(benzyloxy)phenyl]-5-vinyl-1,3-oxazoline.

Triphenylphosphine (235 mg, 0.898 mmol) was added to a solution of the azide **11** (250 mg, 0.749 mmol) in THF (4 mL) and water (1 mL) at 0 °C. The reaction was allowed to warm to ambient temperature and stirred for 12h. Triethylamine (0.5 mL) followed by benzylchloroformate (0.214 mL, 1.5 mmol) was added at 0 °C, and the solution was stirred for 3h. The reaction mixture was diluted with water and extracted with CH_2Cl_2 (2x 5 mL). The organic layer was washed with water and brine, dried over MgSO_4 , concentrated, and purified by column chromatography over silica gel (2:3 ethyl acetate-hexane) to afford the protected amine (232 mg, 70%) as a white solid. mp 75-77°C (hexane); $[\alpha]_D^{25} = 9.02$ ($c = 0.532$, CHCl_3); $^1\text{H NMR}$ (270 MHz, CDCl_3): δ 7.91-7.83 (m, 2H), 7.45-7.27 (m, 10H), 7.01-6.94 (m, 2H), 5.92 (ddd, $J = 17.2, 10.3, 7.1$ Hz, 1H), 5.38 (d, $J = 17.2$ Hz, 1H), 5.25 (d, $J = 10.4$ Hz, 1H), 5.18-5.00 (m, 5H), 4.75 (t, $J = 7.4$ Hz, 1H), 4.04 (m, 1H), 3.64-3.36 (m, 2H); $^{13}\text{C NMR}$ (100.5 MHz, CDCl_3): δ 164.28, 161.55, 156.82, 136.47, 136.41, 135.67, 135.67, 130.24, 128.75, 128.62, 128.26, 128.20, 127.57, 120.00, 118, 114.69, 83.2, 72.27, 70.11, 66.96, 43.80; IR (neat film): 3327, 3238, 3034, 1712, 1643, 1608, 1512 cm^{-1} . Anal. Calcd for $\text{C}_{27}\text{H}_{26}\text{N}_2\text{O}_4$: C, 73.29; H, 5.92; N, 6.33. Found: C, 73.12; H, 5.84; N, 6.60.



(4*R*,5*R*)-4-(Allylbenzyloxycarbonylaminomethyl)-2-[4-(benzyloxy)phenyl]-5-vinyl-1,3-oxazoline (12**).** Into a suspension of sodium hydride (100 mg, 60% in mineral oil, 2.5 mmol) in

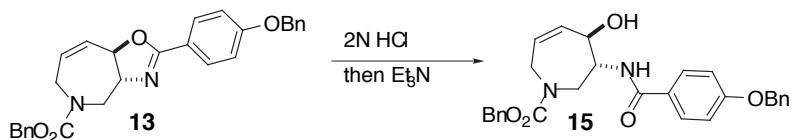
THF (2 mL), the carbamate (550 mg, 1.244 mmol) was added and stirred at room temperature for 0.5 h. Freshly distilled allyl bromide (0.323 mL, 3.73 mmol) was added and the mixture was stirred for 24h. Then it was heated at 50 °C for 5h, cooled to 0 °C, carefully quenched with water and extracted with CH₂Cl₂ (2x25 mL). The combined organic extracts were washed with brine, dried over MgSO₄, concentrated, and purified by column chromatography over silica gel. Elution with (1:3) ethyl acetate-hexane provided the allylated product **12** (522 mg, 87%) as colorless oil and further elution with (2:3) ethyl acetate-hexane provided the unreacted starting material (60 mg, 11%). $[\alpha]_D^{25} = -7.62$ (c = 0.42, CHCl₃); ¹H NMR (270 MHz, CDCl₃): δ 7.92-7.82 (m, 2H), 7.45-7.26 (m, 10 H), 7.01-6.94 (m, 2H), 5.98-5.66 (m, 2H), 5.38-5.00 (m, 8H), 4.92 (t, J = 7 Hz, 0.5 H), 4.73 (t, J = 6.8 Hz, 0.5 H), 4.20-3.86 (m, 3H), 3.76-3.50 (m, 1H), 3.43 (dd, J = 14.3, 5.4 Hz, 1H); ¹³C NMR (100.5 MHz, CDCl₃): δ 163.71, 163.5, 161.45, 156.92, 156.1, 136.77, 136.47, 136.05, 135.89, 133.57, 133.37, 130.15, 128.75, 128.65, 128.56, 128.30, 128.25, 128.05, 127.87, 127.57, 120.41, 120.29, 117.48, 117.37, 116.78, 114.70, 83.66, 83.46, 72.16, 72.67, 72.16, 72.07, 70.11, 67.54, 67.37, 50.92, 50.68, 50.19, 49.73; IR (neat film): 1699, 1645, 1608, 1246 cm⁻¹. Further elution with (2:3) ethyl acetate-hexane provided the unreacted starting material (60 mg, 11%).



Tetrahydroazepine (13). Into a solution of the diene **12** (522 mg, 1.083 mmol) in CH₂Cl₂ (48 mL) at room temperature, Cl₂(PCy₃)₂Ru=CHPh (45 mg, 0.054 mmol) in CH₂Cl₂ (2 mL) was added and the mixture was heated at 45 °C. After 4h, an additional 5 mol% of catalyst (45 mg, 0.054 mmol) was added and heating was continued at 45 °C for 5h. The mixture was concentrated and purified by column chromatography over silica gel (2:3 ethyl acetate-hexane) to afford **14** (378 mg, 77%) as a brownish wax which was recrystallized from diethyl ether. mp 104 °C; $[\alpha]_D^{25} = -46.79$ (c = 0.84, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 7.88 (d, J = 8.9 Hz, 2H), 7.52-7.26 (m, 10H), 6.98 (d, J = 8.8 Hz, 2H), 6.27 (d, J = 10.5 Hz, 1H), 5.98-5.80 (m, 1H), 5.22-5.05 (m, 2H), 4.94 (m, 1H), 4.40-3.72 (m, 5H); ¹³C NMR (100.5 MHz, CDCl₃): δ 165.75, 165.55, 161.68, 161.63, 156.13, 156.06, 136.71, 136.41, 130.1, 129.83, 129.55, 129.46, 129.33, 128.75, 128.62, 128.26, 128.22, 128.14, 128.08, 127.78, 127.6, 120.33, 120.21, 114.77, 84.56, 84.08, 70.68, 70.48, 70.11, 67.75, 67.54, 49.55, 49.33, 45.02, 44.88; IR (neat film): 1701, 1631, 1606, 1510, 1419, 1248 cm⁻¹. Anal. Calcd for C₂₈H₂₆N₂O₄: C, 73.99; H, 5.77; N, 6.16. Found: C, 73.74; H, 5.70; N, 6.43.

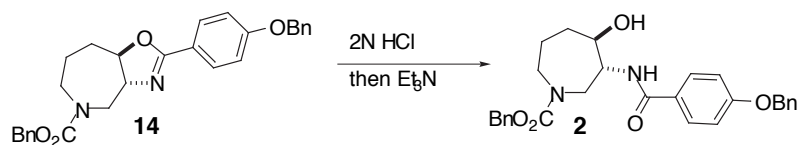


Hexahydroazepine (14). To a solution of olefin **13** (40 mg, 0.088 mmol) and dipotassium azodicarboxylate (341 mg, 1.76 mmol) in methanol (1 mL) at 0 °C, a solution of the acetic acid (0.252 mL, 4.4 mmol) in methanol (1 mL) was added dropwise and stirred for 24h. The mixture was diluted with water and extracted with CHCl₃. The organic layer was washed with brine, dried over MgSO₄, and concentrated. Purification by column chromatography over silica gel (2:3 ethyl acetate-hexane) gave the reduced product **14** (20 mg, 50%) as a colorless oil. ¹H NMR (400 MHz, CDCl₃): δ 7.85 (d, J = 8.6 Hz, 2H), 7.49-7.24 (m, 10H), 6.97 (d, J = 8.9 Hz, 2H), 5.20-5.07 (m, 4H), 4.30-3.71 (m, 5H), 2.88 (dd, J = 14.8, 11.3 Hz, 1H), 2.54-2.40 (m, 1H), 2.00-1.62 (m, 3H); ¹³C NMR (100.5 MHz, CDCl₃): δ 165.15, 164.85, 161.56, 161.49, 156.28, 156.23, 136.81, 136.56, 136.45, 136.40, 130.06, 128.73, 128.61, 128.56, 128.24, 128.09, 127.98, 127.92, 127.58, 120.40, 120.20, 114.71, 114.68, 85.90, 85.36, 71.27, 70.95, 70.10, 67.43, 67.34, 48.06, 47.97, 44.04, 43.93, 30.97, 30.93, 25.65, 25.35.



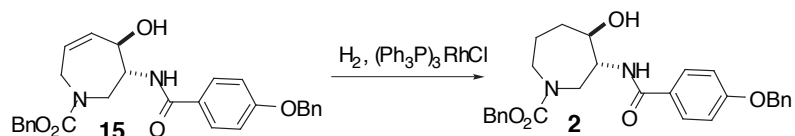
4-Hydroxy-3-[4-(phenylmethoxy)benzoylamino]-1-(phenylmethoxycarbonyl)-2,3,4,7-tetrahydroazepine (15). Oxazoline **13** (180 mg, 0.397 mmol) was dissolved in a mixture of THF (4 mL) and 2N aq. HCl (4 mL), and stirred at ambient temperature for 24h. The reaction mixture was made basic by the addition of sat. aq. NaHCO₃ and extracted with CHCl₃ (3x15 mL). The organic layer was washed with brine, dried over MgSO₄, concentrated, and the residue was dissolved in methanol (4 mL). Triethylamine (0.2 mL) was added to this solution and stirred at room temperature for 36h. Concentration and purification by column chromatography over silica gel (3:1 ethyl acetate-hexane) yielded the hydrolyzed product **15** (135 mg, 72%) which was recrystallized from ether-hexane to give a white crystalline solid. Mp 84 °C; [α]_D²⁵ = -51.74 (c = 0.69, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 8.68 (d, J = 5.9 Hz, 1H), 7.84 (d, J = 8.9 Hz, 2H), 7.50-7.26 (m, 10H), 7.03 (d, J = 8.9 Hz, 2H), 5.74 (m, 1H), 5.42 (m, 1H), 5.39 (s, 1H), 5.20 (ABq, J = 12.4 Hz, 2H), 5.12 (s, 2H), 4.74-4.57 (m, 2H), 4.30 (dd, J = 12.8, 6.1 Hz, 1H), 3.98 (d, J = 15 Hz, 1H), 3.71 (m, 1H), 3.61 (dd, J = 15.0, 5.6 Hz, 1H); ¹³C NMR (100.5 MHz, CDCl₃): δ 168.91, 161.75, 157.84, 136.42, 136.02, 134.66, 129.32, 128.78, 128.74, 128.48, 128.28, 127.99, 127.58,

125.64, 123.85, 114.80, 74.78, 70.16, 68.20, 59.57, 48.90, 48.52; IR (neat film): 3352, 1687, 1637, 1606, 1537, 1305 cm^{-1} . Anal. Calcd for $\text{C}_{28}\text{H}_{28}\text{N}_2\text{O}_5$: C, 71.17; H, 5.97; N, 5.93. Found: C, 71.26; H, 5.91; N, 5.99.



4-Hydroxy-3-[4-(phenylmethoxy)benzoylamino]-1-(phenylmethoxycarbonyl)-

hexahydroazepine (2) (From 14). Oxazoline **14** (23 mg, 0.0504 mmol) was dissolved in a mixture of ethanol (0.5 mL) and 1N HCl (2 mL), and heated at 60 °C for 3h. The reaction mixture was made basic by addition of sat. aq. NaHCO_3 and extracted with CHCl_3 (3x10 mL). The organic layer was washed with brine, dried, concentrated, and the residue was dissolved in methanol (2 mL). Triethylamine (0.2 mL) was added to this solution and stirred at room temperature for 24h. Concentration and purification by column chromatography over silica gel (3:1 ethyl acetate-hexane) to yield the hydrolyzed product **2** (15 mg, 63%).



4-Hydroxy-3-[4-(phenylmethoxy)benzoylamino]-1-(phenylmethoxycarbonyl)-

hexahydroazepine (2) (from 15). Tetrahydroazepine **15** (73 mg, 0.155 mmol) and $(\text{Ph}_3\text{P})_3\text{RhCl}$ (7 mg, 0.008 mmol) were dissolved in benzene (1.5 mL) and shaken under hydrogen pressure (25 psi) for 2 days. It was concentrated and purified by column chromatography (3:1 ethyl acetate-hexane) to afford **2** (69 mg, 94%) which was recrystallized from ether-hexane to afford a white crystalline solid. mp 107-109 °C; $[\alpha]_D^{25} = -83.65$ (c = 0.685, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 8.74 (d, J = 5.6 Hz, 1H), 7.88-7.78 (m, 2H), 7.48-7.26 (m, 10H), 7.06-6.95 (m, 2H), 5.48 (s, 1H), 5.19 (ABq, J = 12.4 Hz, 2H), 5.12 (s, 2H), 4.22-4.06 (m, 3H), 3.76 (m, 1H), 3.34 (dd, J = 15.3, 5.1 Hz, 1H), 2.78 (ddd, J = 13.7, 12.8, 3.5 Hz, 1H) 2.00-1.60 (m, 4H); ^{13}C NMR (100.5 MHz, CDCl_3): δ 168.64, 161.66, 157.80, 136.44, 136.26, 129.23, 128.76, 128.70, 128.39, 128.26, 127.86, 127.56, 125.73, 114.77, 79.90, 70.14, 67.96, 60.90, 50.62, 50.41, 32.88, 27.51; IR (neat film): 3360, 1680, 1635, 1606, 1481 cm^{-1} . Anal. Calcd for $\text{C}_{28}\text{H}_{30}\text{N}_2\text{O}_5$: C, 70.87; H, 6.37; N, 5.90. Found: C, 70.60; H, 6.17; N, 6.00.