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Diastereoselective Addition of Allylzinc Bromide to Imines Derived from (R)-Phenylglycine Amide

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General. Reagents were purchased from Aldrich Chemical Company and were used without further purification. Crotyl bromide was purchased from Fluka. (R)-Phenylglycine amide was provided by DSM (Geleen, The Netherlands). Commercial grade THF was used. Zinc-wool was cut prior to use. NMR spectra were recorded on either a Varian VXR-300 spectrometer (300 MHz) or a Varian Gemini Spectrometer (200 MHz). Chemical shifts are denoted in ppm and were referenced to residual solvent. Melting points (uncorrected) were determined on a Mettler FP-2 melting point apparatus equipped with a Mettler FP-21 microscope. HPLC analysis was performed on a HP1100 equipped with a Chiralpak or Crownpak analytical column.

Typical procedure for the synthesis of (R)-phenylglycine amide-imines 2-11. To a suspension of (R)-phenylglycine amide (200 mmol, 30.0 g) in CH₂Cl₂ (200 mL) at ambient temperature was added benzaldehyde (200 mmol, 21.2 g, 20.3 mL). The reaction mixture was stirred overnight at room temperature. MgSO₄ (10 g) was added and the reaction mixture was filtered. Evaporation of the solvent yields 2 as a colorless solid, which was recrystallized once from acetone/hexane (colorless crystals, 84%). m.p. 138-139 ºC. ¹H NMR (200MHz, CDCl₃): δ 8.31 (s, 1H), 7.80 (m, 2H), 7.25-7.51 (m, 10H), 7.03 (bs, 1H), 5.84 (bs, 1H), 4.99 (s, 1H). ¹³C NMR (50 MHz, CDCl₃): δ 160.79, 129.04, 126.25, 126.03, 125.45, 124.81, 74.50. Anal. Calcd for C₁₅H₁₄N₂O: C, 75.61; H, 5.92; N, 11.76. Found: C, 75.53; H, 5.99; N, 11.78.

(3). To a suspension of (R)-phenylglycine amide (15.0 g, 100 mmol) in CHCl₃, was added piperonal (15.0 g, 100 mmol) and a catalytic amount of p-TolSO₃H (0.3 g). The mixture was refluxed for 2 hours. MgSO₄ was added and the mixture was filtered. The filtrate was evaporated and the residue was recrystallized from Et₂O. (colorless powder, 85%) m.p. 138-139 ºC. ¹H NMR (200MHz, CDCl₃): δ 8.31 (s, 1H), 7.26-7.51 (m, 2H), 7.03 (bs, 1H), 5.84 (bs, 1H), 4.99 (s, 1H). ¹³C NMR (50 MHz, CDCl₃): δ 160.79, 129.04, 126.25, 126.03, 125.45, 124.81, 74.50. Anal. Calcd for C₁₆H₁₄N₂O₃: C, 68.08; H, 5.00; N, 9.92. Found: C, 67.74; H, 5.09; N, 9.97.

(4). (pale yellow powder, 90%) m.p. 139-140 ºC. ¹H NMR (200 MHz, DMSO-d₆): δ 9.5 (bs, 1H), 8.25 (s, 1H), 7.69 (d, J = 8.57 Hz, 2H), 7.20-7.38 (m, 5H), 6.80 (d, J = 8.55 Hz, 2H), 4.73 (s, 1H), 3.34 (bs, 2H). ¹³C NMR (50 MHz, DMSO-d₆): δ 183.25, 172.08, 163.12, 160.88, 159.24, 139.62, 129.28, 127.22, 126.30, 126.06, 114.40, 75.63. Anal. Calcd for C₁₅H₁₂N₂O₂: C, 70.85; H, 5.55; N, 11.02. Found: C, 70.02; H, 5.53; N, 10.69.

(5). (colorless crystals, 87%) m.p. 140-141 ºC. ¹H NMR (200MHz, CDCl₃): 8.73 (s, 1H), 7.59 (d, J = 3.17 Hz, 1H), 7.46-7.51 (m, 2H), 7.26-7.39 (m, 3H), 6.97-7.03 (m, 2H), 6.83-6.88 (m, 1H), 4.99 (s, 1H), 3.83 (s, 3H), 3.81 (s, 3H). ¹³C NMR (50 MHz, CDCl₃): δ 173.14, 152.29, 152.10, 138.03, 128.78, 127.22, 126.30, 125.75, 122.88, 117.37, 111.16, 109.78, 75.89, 54.62, 54.35. Anal. Calcd for C₁₅H₁₂N₂O₂: C, 68.44; H, 6.14; N, 9.26.

(6). (colorless crystals, 90%). m.p. 113-114 ºC. ¹H NMR (200MHz, CDCl₃): δ 8.93 (d, J = 1.46 Hz, 1H), 8.66 (d, J = 3.17 Hz, 1H), 8.33 (s, 1H), 8.14 (m, 1H), 7.26-7.47 (m, 6H), 6.9 (bs, 1H), 6.4 (bs, 1H), 5.00 (s, 1H). ¹³C NMR (50 MHz, CDCl₃): δ 171.34, 159.42, 150.69, 148.99, 139.03, 134.13, 130.11, 127.31, 126.35, 122.78, 75.84. Anal. Calcd for C₁₄H₁₃N₃O: C, 68.44; H, 5.48; N, 17.56. Found: C, 68.44; H, 5.14; N, 17.49.

(7). (colorless crystals, 89 %) m.p. 144-145 ºC. ¹H NMR (200MHz, CDCl₃): δ 8.07 (s, 1H), 7.26-7.56 (m, 6H), 7.04 (bs, 1H), 6.82-6.84 (m, 1H), 6.48-6.51 (m, 1H), 6.19 (bs, 1H), 4.92 (s, 1H). ¹³C NMR (50 MHz, CDCl₃): δ 169.88, 149.91, 149.57, 143.99, 137.67, 127.28, 126.52, 125.96, 114.45, 110.49, 75.64. Anal. Calcd for C₁₃H₁₂N₂O: C, 63.91; H, 4.95; N, 11.47. Found: C, 63.76; H, 4.97; N, 11.30.

(8). (pale yellow crystals, 80 %) m.p. 135-136 ºC. ¹H NMR (200MHz, CDCl₃): δ 8.37 (s, 1H), 7.28-7.49 (m, 7H), 7.07-7.11 (m, 1H), 7.02 (bs, 1H), 5.63 (bs, 1H), 4.96 (s, 1H). ¹³C NMR (50MHz,CDCl₃):δ 172.81, 154.71, 140.12, 137.69, 130.53, 128.5, 127.24, 126.47, 126.19, 125.87, 125.72, 114.45, 110.49, 75.64. Anal. Calcd for C₁₃H₁₁N₂O: C, 63.91; H, 4.95; N, 11.47. Found: C, 63.76; H, 4.97; N, 11.30.
Typical procedure for the allylation of \((R)\)-phenylglycine amide-imines. A solution of allylzinc bromide (1.5 eq) was prepared by adding allylbromide (438 mmol, 38.5 mL) to finely cut zinc-wool (438 mmol, 28.6 g) in THF (250 mL). The solution of allylzinc bromide was cooled to room temperature and was added dropwise to a solution of 2 (292 mmol, 70 g) in THF (150 mL) at 0 ºC. The reaction mixture was warmed to room temperature and was poured into water (500 mL). EtOAc (200 mL) was added and the mixture was stirred vigorously. After filtration through a glass filter, the organic phase was separated and the water layer was washed with EtOAc (2 x 100 mL). The combined organic layers were dried on MgSO4 and concentrated to give 12 as a colorless oil that crystallized on standing (colorless crystals, 93%). m.p. 89-90 ºC. H NMR (200 MHz, CDCl3): δ 7.15-7.31 (m, 10H), 7.05 (bs, 1H), 6.22 (bs, 1H), 5.67-5.78 (m, 1H), 5.01-5.09 (m, 2H), 3.95 (s, 1H), 3.68 (t, J = 7.0 Hz, 1H), 2.40 (dd, J = 7.0 Hz, 2H). 13C NMR (50 MHz, CDCl3): δ 173.57, 140.33, 137.05, 132.60, 126.33, 126.14, 125.56, 124.97, 124.80, 124.59, 115.32, 61.91, 59.12, 40.16. Anal. Calcd for C18H20N2O: C, 77.11; H, 7.19; N, 9.99. Found: C, 76.95; H, 7.22; N, 9.92.

13. (colorless crystals, 81 %) m.p. 110-111 ºC. H NMR (200 MHz, CDCl3) δ 7.21-7.23 (m, 5H), 6.93 (bs, 1H), 6.61-6.72 (m, 3H), 5.90 (s, 2H), 5.64-5.74 (bm, 2H), 5.00-5.07 (m, 2H), 3.99 (s, 1H), 3.60 (t, J = 6.96 Hz, 1H), 2.34-2.39 (dd, J = 6.96 Hz, 2H). 13C NMR (50 MHz, CDCl3) δ 174.24, 146.46, 145.30, 137.87, 135.21, 133.47, 127.31, 126.56, 125.72, 119.03, 116.25, 106.64, 105.47, 99.51, 59.92. 41.12. Anal. Calcd for C19H18N2O3: C, 70.35; H, 6.21; N, 8.64. Found: C, 70.28; H, 6.33; N, 8.59.

14. (colorless solid, 90%) m.p. 158-159 ºC. H NMR (200 MHz, DMSO-d6) δ 9.20 (bs, 1H), 7.54 (s, 1H), 7.22-7.29 (m, 5H), 7.10 (d, J = 8.4 Hz, 2H), 6.71 (d, J = 8.4 Hz, 2H), 5.70 (m, 1H), 5.00 (m, 2H), 3.87 (s, 1H), 3.50 (m, 1H), 2.25 (m, 2H). 13C NMR (50 MHz, DMSO-d6) δ 173.2, 155.2, 139.4, 134.8, 132.6, 127.2, 127.0, 125.9, 115.9, 113.9, 61.5, 58.7, 41.9. Anal. Calcd for C19H18N2O3: C, 72.95; H, 6.80; N, 9.45. Found: C, 72.90; H, 6.84; N, 9.45.

15. (yellow crystals, 93%) m.p. 121-122 ºC. H NMR (200 MHz, CDCl3): δ 7.48 (bs, 1H), 7.12-7.23 (m, 5H), 5.73-5.85 (m, 2H), 4.99-5.07 (m, 2H), 3.95 (s, 1H), 3.85 (t, J = 6.96 Hz, 1H), 3.86 (s, 3H), 3.60 (s, 3H), 2.40-2.45 (dd, J = 6.96 Hz, 2H). 13C NMR (50 MHz, CDCl3): δ 198.91, 174.71, 152.02, 150.14, 138.30, 134.39, 129.75, 127.21, 126.40, 125.75, 115.72, 113.14, 110.91, 110.28, 63.36, 57.30, 54.15, 54.05, 39.02. Anal. Calcd for C20H22N2O2: C, 70.57; H, 7.11; N, 8.23. Found: C, 70.38; H, 7.16; N, 8.13.

16. (pale yellow powder, 43 %). m.p. 96-97 ºC. H NMR (200 MHz, CDCl3) δ 8.58 (s, 2H), 7.77 (d, J = 7.69 Hz, 1H), 7.36 (m, 1H), 7.15 (m, 5H), 6.41 (bs, 1H), 5.45-5.65 (m, 2H), 5.00 (m, 2H), 3.89 (s, 1H), 3.79 (t, J = 7.04 Hz, 1H), 2.38 (m, 2H). 13C NMR (50 MHz, DMSO-d6): δ 172.91, 147.64, 147.00, 138.99, 138.34, 134.94, 133.81, 127.06, 125.92, 122.97, 116.72, 61.72, 56.97, 41.27. Anal. Calcd for C17H16N2O: C, 72.57; H, 6.81; N, 14.93. Found: C, 72.50; H, 6.81; N, 15.1.

17. (colorless solid, 88 %). m.p. 77-78 ºC. H NMR (200 MHz, CDCl3) δ 7.21-7.35 (m, 6H), 6.30-6.33 (m, 1H), 6.17-6.19 (m, 1H), 6.14 (bs, 1H), 5.69-5.90 (m, 1H), 5.07-5.17 (m, 2H), 4.11 (s, 1H), 3.74 (t, J = 6.84 Hz, 1H), 2.47-2.55 (m, 2H).
(18). (yellow crystals, 90%) m.p. 62-63 °C.
\[ \text{H NMR (200 MHz, CDCl}_3 \]: } \delta 7.22-7.30 (m, 6H), 6.90-6.97 (m, 3H), 5.72-5.84 (m, 1H), 4.46-5.58 (m, 1H), 4.98-5.15 (m, 2H), 3.79-3.88 (s, 1H), 3.89 (s, 1H), 3.70 (d, \( J = 6.8 \) Hz, 1H), 2.41-2.65 (m, 1H), 2.31-2.41 (m, 1H), 0.92 (d, \( J = 6.95 \) Hz, 3H), 0.78 (d, \( J = 6.59 \) Hz, 3H). 13C NMR (50 MHz, CDCl3) \( \delta 174.02, 145.95, 137.67, 132.99, 127.28, 126.56, 125.83, 125.03, 123.57, 122.82, 116.66, 62.75, 55.53, 41.63 \). Anal. Calcd for \( \text{C}_{16}\text{H}_{18}\text{N}_2\text{O} \): C, 67.10; H, 6.34; N, 9.78. Found: C, 66.95; H, 6.32; N, 9.73.

(19) (yellow oil, 89%) 1H-NMR (300MHz, CDCl3) \( \delta 7.18-7.26 \) (m, 5H), 6.85 (bs, 1H), 6.77 (bs, 1H), 5.75-5.89 (m, 1H), 4.95-5.06 (m, 2H), 4.28 (s, 1H), 2.33-2.38 (m, 1H), 2.15-2.18 (m, 1H), 1.95-2.06 (m, 1H), 0.76 (s, 9H). 13C NMR (50MHz, CDCl3) \( \delta 176.24, 139.44, 137.76, 128.47, 127.80, 65.89, 63.89, 36.09, 35.22, 26.91 \). MS (CI): m/z=261 (M+1).

(20). (yellowish oil, 77 %). 1H NMR (200MHz, CDCl3) \( \delta 7.20-7.34 \) (m, 6H), 6.02 (bs, 1H), 5.76 (m, 1H), 5.07 (d, \( J = 10.6 \) Hz, 1H), 5.03 (s, 1H), 4.25 (s, 1H), 2.37 (m, 1H), 2.15-2.18 (m, 1H), 1.95-2.06 (m, 1H), 0.76 (s, 9H). 13C NMR (50 MHz, CDCl3) \( \delta 174.9, 140.7, 133.79, 127.3, 126.5, 125.9, 116.1, 63.3, 62.5, 44.9, 21.1 \). Anal. Calcd for \( \text{C}_{15}\text{H}_{22}\text{N}_2\text{O} \): C, 73.13; H, 9.00; N, 11.37. Found: C, 73.13; H, 9.23; N, 11.16.

(21). (pale yellow solid, 94%) m.p. 56-57 °C.
\[ \text{H NMR (200 MHz, CDCl}_3 \]: } \delta 7.19-7.34 (m, 6H), 6.02 (bs, 1H), 5.76 (m, 1H), 5.07 (d, \( J = 10.6 \) Hz, 1H), 5.03 (s, 1H), 4.25 (s, 1H), 2.37 (m, 1H), 2.15-2.18 (m, 1H), 1.95-2.06 (m, 1H), 0.76 (s, 9H). 13C NMR (50 MHz, CDCl3) \( \delta 174.02, 137.24, 134.00, 126.32, 125.54, 124.96, 114.74, 62.39, 58.53, 32.23, 26.79, 16.45, 14.53 \). Anal. Calcd for \( \text{C}_{13}\text{H}_{22}\text{N}_2\text{O} \): C, 73.13; H, 9.00; N, 11.37. Found: C, 73.13; H, 9.23; N, 11.16.

(22). A solution of crotylzinc bromide (1.5 eq) was prepared by adding crotylbromide (62.6 mmol, 6.4 mL) to finely cut zinc-wool (62.6 mmol, 4.0 g) in THF (50 mL). The solution of crotylzinc bromide was cooled to room temperature and was added dropwise to a solution of 2 (41.7 mmol, 10 g) in THF (30 mL) at 0°C. The reaction mixture was warmed to room temperature and was poured into water (100 mL). EtOAc (30 mL) was added and the mixture was stirred vigorously. After filtration through a glass filter, the organic phase was separated and the water layer was extracted with EtOAc (2 x 30 mL). The combined organic phase was dried over MgSO4 and concentrated to give 22. (orange oil, 98%). 1H NMR (200 MHz, CDCl3) \( \delta 6.99-7.29 \) (m, 2x10H), 6.69 (bs, 1H), 6.44 (bs, 1H), 5.72-5.84 (m, 1H), 4.46-5.58 (m, 1H), 4.98-5.15 (m, 2x2H), 3.79 (s, 1H), 3.89 (s, 1H), 3.70 (d, \( J = 5.13 \) Hz, 1H), 3.36 (d, \( J = 8.42 \) Hz, 1H), 2.41-2.65 (m, 1H), 2.31-2.41 (m, 1H), 0.92 (d, \( J = 6.95 \) Hz, 3H), 0.78 (d, \( J = 6.59 \) Hz, 3H). 13C NMR (50 MHz, CDCl3) \( \delta 175.5, 140.7, 133.79, 127.3, 126.8, 126.7, 126.3, 126.2, 126.1, 125.8, 115.3, 114.6, 65.3, 64.9, 62.6, 62.5, 43.2, 42.2, 16.3, 15.0.

(23). A solution of methallylzinc bromide (1.5 eq) was prepared by adding methallylbromide (62.6 mmol, 6.3 mL) to finely cut zinc-wool (62.6 mmol, 4.0 g) in THF (50 mL). The solution of methallylzinc bromide was cooled to room temperature and was added dropwise to a solution of 2 (41.7 mmol, 10 g) in THF (30 mL) at 0°C. The reaction mixture was warmed to room temperature and was poured into water (100 mL). EtOAc (30 mL) was added and the mixture was stirred vigorously. After filtration through a glass filter, the organic phase was separated and the water layer was extracted with EtOAc (2 x 30 mL). The combined organic layers were dried over MgSO4 and concentrated to give 23 that can be crystallized from Et2O (colorless solid, 98%). m.p. 104-109 °C. 1H NMR (50 MHz, CDCl3) \( \delta 175.1, 140.7, 140.5, 136.9, 127.4, 127.2, 126.8, 126.2, 125.9, 125.7, 112.4, 63.7, 62.5, 44.9, 21.1.

Typical procedure for the catalytic hydrogenation of (R)-phenylglycine amide-homoallylamines. Homoolylamine 12 (15.0 mmol, 4.2 g) was dissolved in MeOH (100 mL). Water (10 mL), acetic acid (2.5 mL), and Pd(10%)/C (0.6 gram) were added successively. The mixture was shaken under an atmosphere of H2 (30 psi) for 18 hrs at room temperature. The MeOH was evaporated under reduced pressure. The residue was diluted with water (50 mL) and...
was adjusted to pH = 10 with 10 % NaOH. The water phase was extracted with CH₂Cl₂ (3 x 40 mL). The combined organic phase was dried on MgSO₄ and filtered. After evaporation of the CH₂Cl₂, pentane was added to the residue. Filtration through a glass filter yields crystalline phenylacetamide (0.8 g, 41%). Evaporation of the pentane left \((R)\) as a colorless oil that slowly solidified on standing (1.1 g, 49%).

\[ \alpha \] +18.3 (c = 1.06, CHCl₃). 1H NMR (200MHz, CDCl₃): δ 7.20-7.34 (m, 5H), 3.89 (t, J = 6.84 Hz, 1H), 1.97 (bs, 2H), 1.60-1.69 (m, 2H), 1.21-1.36 (m, 2H), 0.93 (t, J = 7.08 Hz, 3H). 13C NMR (50 MHz, CDCl₃): δ 141.21, 126.95, 125.81, 125.33, 53.85, 37.93, 17.81, 12.41.

\[(R)\] (colorless oil, 64%). \[ \alpha \] +17.0 (c = 0.99, CHCl₃). 1H NMR (200MHz, CDCl₃): δ 2.38-2.44 (m, 1H), 0.85-1.40 (m, 8H), 0.74-0.84 (m, 9H). 13C NMR (50 MHz, CDCl₃): δ 54.5, 35.4, 31.6, 18.0, 17.5, 15.3, 12.5.

\[(R)\] (colorless oil, 88 %). \[ \alpha \] -8.1 (c = 1.04, CHCl₃). 1H NMR (200MHz, CDCl₃): δ 6.83 (s, 1H), 6.74 (s, 2H), 5.92 (s, 2H), 3.80 (bs, 1H), 1.58-1.63 (bm, 2H), 1.18-1.26 (bm, 2H), 0.89 (t, J = 7.3 Hz, 3H). 13C NMR (50 MHz, CDCl₃): δ 146.08, 144.67, 139.35, 117.85, 106.32, 105.00, 99.22, 54.22, 40.30, 18.15, 12.44.

Typical procedure for the non-reductive auxiliary removal (retro-Strecker method). To CH₂Cl₂ (600 mL), cooled with an ice bath, was added DMF (144 mmol, 10.6 g, 11.2 mL). Oxalylchloride (144 mmol, 18.5 g, 12.7 mL) was added dropwise. After the formation of gas (CO and CO₂) had ceased, a solution of amide 20 (97.6 mmol, 24.0 g) in CH₂Cl₂ (100 mL) was added dropwise in 10 minutes. Triethylamine (97.5 mmol, 9.8 g, 13.48 mL) was added dropwise in 5 minutes turning the reaction mixture orange-red. The reaction was stirred at room temperature for 30 minutes. Water (300 mL) was added and the organic phase was separated. The aqueous layer was washed with CH₂Cl₂ (100 mL) additionally. The organic layer was dried over Na₂SO₄ and concentrated to give the crude nitrile 28 (22.0 g, 95.6 mmol, 98%) as an orange oil. The crude nitrile (7.4 g; 33 mmol) was dissolved in ethanol (150 mL) and K₂CO₃ (8.97 g; 65 mmol; 2 eq.) was added. The reaction mixture was refluxed for 2 hours. The ethanol was evaporated and the residue was taken up in water/CH₂Cl₂. The organic phase was separated and dried over Na₂SO₄. Evaporation of the solvent gives the crude imine 30 (6.2 g, 30.8 mmol, 93%) as the only product. 1H NMR (200MHz, CDCl₃): δ 8.11 (s, 1H), 7.68-7.71 (m, 2H), 7.27-7.37 (m, 3H), 5.61-5.70 (m, 1H), 4.91-4.99 (m, 2H), 2.81-2.87 (m, 1H), 2.33-2.41 (m, 2H), 1.82-1.89 (m, 1H), 0.86-0.91 (m, 6H). 13C NMR (50 MHz, CDCl₃): δ 157.99, 135.01, 134.90, 128.74, 126.96, 126.65, 114.87, 75.79, 36.55, 31.14, 18.32, 17.11. MS (EI) [m/e, %]: 201 [M⁺, 4.3]; 201 [M⁺-C₃H₅, 100]. Imine 30 (2.0 g, 9.94 mmol) was dissolved in a 50% aqueous THF. 3 equivalents of NH₂OH.HCl (2.08 g, 29.8 mmol) were added and the reaction mixture was stirred overnight at ambient temperature. The THF was evaporated under reduced pressure and the residue was treated with aqueous HCl (30 %) until pH = 1. The aqueous phase was extracted with EtOAc. The water phase was adjusted to pH = 10 with aqueous NaOH (33 %) and was extracted with CH₂Cl₂. After drying over Na₂SO₄, the solvent was evaporated furnishing the homoallylamine 32 (yellowish oil, 1.03 g, 9.14 mmol, 92 % (98 % overall from 20), er 99/1). 1H NMR (200MHz, CDCl₃): δ 6.83 (s, 1H), 6.74 (s, 2H), 5.92 (s, 2H), 3.80 (bs, 1H), 1.58-1.63 (bm, 2H), 1.18-1.26 (bm, 2H), 0.89 (t, J = 7.3 Hz, 3H). 13C NMR (50 MHz, CDCl₃): δ 134.61, 115.54, 54.23, 37.50, 31.18, 17.50, 15.99. \[ \alpha \] -8.1 (c = 1.04, CHCl₃).

(31) (yellowish oil, 78% overall from 12. 1H NMR (200MHz, CDCl₃): δ 7.10-7.25 (m, 5H), 5.60-5.80 (m, 1H), 5.00-5.20 (m, 2H), 3.90-4.00 (m, 1H), 2.20-2.50 (m, 2H). 13C NMR (50 MHz, CDCl₃): δ 145.85, 135.49, 128.35, 126.91, 126.34, 117.50, 55.37, 44.19.
Signal 1: DAD1 A, Sig=200.8 Ref=400.60

Peak Ret Time Type Width Area Height Area
# [min] [min] [mAU*a] [mAU] [mAU] [mAU] [mAU]
1 11.945 MM 0.9024 3035.57544 56.06339 96.5701
2 13.883 MM 1.1450 1278.01596 1.56940 3.4299

Totals: 3142.39140 57.63278

Results obtained with enhanced integrator!

*** End of Report ***
Injection Date: 3/14/2001 6:21:53 PM  Seq. Line: 1
Sample Name:  Vial: 84
Inj Volume: 10 µl
Method: C:\HPCHEM\2\METHODS\MS0078.M
Chiracel AD

Signal 1: DAD1 A, Sig=260.8 Ref=400.60

Peak RetTime Type Width Area Height Area
# (min) (min) [mAU*sec] [mAU] [mAU]
--- ------ -------- -------- ------- ----
1 10.754 MM 1.3234 24.63638 3.10268e-1 2.5036
2 14.808 MM 1.6755 959.41425 9.54345 97.4964
Totals: 984.05662 9.85372

Results obtained with enhanced integrator:

*** End of Report ***
Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000

Signal 1: DAD1 A, Sig=288.8 Ref=420.66

<table>
<thead>
<tr>
<th>Peak RetTime Type</th>
<th>Width</th>
<th>Area</th>
<th>Height</th>
<th>Area</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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<tr>
<td>1 22.978 PB</td>
<td>2.4471</td>
<td>4807.65620</td>
<td>29.22644</td>
<td>100.0000</td>
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Totals: 4807.65620 29.22644

Results obtained with enhanced integrator:

*** End of Report ***
Data File C: \HPChem\2\DATA\MSS007R\14030102.D

Injection Date : 3/14/2001 6:48:30 PM  Seq. Line : 2
Sample Name : Vial : 85
Acq. Operator : M.Walderbos/E.v.echten  Inj : 1
Method : C: \HPChem\2\METHODS\MSS007.C.M
Last changed : 3/14/2001 5:54:04 PM by M.Walderbos/E.v.echten

Chiralcel AD

![Chiralcel AD graph]

Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 A, Sig=288.8 Ref=400.60

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</thead>
<tbody>
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<td>1 29.504 MM</td>
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<td></td>
<td>3.4633</td>
<td>2265.29687</td>
<td>10.90245</td>
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</tbody>
</table>

Totals : 2265.29687 10.90245

Results obtained with enhanced integrator!

*** End of Report ***