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ASYMMETRIC INTERMOLECULAR CARBENOID C-H INSERTIONS CATALYZED BY RHODIUM(II) (*S*)-*N*-(*p*-DODECYLPHENYL)SULFONYLPROLINATE

Huw M. L. Davies* and Tore Hansen

Department of Chemistry, State University of New York at Buffalo,

Buffalo, New York 14260-3000

Supplementary Data

Experimental Data for Compounds 2-5 and 11

Methyl α-cyclopentylbenzeneacetate (2b). A solution of methyl phenyldiazoacetate (304 mg, 1.72 mmol) in 30 ml dry cyclopentane was added dropwise over 90 min. to a refluxing solution of $Rh_2(DOSP)_4$ (28 mg, 0.015 mmol) in 5 ml dry cyclopentane. The solvent was removed *in vacuo*, and after flash chromatography with 2.5 % Et_2O / pet ether, 316 mg colorless oil was obtained (84%); 87%ee (Chiralcel OD, FR 1.0 ml / min., 0.6% i-propanol / hexane, R_t = 9.2 and 10.8 min, UV 254nm); $[\alpha]^{25}_D$ = -50.2° (c 0.518, $CDCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.37-7.24 (m, 5 H), 3.65 (s, 3 H), 3.29 (d, 1 H, *J* = 6.6 Hz), 2.57 (m, 1 H), 1.70-1.39 (m, 5 H), 1.20-1.29 (m, 1 H), 0.97-1.04 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 174.5, 139.0 , 128.4 (CH), 128.2 (CH), 128.1 (CH), 57.6 (CH), 51.7 (CH_3), 43.4 (CH), 31.5 (CH_2), 30.6 (CH_2), 25.0 (CH_2), 24.7 (CH_2); IR (neat): 3027, 2951, 2867, 1734, 1602, (cm^{-1}); Anal. calcd for $C_{14}H_{18}O_2$: C, 77.03; H, 8.31. Found: C, 77.16; H, 8.34.

Methyl α-cyclohexylbenzeneacetate (3b). A solution of methyl phenyldiazoacetate (300 mg, 1.70 mmol) in 30 ml anhydrous cyclohexane was added dropwise over 90 min. to a refluxing solution of $Rh_2(DOSP)_4$ (28 mg, 0.015 mmol) in 5 ml anhydrous cyclohexane. The solvent was removed *in vacuo*, and after flash chromatography with 2.5 % Et_2O / pet ether, 327 mg colorless oil was obtained (83%); 81%ee (Chiralcel OD, FR 1.0 ml / min., 0.6% i-propanol / hexane, R_t = 8.4 and 10.0 min, UV 254nm); $[\alpha]^{23}_D$ = -35.1° (c 0.57, $CDCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.31-7.22 (m, 5 H), 3.64 (s, 3 H), 3.22 (d, 1 H, *J* = 10.6 Hz), 2.03-1.97 (m, 1 H), 1.81-1.56 (m, 4 H), 1.32-1.01 (5 H), 0.87-0.70 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 174.4, 137.9, 128.6 (CH), 128.4 CH), 127.2 (CH), 58.8 (CH), 51.7 (CH_3), 41.0 (CH), 32.0 (CH_2), 30.4 (CH_2), 26.2 (CH_2), 25.94 (CH_2), 25.90 (CH_2); IR (neat): 3028, 2926, 2850, 1735, 1497 cm^{-1} ; Anal. calcd for $C_{15}H_{20}O_2$: C, 77.55; H, 8.68. Found: C, 77.47; H, 8.69.

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Huw M.L. Davies, et al.
Department of Chemistry
State University of New York at Buffalo
NSM Complex
Box 603000
Buffalo NY 14260-3000
Ms. Type: c:communication

Methyl α-cycloheptylbenzeneacetate (4b). A solution of methyl phenyldiazoacetate (195 mg, 1.10 mmol) in 20 ml dry cycloheptane was added dropwise over 90 min. to a refluxing solution of $Rh_2(DOSP)_4$ (19 mg, 0.01 mmol) in 5 ml dry cycloheptane. The solvent was removed *in vacuo*, and after flash chromatography with 2.5 % Et_2O / pet ether, 227 mg colorless oil was obtained (84%); 70%ee (Chiralcel OD, FR 1.0 ml / min., 0.6% i-propanol / hexane, R_t = 8.2 and 9.7 min, UV 254nm); $[\alpha]^{24}_D$ = -31.7° (c 0.69, $CDCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.34-7.24 (m, 5 H), 3.67 (s, 3 H), 3.30 (d, 1 H, *J* = 11.0 Hz), 2.27-2.24 (m, 1 H), 1.77-1.24 (m, 11 H), 1.03-0.92 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 174.7, 138.4, 128.6 (CH), 128.4 (CH), 127.1 (CH), 58.8 (CH), 51.7 (CH_3), 42.1 (CH), 33.1 (CH_2), 31.2 (CH_2), 28.3 (CH_2), 28.2 (CH_2), 26.2 (CH_2), 26.1 (CH_2); IR (neat): 3027, 2924, 2853, 1734, 1599, 1495 cm^{-1} ; Anal. calcd for $C_{16}H_{22}O_2$: C, 78.01; H, 9.00. Found: C, 77.99; H, 8.93.

Methyl α-cyclopentyl-4-methoxy-benzeneacetate (2a). A solution of methyl 4-methoxy-phenyldiazoacetate (188 mg, 0.91 mmol) in 30 ml dry cyclopentane was added dropwise over 90 min. to a refluxing solution of $Rh_2(DOSP)_4$ (18 mg, 0.01 mmol) in 5 ml dry cyclopentane. The solvent was removed *in vacuo*, and after flash chromatography with 10 % Et_2O / pet ether, 125 mg colorless oil was obtained (55%); 83%ee (Chiralcel OD, FR 1.0 ml / min., 1.0% i-propanol / hexane, R_t = 7.0 and 8.3 min, UV 254nm); $[\alpha]^{24}_D$ = -39.2° (c 0.53, $CDCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.36 (d, 2 H, *J* = 8.8 Hz), 6.83 (d, 2 H *J* = 8.8 Hz), 3.78 (s, 3 H), 3.63 (s, 1 H), 3.22 (d, 1 H, *J* = 11.0 Hz), 2.52 (m, 1 H), 1.88 - 1.39 (m, 5 H), 1.25 - 1.18 (m, 1 H), 1.01 - 0.90 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 174.7, 158.7, 131.1, 129.2 (CH), 113.8 (CH), 56.8 (CH_3), 55.1 (CH), 51.7 (CH_3), 43.4 (CH), 31.4 (CH_2), 30.6 (CH_2), 25.1 (CH_2), 24.8 (CH_2); IR (neat): 3034, 2952, 2865, 1734,1608,1582 cm^{-1}; Anal. calcd for $C_{15}H_{20}O_3$: C, 72.55; H, 8.12. Found: C, 72.47; H, 8.20.

Methyl α-cyclohexyl-4-methoxy-benzeneacetate (3a). A solution of methyl 4-methoxy-phenyldiazoacetate (195 mg, 0.94 mmol) in 30 ml anhydrous cyclohexane was added dropwise over 90 min. to a refluxing solution of $Rh_2(DOSP)_4$ (18 mg, 0.010 mmol) in 10 ml anhydrous cyclohexane. The solvent was removed *in vacuo*, and after flash chromatography with 10 % Et_2O / pet ether, 211 mg white solid was obtained (85%); 69%ee (Chiralcel OD, FR 1.0 ml / min., 1.0% i-propanol / hexane, R_t = 6.4 and 7.5 min, UV 254nm); $[\alpha]^{24}_D$ = -23.5° (c 0.714, $CDCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.23 (d, 2 H, J = 8.4 Hz), 6.84 (d, 2H, J = 8.4 Hz), 3.78 (s, 3 H), 3.63 (s, 3 H), 3.16 (d, 1 H, *J* = 10.6 Hz), 1.93 (m, 1 H), 1.78-1.56 (m, 4 H), 1.35-0.99 (5 H), 0.71 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 174.7, 158.7, 129.9, 129.5 (CH), 113.8 (CH), 57.8 (CH), 55.1 (CH_3), 51.6 (CH_3), 41.0 (CH), 31.9 (CH_2), 30.3 (CH_2), 26.2 (CH_2), 25.9 (CH_2), 25.9 (CH_2); IR (neat): 3034, 2924, 2845, 1733, 1610, 1583, cm^{-1}. Anal. calcd for $C_{16}H_{22}O_3$: C, 73.25; H, 8.45. Found: C, 73.37; H, 8.54.

Methyl α-cycloheptyl-4-methoxy-benzeneacetate (4a). A solution of methyl 4-methoxy-phenyldiazoacetate (207 mg, 1.00 mmol) in 15 ml dry cycloheptane was added dropwise over 4 hrs to a refluxing solution of $Rh_2(DOSP)_4$ (19 mg, 0.01 mmol) in 5 ml dry cycloheptane. The solvent was

removed *in vacuo*, and after flash chromatography with 5 % Et_2O / pet ether, 216 mg colorless oil was obtained (78%); 62%ee (Chiralcel OD, FR 1.0 ml / min., 1.0% i-propanol / hexane, R_t = 6.4 and 7.3 min, UV 254nm); $[\alpha]^{24}_D$ = -22.5° (c 0.222, $CDCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.23 (d, 2 H, *J* = 6.6 Hz), 6.82 (d, 2 H, *J* = 6.6 Hz), 3.78 (s, 3 H), 3.63 (s, 3 H), 3.24 (d, 1 H, *J* = 8.2 Hz), 2.20 (m, 1 H), 1.86-1.24 (m, 11 H), 0.95 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 158.7, 130.5, 129.6 (CH), 113.8 (CH), 57.9 (CH), 55.1 (CH_3), 51.6 (CH_3), 42.1 (CH), 33.1 (CH_2), 33.2 (CH_2), 28.3 (CH_2), 28.2 (CH_2), 26.3 (CH_2), 26.1 (CH_2); IR (neat): 3033, 2997, 2856, 1734,1610, 1582 cm^{-1} ; Anal. calcd for $C_{17}H_{24}O_3$: C, 73.88; H, 8.75. Found: C, 74.01; H, 8.71.

Methyl α-cyclopentyl-4-chloro-benzeneacetate (2c). A solution of methyl 4-chloro-phenyldiazoacetate (216 mg, 1.02 mmol) in 10 ml dry cyclopentane was added dropwise over 90 min. to a refluxing solution of $Rh_2(DOSP)_4$ (19 mg, 0.01 mmol) in 5 ml dry cyclopentane. The solvent was removed *in vacuo*, and after flash chromatography with 5 % Et_2O / pet ether, 202 mg colorless oil was obtained (78%); 89%ee (Eu tris [3-(heptafluoropropylhydroxymethylene) - (+) -camphorate]; $[\alpha]^{24}_D$ = -49.2° (c 0.650, $CDCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.27 (s, 4 H), 3.64 (s, 3 H), 3.25 (d, 1 H, *J* = 11.0 Hz), 1.86 (m, 1 H), 1.69 - 1.36 (m, 5 H), 1.29 - 1.17 (m, 1 H), 0.99 - 0.90 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 174.1, 137.4, 133.0, 129.5 (CH), 128.6 (CH), 57.0 (CH), 51.9 (CH_3), 43.4 (CH), 31.4 (CH_2), 30.6 (CH_2), 25.0 (CH_2), 24.7 (CH_2); IR (neat): 3027, 2950,2851, 1735, 1593 cm^{-1}; Anal. calcd for $C_{14}H_{17}ClO_2$: C, 66.53; H, 6.78. Found: C, 66.62; H, 6.80.

Methyl α-cyclohexyl-4-chloro-benzeneacetate (3c). A solution of methyl 4-chloro-phenyldiazoacetate (212 mg, 1.00 mmol) in 10 ml anhydrous cyclohexane was added dropwise over 60 min. to a refluxing solution of $Rh_2(DOSP)_4$ (19 mg, 0.010 mmol) in 5 ml anhydrous cyclohexane. The solvent was removed *in vacuo*, and after flash chromatography with 5 % Et_2O / pet ether, 244 mg colorless oil was obtained (91%); 86%ee (Eu tris [3-(heptafluoropropylhydroxymethylene) - (+) -camphorate]); $[\alpha]^{24}_D$ = -18.1° (c 0.342, $CDCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.27 (s, 4 H), 3.64 (s, 3 H), 3.19 (d, 1 H, *J* = 10.6 Hz), 1.95 (m, 1 H), 1.79-1.55 (m, 4 H), 1.31-1.00 (5 H), 0.72 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 174.0, 136.3, 133.0, 129.9 (CH), 128.5 (CH), 58.0 (CH), 51.7 (CH_3), 41.0 (CH), 31.8 (CH_2), 30.3 (CH_2), 26.1 (CH_2), 25.84 (CH_2), 25.81 (CH_2); IR (neat): 3027, 2950, 2851,1733, 1592 cm^{-1}. Anal. calcd for $C_{15}H_{19}ClO_2$: C, 67.54; H, 7.18. Found: C, 67.56; H, 7.16.

Methyl α-cycloheptyl-4-chloro-benzeneacetate (4c). A solution of methyl 4-chloro-phenyldiazoacetate (212 mg, 1.00 mmol) in 10 ml dry cycloheptane was added dropwise over 60 min. to a refluxing solution of $Rh_2(DOSP)_4$ (19 mg, 0.01 mmol) in 5 ml dry cycloheptane. The solvent was removed *in vacuo*, and after flash chromatography with 5 % Et_2O / pet ether, 271 mg colorless oil was obtained (96%); 81%ee (Eu tris [3-(heptafluoropropylhydroxymethylene) - (+) -camphorate]); $[\alpha]^{25}_D$ = -21.6° (c 0.564, $CDCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 7.26 (s, 4 H), 3.63 (s, 3 H), 3.28(d, 1 H, *J* = 11.0 Hz), 2.20 (m, 1 H), 1.75-1.25 (m, 11 H), 0.94 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 174.3,

136.8, 133.0, 130.0 (CH), 128.6 (CH), 58.1 (CH), 51.8 (CH_3), 42.2 (CH), 33.0 (CH_2), 31.2 (CH_2), 28.2 (CH_2), 28.1 (CH_2), 26.2 (CH_2), 26.1 (CH_2); IR (neat): 3026, 2942, 2852, 1734, 1592 cm^{-1}

Methyl α-phenyl-2-tetrahydrofuran acetate (11b, 12b). A solution of methyl phenyldiazoacetate (375 mg, 2.12 mmol), in 20 ml dry THF was added dropwise over 2 hrs to a refluxing solution of $Rh_2(DOSP)_4$ (40 mg, 0.021 mmol) in 5 ml dry THF. The solvent was removed *in vacuo*, and flash chromatography with 10 % Et_2O / pet ether gave separation of the two diastereomers in a total recovery of 381 mg (82%) colorless oil (2.3 / 1 de, 60% ee major ds (Eu tris [3-(heptafluoropropylhydroxymethylene) - (+) -camphorate]), 61% ee minor ds (Chiralcel OD, FR 1.0 ml / min., 1.0% i-propanol / hexane, R_t = 13.7 and 14.6 min, UV 254nm)); $[\alpha]^{25}_D$ = -53.3° (c 0.652, $CDCl_3$, major isomer); ^1H NMR (300 MHz, $CDCl_3$) (major diastereomer) δ 7.40 - 7.26 (m, 5 H), 4.46 (dt, 1 H, *J* = 8.3, 7.0 Hz), 3.84 - 3.70 (m, 2 H), 3.67 (s, 3H), 3.62 (d, 1 H, *J* = 8.4 Hz), 2.11 (m, 1 H), 1.88 (m, 1 H), 1.67 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.6, 136.7, 128.56 (CH), 128.54 (CH), 127.5 (CH), 79.9 (CH), 68.3 (CH_2), 56.7 (CH), 52.0 (CH_3), 30.1 (CH_2), 25.5 (CH_2); IR (neat): 3030, 2954, 2851, 1734, 1601, 1585; ^1H NMR (300 MHz, $CDCl_3$) (minor diastereomer) δ 7.36 - 7.26 (m, 5 H), 4.52 (dt, 1 H, *J* = 9.9, 7.0 Hz), 3.95 - 3.79 (m, 2 H), 3.69 (s, 3H), 3.52 (d, 1 H, J = 9.9 Hz), 1.90 - 1.77 (m, 2 H), 1.68 (m, 1 H), 1.43 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.0, 135.8, 128.7 (CH), 128.4 (CH), 127.7 (CH), 80.6 (CH), 68.4 (CH_2), 57.5 (CH), 52.1 (CH_3), 39.5 (CH_2), 25.4 (CH_2);

Methyl α-(4-methoxy-phenyl)-2-tetrahydrofuran acetate (11a, 12a). A solution of methyl 4-methoxy-phenyldiazoacetate (226 mg, 1.09 mmol), in 15 ml dry THF was added dropwise over 2 hrs to a refluxing solution of $Rh_2(DOSP)_4$ (19 mg, 0.01 mmol) in 5 ml dry THF. The solvent was removed *in vacuo*, and flash chromatography with 20 % Et_2O / pet ether gave separation of the two diastereomers in a total recovery of 173 mg (63%) colorless oil (2.5 / 1 de, 68% ee major ds (Chiralcel OD, FR 1.0 ml / min., 2.0% i-propanol / hexane, R_t = 12.5 and 15.2 min, UV 254nm), 66% ee minor ds (Chiralcel OD, FR 1.0 ml / min., 2.0% i-propanol / hexane, R_t = 12.0 and 15.3 min, UV 254nm));.$[\alpha]^{25}_D$ = -57.0° (c 0.228, $CDCl_3$, major isomer), $[\alpha]^{25}_D$ = -23.2° (c 0.284, $CDCl_3$, minor isomer); ^1H NMR (300 MHz, $CDCl_3$) (major diastereomer) δ 7.30 (d, 2 H, J = 8.6 Hz), 6.87 (d, 2H, 8.6 Hz), 4.52 (dt, 1 H, *J* = 8.3, 7.0 Hz), 3.83 - 3.69 (m, 2 H), 3.78 (s, 3H), 3.66 (s, 3 H), 3.57 (d, 1 H, *J* = 8.4 Hz), 2.09 (m, 1 H), 1.86 (m, 2 H), 1.63 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.8, 158.9, 129.6 (CH), 128.7 (CH), 114.0 (CH), 80.0 (CH), 68.3 (CH_2), 55.9 (CH), 55.2 (CH_3), 52.0, 30.1 (CH_2), 25.6 (CH_2); IR (neat): 3034, 2951, 2872, 1733, 1609, 1585;^1H NMR (300 MHz, $CDCl_3$) (minor diastereomer) δ 7.26 (d, 2 H, J = 8.4 Hz), 6.85 (d, 2 H, J, = 8.4 Hz), 4.47 (dt, 1 H, *J* = 9.9, 7.0 Hz), 3.92 - 3.81 (m, 2 H), 3.78 (s, 3H), 3.69 (s, 3 H), 3.46 (d, 1 H, J = 10.2 Hz), 1.88 - 1.79 (m, 2 H), 1.69 (m, 1 H), 1.43 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 173.3, 159.1, 129.4, 128.0, 114.1, 80.6 , 68.5, 56.6, 55.2, 29.5, 25.4 ;

Methyl α-(4-chloro-phenyl)-2-tetrahydrofuran acetate (11c, 12c). A solution of methyl 4-chloro-phenyldiazoacetate (215 mg, 1.02 mmol), in 15 ml dry THF was added dropwise over 60 min. to a refluxing solution of $Rh_2(DOSP)_4$ (19 mg, 0.01 mmol) in 5 ml dry THF. The solvent was removed *in*

vacuo, and flash chromatography with 15 % Et_2O / pet ether gave separation of the two diastereomers in a total recovery of 173 mg (48%) (57% at RT) colorless oil (1.7 / 1 de, 47% ee major ds (Chiralcel OD, FR 1.0 ml / min., 1.0% i-propanol / hexane, R_t = 10.4 and 11.2 min, UV 254nm), 51% ee minor ds (Eu tris [3-(heptafluoropropylhydroxymethylene) - (+) -camphorate]), (1.9 / 1 de, 76%ee major ds, 72% ee minor ds at RT);.$[\alpha]^{25}_D$ = -57.0° (c 0.228, $CDCl_3$, major isomer), $[\alpha]^{25}_D$ = -23.2° (c 0.284, $CDCl_3$, minor isomer); ^1H NMR (300 MHz, $CDCl_3$) (major diastereomer) δ 7.30 (s, 4 H), 4.42 (dt, 1 H, *J* = 7.7, 7.3 Hz), 3.82 - 3.69 (m, 2 H), 3.67 (s, 3 H), 3.58 (d, 1 H, *J* = 8.4 Hz), 2.10 (m, 1 H), 1.86 (m, 2 H), 1.63 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.2, 135.1, 133.4, 130.0 (CH), 128.7 (CH), 79.8 (CH), 68.3 (CH_2), 56.1 (CH), 52.1 (CH_3), 30.1 (CH_2), 25.5 (CH_2); 1521, 1349; IR (neat): 3029, 2948, 2869, 1734, 1592, 1496; ^1H NMR (300 MHz, $CDCl_3$) (minor diastereomer) δ 7.29 (s, 4 H), 4.47 (dt, 1 H, *J* = 9.5, 7.0 Hz), 3.94 - 3.81 (m, 2 H), 3.69 (s, 3 H), 3.49 (d, 1 H, J = 9.9 Hz), 1.87 - 1.79 (m, 2 H), 1.70 (m, 1 H), 1.41 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.7, 134.3, 133.7, 129.7 (CH), 128.9 (CH), 80.4 (CH) , 68.5 (CH_2), 56.7 (CH), 52.2 (CH_3), 29.5 (CH_2), 25.4 (CH_2);

Methyl α-(4-nitro-phenyl)-2-tetrahydrofuran acetate (11d, 12d). A solution of methyl 4-nitro-phenyldiazoacetate (221.16 mg, 1.00 mmol), in 15 ml dry THF was added dropwise over 90 min. to a refluxing solution of $Rh_2(DOSP)_4$ (20 mg, 0.01 mmol) in 5 ml dry THF. The solvent was removed *in vacuo*, and flash chromatography with 30 % Et_2O / pet ether gave separation of the two diastereomers in a total recovery of 134 mg (50%) pale yellow oil (1.8 / 1 de, 69% ee major ds (Chiralcel OD, FR 1.0 ml / min., 2.0% i-propanol / hexane, R_t = 19.5 and 21.6 min, UV 254nm), 58% ee minor ds (Eu tris [3-(heptafluoropropylhydroxymethylene) - (+) -camphorate])); $[\alpha]^{25}_D$ = -60.7° (c 0.112, $CDCl_3$, major isomer), $[\alpha]^{25}_D$ = -21.1° (c 0.284, $CDCl_3$, minor isomer); ^1H NMR (300 MHz, $CDCl_3$) (major diastereomer) δ 8.18 (d, 2 H, *J* = 8.8 Hz), 7.55 (d, 2 H, J = 8.8 Hz) 4.45 (dt, 1 H, *J* = 8.0, 7.3 Hz), 3.81 - 3.65 (m, 2 H), 3.69 (s, 3 H), 3.71 (d, 1 H, *J* = 8.4 Hz), 2.13 (m, 1 H), 1.85 (m, 2 H), 1.63 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.2, 147.2, 143.9, 129.7 (CH), 123.5 (CH), 79.6 (CH), 68.3 (CH_2), 56.5 (CH), 52.3 (CH_3), 30.2 (CH_2), 25.4 (CH_2); IR (neat): 3078, 2953, 23872, 1735, 1606; ^1H NMR (300 MHz, $CDCl_3$) (minor diastereomer) δ 8.17 (d, 2 H, J = 8.8 Hz), 7.54 (d, 2 H, J = 8.8 Hz) 4.47 (dt, 1 H, *J* = 9.2, 7.3 Hz), 3.91 - 3.78 (m, 2 H), 3.71 (s, 3 H), 3.66 (d, 1 H, J = 9.5 Hz), 1.84 (m, 2 H), 1.72 (m, 1 H), 1.41 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.8, 147.5, 143.0, 129.4 (CH), 123.8 (CH), 80.3 (CH) , 68.5 (CH_2), 57.0 (CH), 52.4 (CH_3), 29.4 (CH_2), 25.4 (CH_2).