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Synthesis of Highly Functionalized Chiral Cyclopentanes by Catalytic Enantio- and Diastereoselective Double Michael Addition Reactions

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General Information: Commercial reagents were used as received, unless otherwise stated. Merck 60 silica gel was used for chromatography, and Whatman silica gel plates with fluorescence F_{254} were used for thin-layer chromatography (TLC) analysis. ^{1}H and ^{13}C NMR spectra were recorded on Bruker Avance 500, and tetramethylsilane (TMS) was used as a reference. Data for ^{1}H are reported as follows: chemical shift (ppm), and multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet). Data for ^{13}C NMR are reported as ppm.

Procedure for preparation of (E) 4-Methoxycarbonyl-pent-2-enedioic acid 1-ethyl ester 5-methyl ester $(2a)^1$

LDA (12 mmol) in 80 mL THF was cooled to -78 °C and dimethyl malonate (10 mmol) in 10 mL of THF was introduced via syringe. The reaction temperature was raised to 0 °C for 30 min, then cooled to -78 °C again. A solution of methyl 4-bromocrotonate (11 mmol) in 10 mL THF was added. The reaction temperature was raised to RT and kept there overnight with stirring. After the reaction was quenched with saturated ammonium chloride solution, the crude product was extracted into methylene chloride. The organic phase was washed with brine, dried over MgSO₄. The unpurified product was purified by silica gel column, eluted with hexane/EtOAc = 10:1. 42% yield; 1 H NMR (500 MHz, CDCl₃): 6.75 (m, 1H), 5.76 (d, 1H; J = 16.5 Hz), 4.05 (q, 2H; J = 7.0 Hz), 3.63 (s, 6H), 3.43 (t, 1H; J = 7.5 Hz), 2.68 (t, 2H; J = 7.0 Hz), 1.16 (t, 3H; J = 7.5 Hz).

Compounds **2b**, **2c**, **2d** were prepared in a similar manner.

(*E*) 5-Ethoxycarbonyl-hex-2-enedioic acid diethyl ester (2b). 40% yield; ¹H NMR (500 MHz, CDCl₃): 6.89 (t, 1H; J = 7.0 Hz), 5.90 (d, 1H; J = 15.5 Hz), 4.17-4.22 (m, 6H), 3.51 (t, 1H; J = 6.5 Hz), 2.78-2.80 (m, 2H), 1.26-1.29 (m, 9H); ¹³C NMR (125 MHz, CDCl₃): δ 167.9, 165.5, 143.5, 123.5, 61.3, 59.9, 50.2, 30.7, 13.8, 13.7.

(*E*) 5-Isopropoxycarbonyl-hex-2-enedioic acid 1-ethyl ester 6-isopropyl ester (2c). 35% yield; 1 H NMR (500 MHz, CDCl₃): 6.89 (t, 1H; J = 8.0 Hz), 5.90 (d, 1H; J = 15.5 Hz), 5.04-5.09 (m, 2H), 4.18 (q, 2H; J = 7.0 Hz), 3.42 (t, 1H; J = 7.5 Hz), 2.77 (t, 2H; J = 7.5 Hz), 1.24-1.29 (m, 15H); 13 C NMR (125 MHz, CDCl₃): δ 167.8, 165.9, 143.9, 123.6, 69.1, 60.2, 50.8, 30.9, 21.5, 21.4, 14.1.

(*E*) 5-Benxyloxycarbonyl-hex-2-enedioic acid 6-benzyl ester 1-ethyl ester (2d). 43% yield; ¹H NMR (500 MHz, CDCl₃): 7.81-7.87 (m, 10H), 7.43 (t, 1H; J = 8.0 Hz), 6.42 (d, 1H; J = 15.5 Hz), 5.67-5.70 (m, 4H), 4.71 (q, 2H; J = 5.0 Hz), 4.16-4.18 (m, 1H), 3.37 (m, 2H), 1.82 (m, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 167.8, 165.6, 143.2,134.9, 128.4, 128.2, 128.0, 123.8, 67.2, 60.1, 50.4, 30.8, 14.0.

General procedure for double Michael addition reactions:

A mixture of **1** (0.10 mmol), **2** (0.12 mmol) and the catalyst **IV** (0.01 mmol) in EtOH (0.2 mL) was stirred at RT. The unpurified product was purified by column chromatography on silica gel, eluted by hexanes/EtOAc= 10:1 to 3:1 to give the desired product **3**.

4-Ethoxycarbonylmethyl-3-formyl-2-(4-methoxy-phenyl)-cyclopentane-1,1-dicarboxylic acid dimethyl ester (3a) (Table 2, entry 1). The title compound was prepared according to the general procedure, as described above in 92% yield. ¹H NMR (500 MHz, CDCl₃): 9.54 (d, 1H; J = 3.5 Hz), 7.18 (d, 2H; J = 8.5 Hz), 6.80 (d, 2H; J = 8.5 Hz), 4.31 (d, 1H; J = 10.5 Hz), 4.13 (q, 2H; J = 7.0 Hz), 3.77 (s, 3H), 3.76 (s, 3H), 3.21 (s, 3H), 2.96 (m, 1H), 2.51-2.69 (m, 4H), 1.26 (t, 3H; J = 7.0 Hz); ¹³C NMR (125 MHz, CDCl₃): δ 201.3, 172.1, 171.8, 170.3, 159.0, 129.7, 129.1, 113.6, 64.7, 61.6, 60.7, 55.2, 52.9, 52.2, 51.2, 39.8, 37.9, 35.0, 14.1; HPLC (Chiralcel OD-H, iPrOH/hexane = 20/80, flow rate = 0.5 mL/min, λ = 254 nm): t_{minor} = not observed, t_{major} = 23.38 min, ee = 99%, dr = 10:1; $[\alpha]_D^{23}$ = -19.4 (c = 1.0 in CHCl₃).

4-Ethoxycarbonylmethyl-3-formyl-2-(4-methoxy-phenyl)-cyclopentane-1,1-dicarboxylic acid diethyl ester (3b) (Table 2, entry 2). The title compound was prepared according to the general procedure, as described above in 90% yield. 1 H NMR (500 MHz, CDCl₃): 9.55 (d, 1H; J = 3.5 Hz), 7.19 (d, 2H; J = 8.5 Hz), 6.79 (d, 2H; J = 8.5 Hz), 4.11-4.34 (m, 5H), 3.79-3.82 (m, 1H), 3.76 (s, 3H), 3.48-3.52 (m, 1H), 2.93 (m, 1H), 2.51-2.68 (m, 4H), 1.23-1.27 (m, 6H), 0.83 (t, 3H; J = 7.0 Hz); 13 C NMR (125 MHz, CDCl₃): δ 201.4, 171.8, 171.6, 169.8, 159.0, 129.9, 129.3, 113.6, 64.7, 62.0, 61.8, 61.3, 60.7, 55.2, 50.9, 40.0, 38.0, 35.0, 14.1, 14.0, 13.4; HPLC (Chiralcel OD-H, iPrOH/hexane = 20/80, flow rate = 0.5 mL/min, λ = 254 nm): t_{minor} = not observed, t_{major} = 17.76 min, ee = 99%, dr = 11:1; $[\alpha]_D^{23}$ = -39.4 (c = 1.0 in CHCl₃).

4-Ethoxycarbonylmethyl-3-formyl-2-(4-methoxy-phenyl)-cyclopentane-1,1-dicarboxylic acid diisopropyl ester (3c) (Table 2, entry 3). The title compound was prepared according to the general procedure, as described above in 87% yield. ¹H NMR (500 MHz, CDCl₃): 9.55 (d, 1H; J = 3.5 Hz), 7.21 (d, 2H; J = 8.5 Hz), 6.79 (d, 2H; J = 8.5 Hz), 5.06-5.10 (m, 1H), 4.50-4.53 (m, 1H), 4.30-4.33 (m, 1H), 4.11-4.15 (m, 2H), 3.76 (s, 3H), 2.88-2.90 (m, 1H), 2.45-2.63 (m, 4H), 1.21-1.27 (m, 9H), 0.99-1.03 (m, 3H), 0.52-0.58 (m, 3H); ¹³C NMR (125 MHz, CDCl₃): δ 201.5, 171.8, 171.3, 169.3, 159.0, 130.1, 113.6, 77.3, 77.0, 76.8, 69.3, 69.1, 64.6, 62.5, 60.6, 55.2, 50.7, 40.3, 38.0, 34.9, 21.6, 21.4, 21.3, 20.7, 14.1; HPLC (Chiralcel OD-H, *i*PrOH/hexane = 20/80, flow rate = 0.5 mL/min, λ = 254 nm): t_{minor} = not observed, t_{major} = 14.05 min, ee = 99%, dr = 11:1; [α]_D²³ = -21.7 (c = 1.0 in CHCl₃).

4-Ethoxycarbonylmethyl-3-formyl-2-(4-methoxy-phenyl)-cyclopentane-1,1-dicarboxylic acid dibenzyl ester (3d) (Table 2, entry 4). The title compound was prepared according to the general procedure, as described above in 90% yield. ¹H NMR (500 MHz, CDCl₃): 9.53 (d, 1H; J = 3.5 Hz), 7.17-7.28 (m, 10H), 6.85 (d, 2H; J = 7.0 Hz), 6.73 (d, 2H; J = 8.5 Hz), 5.11 (d, 2H; J = 3.5 Hz), 4.74 (d, 1H; J = 3.5 Hz)

= 12.0 Hz), 4.35 (d, 2H; J = 11.5 Hz), 4.11 (q, 2H; J = 7.0 Hz), 3.72 (s, 3H), 2.97-2.98 (m, 1H), 2.53-2.69 (m, 4H), 1.24 (t, 3H; J = 7.5 Hz); ¹³C NMR (125 MHz, CDCl₃): δ 201.2, 171.8, 171.3, 169.7, 159.0, 135.0, 134.6, 129.8, 129.1, 128.5, 128.3, 128.2, 128.1, 128.0, 127.9, 113.7, 67.6, 67.2, 64.8, 61.7, 60.7, 55.1, 51.2, 40.0, 38.0, 35.0, 14.1; HPLC (Chiralcel OD-H, *i*PrOH/hexane = 20/80, flow rate = 0.5 mL/min, $\lambda = 254$ nm): $t_{minor} = not$ observed, $t_{major} = 35.01$ min, ee = 99%, dr > 20:1; $[\alpha]_D^{23} = -17.1$ (c = 1.0 in CHCl₃).

4-Ethoxycarbonylmethyl-3-formyl-2-(2-methoxy-phenyl)-cyclopentane-1,1-dicarboxylic acid dimethyl ester (3e) (Table 2, entry 5). The title compound was prepared according to the general procedure, as described above in 91% yield. ¹H NMR (500 MHz, CDCl₃): 9.67 (d, 1H; J = 3.0 Hz), 7.16-7.24 (m, 2H), 6.85-6.93 (m, 2H), 4.79 (d, 1H; J = 9.0 Hz), 4.16 (m, 2H), 3.83 (s, 3H), 3.81 (s, 3H), 3.22 (s, 3H), 3.02 (m, 1H), 2.51-2.74 (m, 4H), 1.30 (t, 3H; J = 7.0 Hz); ¹³C NMR (125 MHz, CDCl₃): δ 201.7, 172.3, 171.8, 169.6, 157.3, 130.2, 128.7, 127.2, 120.5, 110.6, 64.5, 62.2, 60.6, 55.2, 53.1, 51.9, 46.4, 40.6, 38.0, 35.2, 14.1; HPLC (Chiralpak AS-H, iPrOH/hexane = 20/80, flow rate = 0.5 mL/min, λ = 210 nm): t_{minor} = not observed, t_{major} = 43.72 min, ee = 99%, dr = 19:1; $[\alpha]_D^{23}$ = -22.5 (c = 1.0 in CHCl₃).

4-Ethoxycarbonylmethyl-3-formyl-2-phenyl-cyclopentane-1,1-dicarboxylic acid dimethyl ester (3f) (Table 2, entry 6). The title compound was prepared according to the general procedure, as described above in 92% yield. ¹H NMR (500 MHz, CDCl₃): 9.56 (d, 1H; J = 3.0 Hz), 7.26-7.28 (m, 5H), 4.37 (d, 1H; J = 10.5 Hz), 4.13 (q, 2H; J = 7.0 Hz), 3.76 (s, 3H), 3.14 (s, 3H), 3.00 (m, 1H), 2.53-2.69 (m, 4H), 1.26 (t, 3H; J = 7.0 Hz); ¹³C NMR (125 MHz, CDCl₃): δ 201.1, 171.9, 171.7, 170.1, 137.4, 128.6, 128.3, 127.7, 64.8, 61.5, 60.6, 53.0, 52.1, 51.8, 39.9, 37.9, 35.1, 14.1; HPLC (Chiralpak AS-H, iPrOH/hexane = 20/80, flow rate = 0.5 mL/min, λ = 254 nm): t_{minor} = 22.78 min, t_{major} = 24.93 min, ee = 84%, dr = 16:1; $[\alpha]_D^{23}$ = -22.8 (c = 1.0 in CHCl₃).

EtOOC
$$4-NO_2-C_6H_2$$
 H_3COOC $COOCH_3$

4-Ethoxycarbonylmethyl-3-formyl-2-(4-nitro-phenyl)-cyclopentane-1,1-dicarboxylic acid dimethyl ester (3g) (Table 2, entry 7). The title compound was prepared according to the general procedure, as described above in 90% yield. 1 H NMR (500 MHz, CDCl₃): 9.53 (s, 1H), 8.15 (d, 2H; J = 7.5 Hz), 7.48 (d, 2H; J = 7.5 Hz), 4.46 (d, 1H; J = 11.0 Hz), 4.14 (q, 2H; J = 7.0 Hz), 3.78 (s, 3H), 3.22 (s, 3H), 3.06 (m, 1H), 2.57-2.75 (m, 4H), 1.27 (t, 3H; J = 7.0 Hz); 13 C NMR (125 MHz, CDCl₃): δ 200.0, 171.6, 171.5, 169.9, 147.3, 144.8, 129.7, 123.3, 64.6, 61.1, 60.8, 53.2, 52.4, 51.3, 39.9, 37.7, 35.4, 14.1; HPLC (Chiralpak AS-H, iPrOH/hexane = 30/70, flow rate = 0.5 mL/min, λ = 254 nm): t_{minor} = 37.15 min, t_{major} = 50.00 min, ee = 98%, dr = 17:1; $[\alpha]_D^{23}$ = -8.1 (c = 1.0 in CHCl₃).

2-(4-Cyano-phenyl)-4-ethoxycarbonylmethyl-3-formyl-cyclopentane-1,1-dicarboxylic acid dimethyl ester (3h) (Table 2, entry 8). The title compound was prepared according to the general procedure, as

described above in 87% yield. 1 H NMR (500 MHz, CDCl₃): 9.54 (d, 1H; J = 3.0 Hz), 7.58 (d, 2H; J = 8.0 Hz), 7.41 (d, 2H; J = 8.5 Hz), 4.40 (d, 1H; J = 11.0 Hz), 4.14 (q, 2H; J = 7.0 Hz), 3.77 (s, 3H), 3.20 (s, 3H), 3.00-3.04 (m, 1H), 2.56-2.75 (m, 4H), 1.27 (t, 3H; J = 7.0 Hz); 13 C NMR (125 MHz, CDCl₃): δ 200.2, 171.6, 169.9, 142.8, 132.0, 129.6, 118.4, 111.6, 64.6, 61.0, 60.8, 53.2, 52.4, 51.5, 39.9, 37.7, 35.4, 14.1; HPLC (Chiralpak AS-H, iPrOH/hexane = 30/70, flow rate = 0.5 mL/min, λ = 210 nm): t_{minor} = not observed, t_{major} = 48.89 min, ee = 99%, dr = 9:1; $[\alpha]_{D}^{23}$ = -12.4 (c = 1.0 in CHCl₃).

4-Ethoxycarbonylmethyl-2-(4-fluoro-phenyl)-3-formyl-cyclopentane-1,1-dicarboxylic acid dimethyl ester (**3i**) (Table 2, entry 9). The title compound was prepared according to the general procedure, as described above in 95% yield. ¹H NMR (500 MHz, CDCl₃): 9.54 (d, 1H; J = 3.5 Hz), 7.24-7.27 (m, 2H), 6.95-6.99 (m, 2H), 4.35 (d, 1H; J = 11.0 Hz), 4.14 (q, 2H; J = 7.0 Hz), 3.76 (s, 3H), 3.21 (s, 3H), 2.96 (m, 1H), 2.53-2.69 (m, 4H), 1.26 (t, 3H; J = 7.0 Hz); ¹³C NMR (125 MHz, CDCl₃): δ 200.9, 171.9, 171.7, 170.1, 132.9, 130.4, 130.3, 115.2, 115.1, 64.6, 61.5, 60.7, 53.0, 52.2, 51.1, 39.8, 37.8, 35.1, 14.1; HPLC (Chiralcel OD-H, iPrOH/hexane = 8/92, flow rate = 0.5 mL/min, λ = 254 nm): t_{minor} = 18.99 min, t_{major} = 22.00 min, ee = 97%, dr = 15:1; $[\alpha]_D^{23}$ = -29.4 (c = 1.0 in CHCl₃).

2-(2-Chloro-phenyl)-4-ethoxycarbonylmethyl-3-formyl-cyclopentane-1,1-dicarboxylic acid dimethyl ester (3j) (Table 2, entry 10). The title compound was prepared according to the general procedure, as described above in 93% yield. ¹H NMR (500 MHz, CDCl₃): 9.72 (s, 1H), 7.21-7.42 (m, 4H), 5.22 (d, 1H; J = 9.0 Hz), 4.18 (m, 2H), 3.84 (s, 3H), 3.24 (s, 3H), 2.87 (m, 1H), 2.58-2.73 (m, 4H), 1.31 (t, 3H; J = 7.0 Hz); ¹³C NMR (125 MHz, CDCl₃): δ 200.4, 171.6, 171.5, 169.3, 137.2, 135.0, 129.6, 129.2, 128.6, 126.9, 64.9, 64.4, 60.7, 53.3, 52.1, 46.3, 40.6, 37.5, 35.1, 14.1; HPLC (Chiralpak AS-H, *i*PrOH/hexane = 20/80, flow rate = 0.5 mL/min, λ = 210 nm): t_{minor} = not observed, t_{major} = 26.44 min, ee = 99%, dr > 20:1; $[\alpha]_D^{22} = -17.5$ (c = 1.0 in CHCl₃).

4-Ethoxycarbonylmethyl-3-formyl-2-naphthalen-1-yl-cyclopentane-1,1-dicarboxylic acid dimethyl ester (3k) (Table 2, entry 11). The title compound was prepared according to the general procedure, as described above in 86% yield. ¹H NMR (500 MHz, CDCl₃): 9.59 (d, 1H; J = 3.0 Hz), 7.39-7.78 (m, 7H), 4.53 (d, 1H; J = 10.5 Hz), 4.14 (q, 2H; J = 7.5 Hz), 3.77 (s, 3H), 3.14 (m, 1H), 3.04 (s, 3H), 2.55-2.75 (m, 4H), 1.31 (t, 3H; J = 7.0 Hz); ¹³C NMR (125 MHz, CDCl₃): δ 201.1, 172.0, 171.8, 170.3, 134.8, 133.1, 127.9, 127.7, 127.5, 126.5, 126.2, 126.1, 64.8, 61.5, 60.7, 53.0, 52.2, 52.0, 40.1, 38.0, 35.2, 14.2; HPLC (Chiralcel OD-H, iPrOH/hexane = 20/80, flow rate = 0.5 mL/min, λ = 210 nm): t_{minor} = 20.40 min, t_{major} = 25.39 min, ee = 98%, dr > 20:1; $[\alpha]_D^{23}$ = -19.3 (c = 1.0 in CHCl₃).

4-Ethoxycarbonylmethyl-3-formyl-2-propyl-cyclopentane-1,1-dicarboxylic acid dibenzyl ester (3l) (Table 2, entry 12). The title compound was prepared according to the general procedure, as described above in 85% yield. ¹H NMR (500 MHz, CDCl₃): 9.69 (d, 1H; *J* = 3.5 Hz), 7.37-7.44 (m, 10H), 5.15-5.28

(m, 4H), 4.21 (q, 2H; J = 7.0 Hz), 3.12 (m, 1H), 2.55-2.61 (m, 3H), 2.38-2.41 (m, 2H), 1.28-1.44 (m, 7H), 1.25 (t, 3H; J = 7.0 Hz); ¹³C NMR (125 MHz, CDCl₃): δ 202.1, 171.7, 171.3, 169.9, 135.2, 135.0, 128.5, 128.4, 128.2, 67.5, 67.3, 63.7, 62.3, 60.6, 46.2, 39.3, 38.5, 34.8, 33.8, 20.8, 14.1, 13.9; HPLC (Chiralcel OD-H, iPrOH/hexane = 10/90, flow rate = 0.5 mL/min, λ = 210 nm): t_{minor} = 18.25, t_{major} = 21.16 min, ee = 94%, dr = 9:1; $[\alpha]_D^{24}$ = -42.5 (c = 1.0 in CHCl₃).

The preparation of 3j derivative 4:

EtOOC COOCH₃

$$\mathbf{H}_{3}COOC$$

$$\mathbf{J}_{2}-CI-C_{6}H_{4}$$

$$\mathbf{H}_{2}O_{2}$$

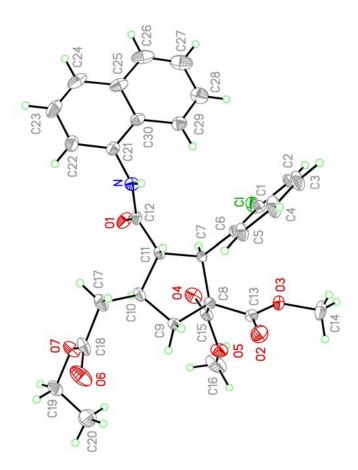
$$\mathbf{J}_{3}$$

$$\mathbf{J}$$

2-(2-Chloro-phenyl)-4-ethoxycarbonylmethyl-cyclopentane-1,1,3-tricarboxylic acid dimethyl ester (**4a**). The title compound was prepared according to the procedure in literature^[2] in 90% yield. ¹H NMR (500 MHz, CDCl₃): 7.14-7.35 (m, 4H), 5.18 (d, 1H; J = 10.0 Hz), 4.11 (m, 2H), 3.74 (s, 3H), 3.19 (s, 3H), 3.01 (m, 1H), 2.79 (dd, 1H; J = 4.5 Hz), 2.51-2.67 (m, 4H), 1.31 (t, 3H; J = 7.0 Hz); ¹³C NMR (125 MHz, CDCl₃): δ 178.1, 171.7, 171.5, 169.8, 137.1, 135.5, 129.6, 128.8, 128.4, 126.6, 64.2, 60.6, 56.0, 53.1, 52.0, 48.3, 40.5, 37.5, 37.4, 14.0.

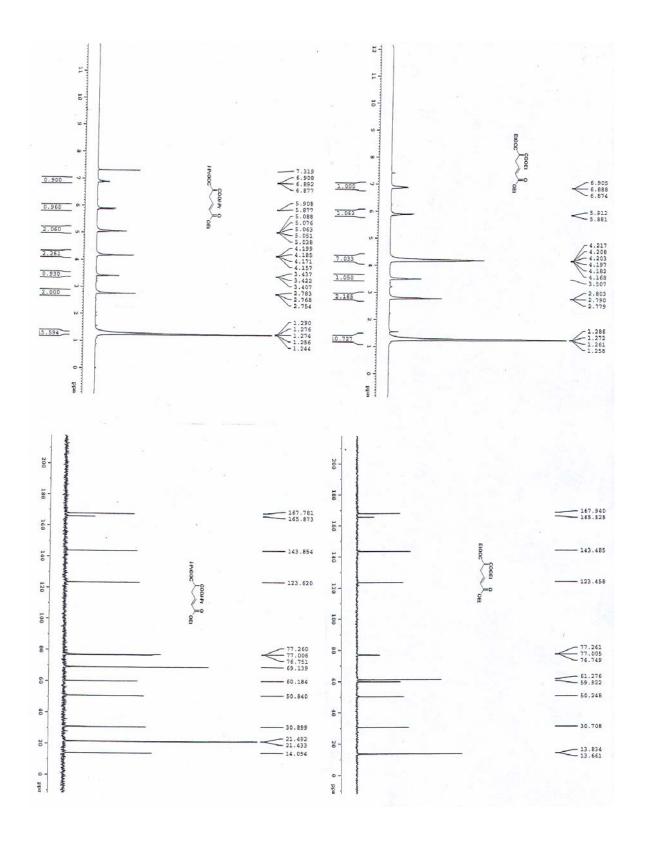
2-(2-Chloro-phenyl)-4-ethoxycarbonylmethyl-3-(naphthalene-1-ylcarbamoyl)-cyclopentane-1,1-dicarboxylic acid dimethyl ester (4). The title compound was prepared by using DCC/DMAP as the coupling reagent in 10% yield. 1 H NMR (500 MHz, CDCl₃): 8.24 (s, 1H), 7.90 (d, 1H; J = 6.5 Hz), 7.81 (d, 1H; J = 7.0 Hz), 7.61-7.81 (m, 2H), 7.20-7.44 (m, 7H), 5.32 (m, 1H), 4.19 (m, 2H), 3.82 (s, 3H), 3.23 (s, 3H), 2.81-2.87 (m, 2H), 2.64-2.72 (m, 3H), 1.28 (t, 3H; J = 7.0 Hz).

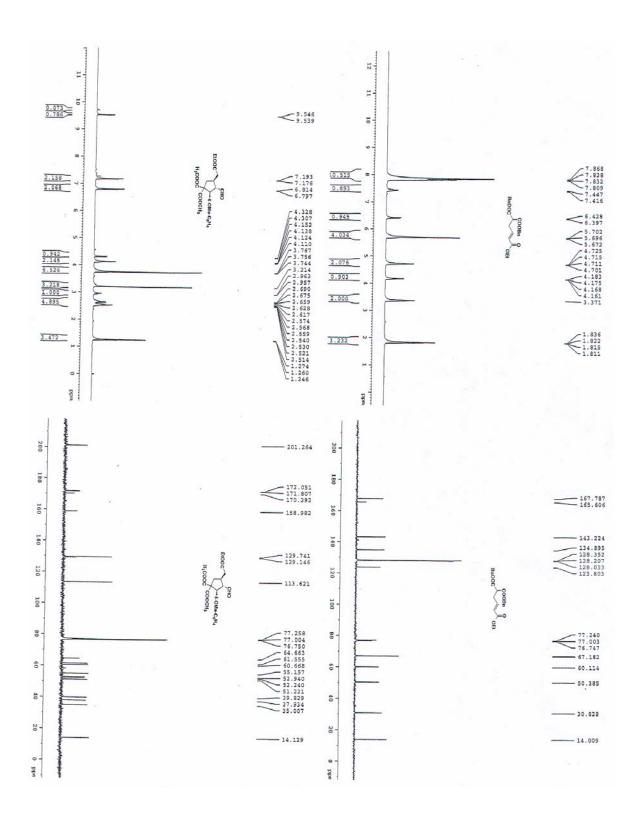
X-Ray Structure of compound 4

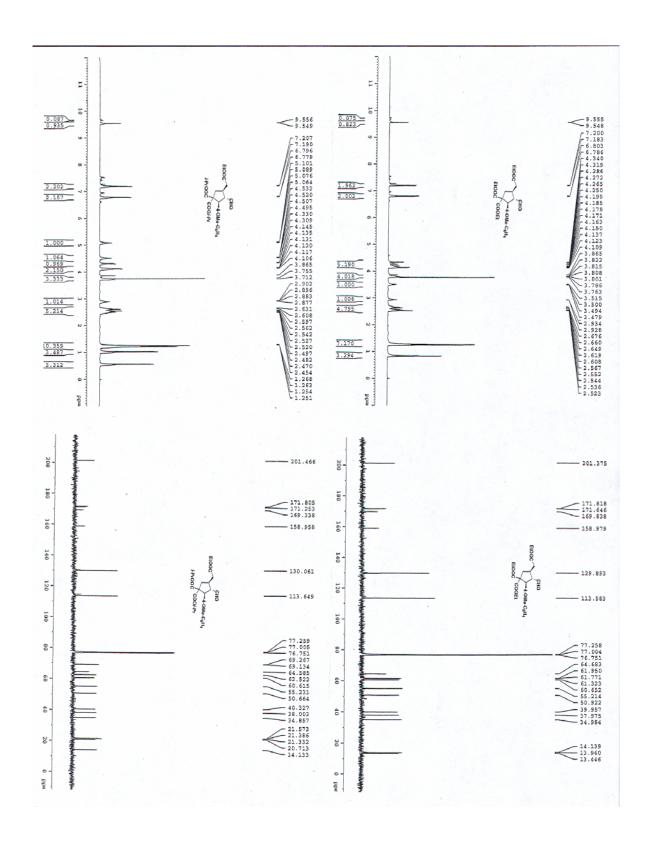


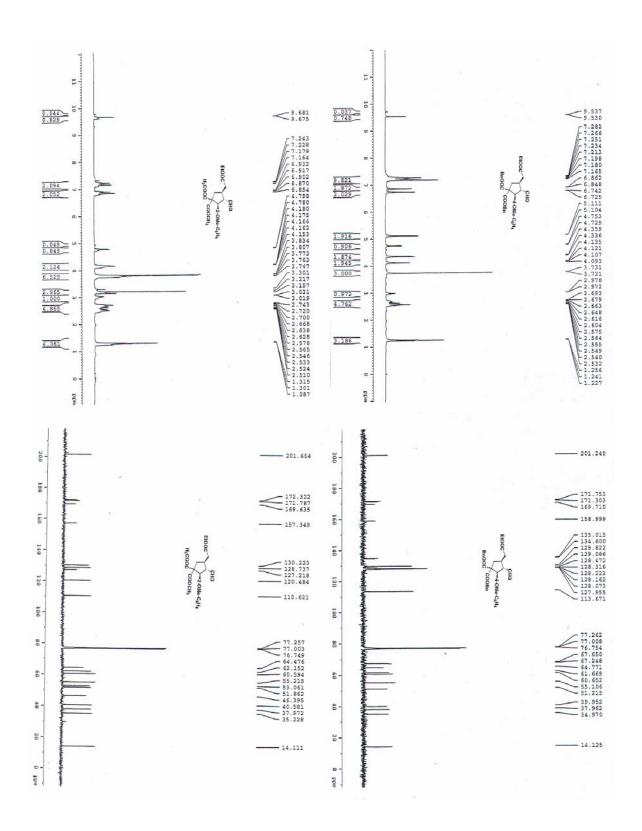
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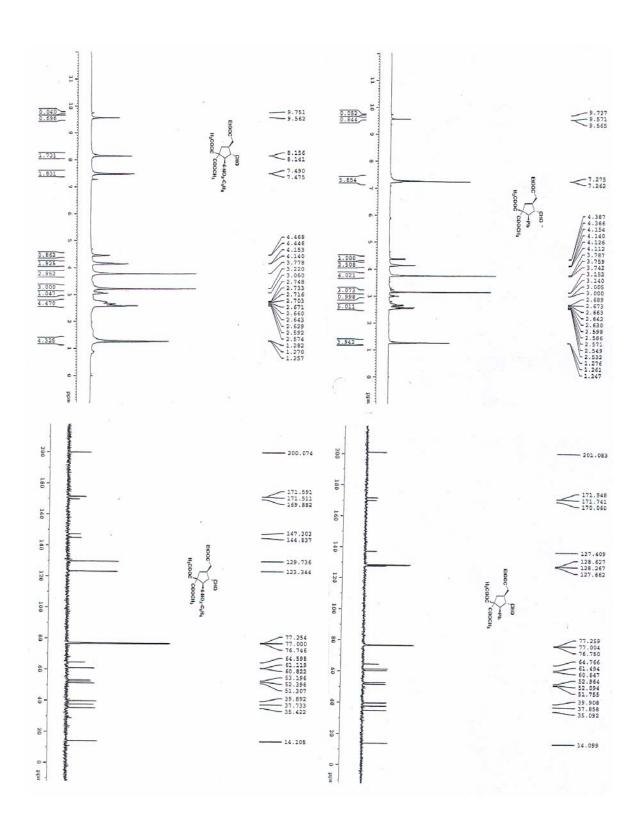
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- [2] R. Beumer, C. Bubert, C. Cabrele, O. Vielhauer, M. Pietzsch, O. Reiser, *J. Org. Chem.* **2000**, *65*, 8960.

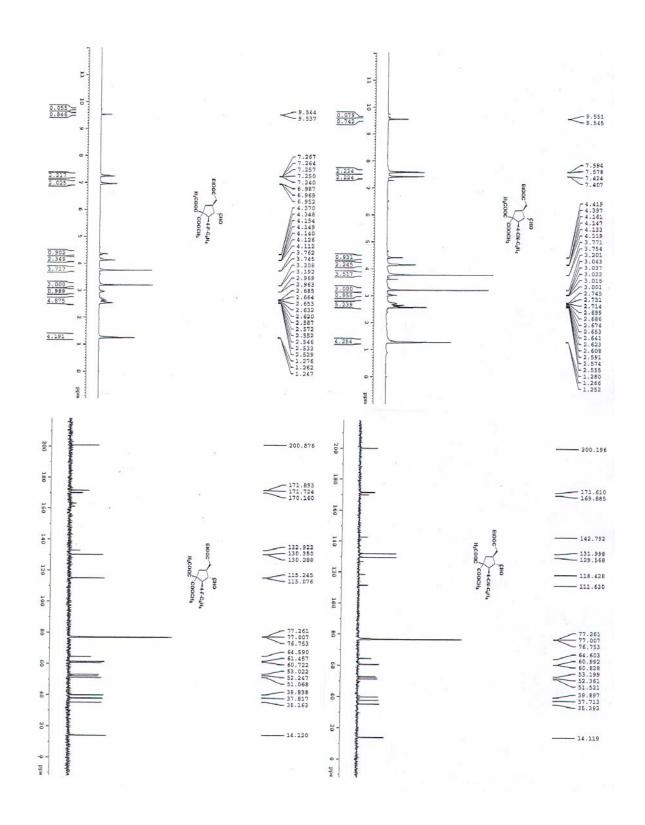


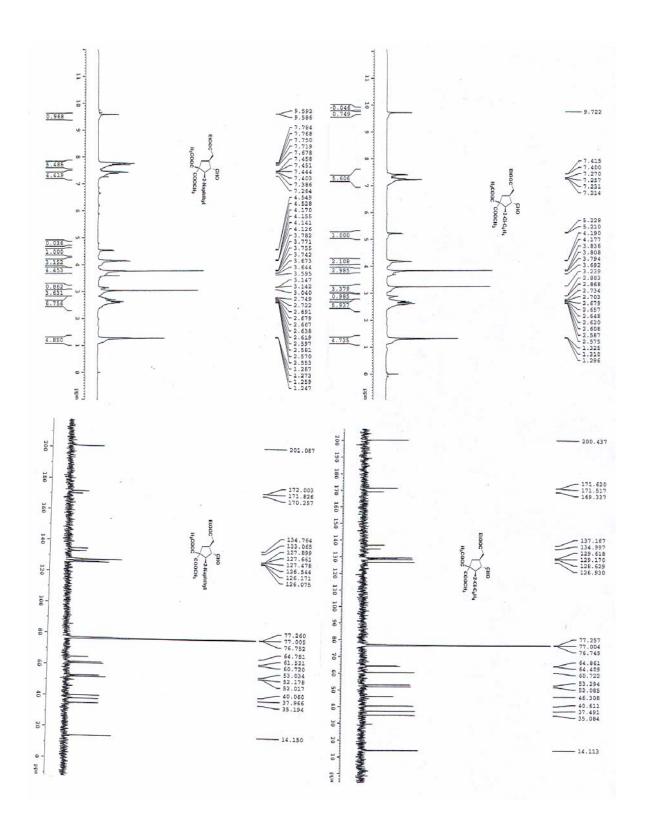


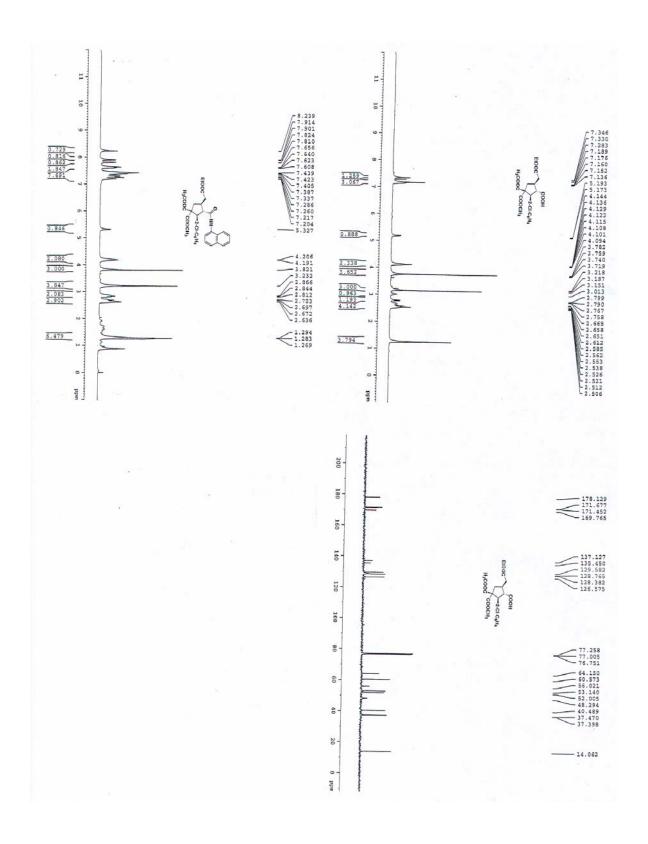


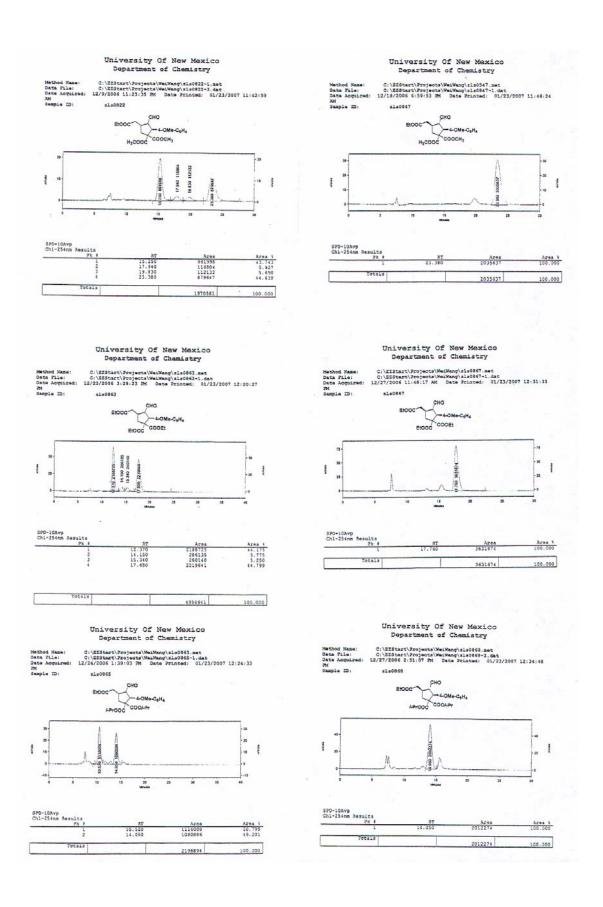








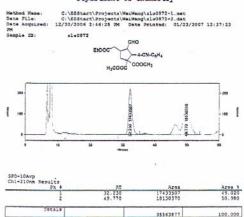


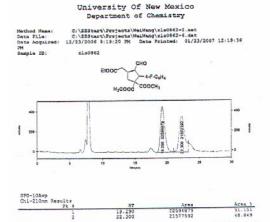


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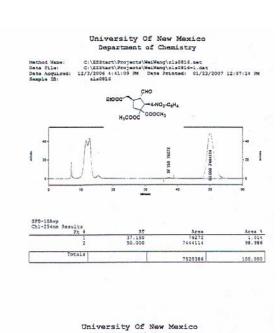
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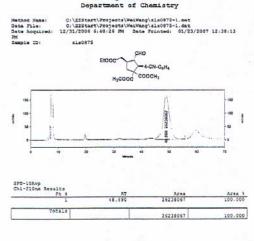
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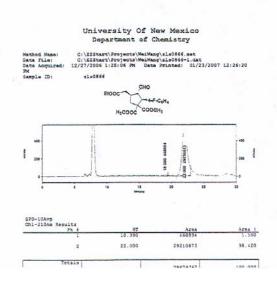




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