**Direct Atom-efficient Esterification between Carboxylic Acids and Alcohols Catalyzed by
Amphoteric, Water-tolerant TiO(acac)₂**

Chien-Tien Chen* and Yogesh S. Munot

Department of Chemistry, National Taiwan Normal University, Taipei, Taiwan 11650

SUPPORTING INFORMATION

Table of Content

General experimental methods-----	S3
Data for end compounds 1a-f and 2-18 -----	S3-S10
¹ H and ¹³ C NMR spectra of benzyl benzoate (1a)-----	S11
¹ H and ¹³ C NMR spectra of 2-phenylethyl benzoate (1b)-----	S12
¹ H and ¹³ C NMR spectra of 1,3-propyl dibenzoate (1c)-----	S13
¹ H and ¹³ C NMR spectra of 1-phenylethyl benzoate (1d)-----	S14
¹ H and ¹³ C NMR spectra of 4-methoxyphenyl benzoate (1e)-----	S15
¹ H and ¹³ C NMR spectra of 2-naphthyl benzoate (1f)-----	S16
¹ H and ¹³ C NMR spectra of benzyl thiophene-2-carboxylate (2)-----	S17
¹ H and ¹³ C NMR spectra of benzyl ferrocene-carboxylate (3)-----	S18
¹ H and ¹³ C NMR spectra of benzyl <i>trans</i> -3-phenylpropenoate (4)-----	S19
¹ H and ¹³ C NMR spectra of benzyl 3-methyl-2-butenolate (5)-----	S20
¹ H and ¹³ C NMR spectra of benzyl 2-bromophenylacetate (6)-----	S21
¹ H and ¹³ C NMR spectra of 2,2-dimethyl-[1,3]-dioxolan-4-ylmethyl benzoate (7)-----	S22
¹ H and ¹³ C NMR spectra of 4-nitro-phenyl 4-methoxy-benzoate (8)-----	S23
¹ H and ¹³ C NMR spectra of 4-(tetrahydro-pyran-2-yloxy)-butyl nicotinate (9)-----	S24
¹ H and ¹³ C NMR spectra of 4-(<i>tert</i> -butyl-dimethylsilanyloxy)-butyl 3-phenylpropanoate (10)-----	S25
¹ H and ¹³ C NMR spectra of diphenylmethyl 3-phenylpropanoate (11)-----	S26
¹ H and ¹³ C NMR spectra of 1-phenethyl-but-3-enyl 3-phenylpropanoate (12)-----	S27
¹ H and ¹³ C NMR spectra of 2'-hexanoyloxy-biphenyl-2-yl hexanoate (13)-----	S28
¹ H and ¹³ C NMR spectra of 2-cholesteryl oleate (14)-----	S29

Expanded ^{13}C NMR spectra of 2-cholesteryl oleate (14)-----	S30-S31
^1H and ^{13}C NMR spectra of benzyl 2-hydroxy-2-phenyl-acetate (15)-----	S32
^1H and ^{13}C NMR spectra of 4-methoxyphenyl 1-carboxyl-1,2,2-trimethyl-cyclopentane-3-carboxylate (16)-----	S33
^1H and ^{13}C NMR spectra of 4-methoxy-phenyl 7,7-dimethyl-2-oxo-bicyclo[2.2.1]-heptane-1-carboxylate (17)-----	S34
^1H and ^{13}C NMR spectra of polymer of adipic acid and diethylene glycol (18)-----	S35

General. ^1H NMR and ^{13}C NMR spectra were recorded on 400 MHz (100 MHz ^{13}C) spectrometers in deuteriochloroform with chloroform as an internal reference unless otherwise stated. Chemical shifts are reported in ppm (δ). Coupling constants, J , are reported in Hz. Infrared spectra were recorded on a FTIR spectrometer. Peaks are reported in units of cm^{-1} with the following relative intensities: br (broad), s (strong 67-100 %), m (medium 33-67 %), or w (weak 0-33 %). Mass spectra were recorded with an ionization voltage of 70 or 20 eV unless otherwise stated. Visualization was accomplished with UV light or with phosphomolybdic acid (PMA) and KMnO_4 staining agents. Column (flash) chromatography was performed using 32-63 μm silica gel. Solvents for extraction and chromatography were reagent grade. Xylene was dried over CaH_2 . All reactions were run under nitrogen and the direct condensation products were isolated as chromatographically pure materials. The VOX_2 series compounds (brand name as *Clip-all*[®] series, US patent # 6,541,659 B1, 2003) are now available from the institution (e-mail: chefv043@ntnu.edu.tw).

Benzyl benzoate (1a)¹

^1H NMR (400 MHz, CDCl_3) δ 8.12 (dd, $J = 8.3, 1.2$ Hz, 2H), 7.56 (t, $J = 7.3$ Hz, 1H), 7.50-7.37 (m, 7H), 5.40 (s, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.3, 136.0, 132.9, 130.1, 129.6, 128.5, 128.3, 128.2, 128.1, 66.6; MS (EI, 70 eV) 212 (M^+ , 15), 104 (100), 90 (50), 77 (100).

2-Phenylethyl benzoate (1b)²

^1H NMR (400 MHz, CDCl_3) δ 8.06 (dd, $J = 7.9, 1.4$ Hz, 2H), 7.55 (t, $J = 7.6$ Hz, 1H), 7.48-7.44 (m, 2H), 7.38-7.26 (m, 5H), 4.57 (t, $J = 7.0$ Hz, 2H), 3.12 (t, $J = 7.0$ Hz, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.4, 137.8, 132.8, 130.2, 129.5, 128.9, 128.5, 128.3, 126.5, 65.4, 35.2; MS (EI, 70 eV) 226 (M^+ , 5), 106 (60), 104 (100), 91 (35), 77 (50).

(1) (a) Steliou, K.; Szczygielska-Nowosielska, A.; Favre, A.; Poupart, M. A.; Hanessian, S. *J. Am. Chem. Soc.* **1980**, 102, 7578. (b) Rosenberg, M. G.; Brinker, U. H. *J. Org. Chem.* **2003**, 68, 4819.
 (2) (a) Fujisawa, T.; Mori, T.; Fukumoto, K.; Sato, T. *Chem. Lett.* **1982**, 1891. (b) Katritzky, A. R.; Rachwal, S.; Rachwal, B. *Synthesis* **1991**, 1, 69.

1,3-Propyl dibenzoate (1c)³

m.p. 58-59 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.04 (dd, *J* = 8.0, 1.3 Hz, 4H), 7.54 (t, *J* = 7.4 Hz, 2H), 7.42 (t, *J* = 7.8 Hz, 4H), 4.51 (t, *J* = 6.2 Hz, 4H), 2.27 (quin, *J* = 6.2 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 166.5, 133.0, 130.0, 129.6, 128.3, 61.7, 28.2; MS (EI, 70 eV) 284 (M⁺, 15), 227 (16), 122 (10), 105 (100), 77 (95), 51 (10).

1-Phenylethyl benzoate (1d)⁴

¹H NMR (400 MHz, CDCl₃) δ 8.13 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.50-7.42 (m, 4H), 7.41 (t, *J* = 7.2 Hz, 2H), 7.33 (t, *J* = 7.2 Hz, 1H), 6.19 (q, *J* = 6.6 Hz, 1H), 1.71 (d, *J* = 6.6 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 165.7, 141.7, 132.8, 130.5, 129.6, 128.5, 128.3, 127.8, 126.0, 72.8, 22.3; MS (EI, 70 eV) 226 (M⁺, 32), 117 (13), 105 (100), 82 (20), 77 (18).

4-Methoxyphenyl benzoate (1e)⁵

m.p. 87-88 °C, ¹H NMR (400 MHz, CDCl₃) δ 8.25 (dd, *J* = 8.2, 1.2 Hz, 2H), 7.64-7.62 (m, 1H), 7.51 (t, *J* = 7.5 Hz, 2H), 7.19 (dd, *J* = 6.8, 2.3 Hz, 2H), 6.97 (dd, *J* = 6.8, 2.3 Hz, 2H), 3.81 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 165.2, 157.1, 144.2, 133.2, 129.9, 129.4, 128.3, 122.2, 114.3, 55.2; MS (EI, 70 eV) 228 (M⁺, 20), 123 (20), 105 (100), 95 (19), 77 (59), 51 (20).

2-Naphthyl benzoate (1f)⁶

m.p. 107-108 °C; ¹H NMR (400 MHz, CDCl₃) δ 8.32-8.29 (m, 2H), 7.96-7.85 (m, 3H), 7.74 (d, *J* = 2.3 Hz, 1H), 7.70-7.66 (m, 1H), 7.58-7.50

(3) (a) Holcapek, M.; Virellzler, H.; Chamot-Rooke, J.; Jandera, P.; Moulin, C. *Anal. Chem.* **1999**, 71, 13, 2288. (b) Sharghi, H.; Sarvari, M. H. *Tetrahedron* **2003**, 59, 3627.

(4) (a) Nakamura, K.; Takenaka, K. *Tetrahedron: Asymmetry* **2002**, 13, 415. (b) Okamoto, Y.; Cao, Z.-K.; Aburatani, R.; Hatada, K. *Bull. Chem. Soc. Jpn.* **1987**, 60, 3999.

(5) (a) Lee, C. K.; Yu, J. S.; Lee, H.-J. *J. Heterocycl. Chem.* **2002**, 39, 1207. (b) Shelkov, R.; Nahmany, M.; Melman, A. *Org. Biomol. Chem.* **2004**, 2, 397. (c) Hashimoto, S.; Furukawa, I. *Bull. Chem. Soc. Jpn.* **1981**, 54, 2227.

(6) (a) Banu, H. S.; Pitchumani, K.; Srinivasan, C. *Tetrahedron* **1999**, 55, 9601. (b) Diago-Meseguer, J.; Palomo-Coll, A. L.; Fernandez-Lizarbe, J. R.; Zugaza-Bilbao, A. *Synthesis* **1980**, 7, 547. (c) Chakraborti, A. K.; Gulhane, R. *Tetrahedron Lett.* **2003**, 44, 6749.

(m, 4H), 7.41 (dd, $J = 8.8, 2.3$ Hz, 1H); ^{13}C NMR (100 MHz, CDCl_3) δ 165.3, 148.6, 133.8, 133.6, 131.5, 130.2, 129.5, 129.4, 128.6, 127.8, 127.6, 126.5, 125.7, 121.2, 118.7; MS (EI, 70 eV) 248 (M^+ , 20), 143 (10), 115 (100), 105 (60), 89 (15), 77 (37).

Benzyl thiophene-2-carboxylate (2)⁷

^1H NMR (400 MHz, CDCl_3) δ 7.86 (dd, $J = 3.8, 1.3$ Hz, 1H), 7.56 (dd, $J = 5.0, 1.3$ Hz, 1H), 7.47-7.44 (m, 2H), 7.43-7.34 (m, 3H), 7.10 (dd, $J = 5.0, 3.8$ Hz, 1H), 5.37 (s, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 161.9, 135.8, 133.5, 132.5, 128.5, 128.2, 128.1, 127.7, 66.6; MS (EI, 70 eV) 218 (M^+ , 98), 152 (15), 111 (100), 90 (43).

Benzyl ferrocene-carboxylate-3

IR (CCl_4) 3067 (w), 3034 (w), 2955 (w), 1717 (s), 1498 (m), 1459 (s), 1379 (s), 1272 (s), 1126 (m), 1107 (m), 1050 (w), 1025 (m), 1002 (m); ^1H NMR (500 MHz, CDCl_3) δ 7.46 (d, $J = 7.0$ Hz, 2H), 7.39 (t, $J = 7.5$ Hz, 2H), 7.34 (t, $J = 7.5$ Hz, 1H), 5.27 (s, 2H), 4.83 (t, $J = 1.8$ Hz, 2H), 4.31 (t, $J = 2.0$ Hz, 2H), 4.10 (s, 5H); ^{13}C NMR (125.5 MHz, CDCl_3) δ 171.5, 136.6, 128.5, 128.3, 128.2, 71.3, 71.0, 70.2, 69.7, 65.8; MS (70 eV) 320 (M^+ , 100), 226 (23), 129 (20), 91 (35); TLC R_f 0.45 (EtOAc/*n*-hexane, 2/98); HR-MS calcd for $\text{C}_{18}\text{H}_{16}\text{O}_2\text{Fe}$: 320.0495, found: 320.0494.

Benzyl *trans*-3-phenylpropenoate (4)⁸

m.p. 38-39 °C; ^1H NMR (400 MHz, CDCl_3) δ 7.81 (d, $J = 16.0$ Hz, 1H), 7.55-7.54 (m, 2H), 7.48-7.39 (m, 8H), 6.55 (d, $J = 16.0$ Hz, 1H), 5.32 (s, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 166.4, 144.9, 135.9, 134.1, 130.1, 128.6, 128.4, 128.04, 128.00, 127.9, 117.7, 66.1; MS (EI, 70 eV) 238 (M^+ , 13), 192 (43), 190 (23), 131 (61), 103 (40), 90 (100), 76 (63), 64 (17).

(7) (a) Khan, K. M.; Maharvi, G. M.; Hayat, S.; Ullah, Zia; Choudhary, M. I.; Rahmen, A. *Tetrahedron* **2003**, 59, 5549. (b) Grasa, G. A.; Gueveli, T.; Singh, R.; Nolan, S. P. *J. Org. Chem.* **2003**, 68, 2812.
 (8) (a) Parrish, J. P.; Dueno, E. E.; Kim, S.-I.; Jung, K. W. *Synth. Commun.* **2000**, 30, 2687. (b) Ishihara, K.; Nakayama, M.; Ohara, S.; Yamamoto, H. *Tetrahedron* **2002**, 58, 8179.

Benzyl 3-methyl-2-butenolate (5)⁹

¹H NMR (400 MHz, CDCl₃) δ 7.43-7.32 (m, 5H), 5.81 (s, 1H), 5.20 (s, 2H), 2.25 (d, *J* = 0.6 Hz, 3H), 1.92 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 166.0,

156.9, 136.3, 128.2, 127.9, 127.7, 115.6, 65.1, 27.1, 20.0, MS (EI, 70 eV) 190 (M⁺, 2), 189 (27), 181 (39), 165.1(43), 91 (100).

Benzyl 2-bromophenylacetate (6)¹⁰

m.p. 48-49 °C; IR (CCl₄) 3054 (w), 2956 (w), 1736 (s), 1570 (w), 1472 (m), 1377 (m), 1159 (s), 1029 (m), 977 (m); ¹H NMR (400 MHz, CDCl₃)

δ 7.59 (dd, *J* = 7.6, 0.8 Hz, 1H), 7.38-7.30 (m, 5H), 7.30-7.29 (m, 2H), 7.16-7.14 (m, 1H) 5.20 (s, 2H), 3.87 (s, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 170.3, 135.7, 134.1, 132.8, 131.5, 128.9, 128.5, 128.2, 128.1, 127.5, 125.0, 66.7, 41.6; MS (EI, 70 eV) 306 (M+2⁺, 2), 304 (M⁺, 3), 224 (7), 169 (23), 91 (100), 64 (14); TLC R_f 0.30 (EtOAc/*n*-hexane, 2/98).

2,2-Dimethyl-[1,3]-dioxolan-4-ylmethyl benzoate (7)¹¹

¹H NMR (400 MHz, CDCl₃) δ 7.99 (dd, *J* = 8.0, 1.5 Hz, 2H), 7.48 (t, *J* = 6.8 Hz, 1H), 7.38-7.34 (m, 2H), 4.40-4.36 (m, 1H), 4.35-4.26 (m, 2H), 4.08-4.04 (m, 1H), 3.82-3.78 (m, 1H), 1.39 (s, 3H), 1.32 (s, 3H); ¹³C NMR

(100 MHz, CDCl₃) δ 166.0, 132.9, 129.6, 129.4, 128.1, 109.5, 73.4, 66.1, 64.8, 26.5, 25.1; MS (EI, 70 eV) 236 (M⁺, 2), 221 (70), 105 (100), 100 (15), 86 (15), 77 (58), 51 (12).

4-Nitrophenyl 4-methoxybenzoate (8)¹²

(9) (a) Bryant, H. J.; Dardonville, C. Y.; Hodgkinson, T. J.; Hursthouse, M. B.; Malik, K. M. Abdul; Shipman, M. J. *Chem. Soc. Perkin Trans. 1*. **1998**, 7, 1249. (b) Ishihara, K.; Nakayama, M.; Ohara, S.; Yamamoto, H. *Tetrahedron* **2002**, 58, 8179.

(10) Tilley, J. W.; Danho, W.; Lovey, K.; Wagner, R.; Swistok, J. *J. Med. Chem.* **1991**, 34, 1125.

(11) (a) Shiao, M. J.; Yang, C. Y.; Lee, S. H.; Wu, T. C. *Synth. Commun.* **1988**, 18, 359. (b) Ramaiah, M. *J. Org. Chem.* **1985**, 50, 4991.

(12) (a) Ghosh, U.; Ganessunker, D.; Sattigeri, V. J.; Carlson, K. E.; Mortensen, D. J.; Katzenellenbogen, B. S.; Katzenellenbogen, J. A. *Bioorg. Med. Chem.* **2003**, 11, 629. (b) Stauffer, S. R.; Coletta, C. J.; Tedesco, R.; Nishiguchi, G.; Carlson, K.; Sun, J.; Katzenellenbogen, B. S.; Katzenellenbogen, J. A. *J. Med. Chem.* **2000**, 43, 4934.

m.p. 167-168 °C; ^1H NMR (400 MHz, CDCl_3) δ 8.31 (d, $J = 9.1$ Hz, 2H), 8.15 (d, $J = 8.9$ Hz, 2H), 7.40 (d, $J = 9.1$ Hz, 2H), 7.01 (d, $J = 8.9$ Hz, 2H), 3.91 (s, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 164.4, 155.9, 145.3, 132.5, 125.2, 122.7, 120.7, 114.1, 55.6; MS (EI, 70 eV) 273 (M^+ , 3), 219 (5), 135 (100), 91 (5), 77 (5).

4-(Tetrahydro-pyran-2-yloxy)-butyl Nicotinate-9

IR (CCl_4) 2945 (m), 1726 (s), 1590 (m), 1419 (m), 1384 (w), 1282 (s), 1123 (s), 1024 (s); ^1H NMR (400 MHz, CDCl_3) δ 9.22 (d, $J = 2.0$ Hz, 1H), 8.77 (dd, $J = 4.9, 1.5$ Hz, 1H), 8.33 (dt, $J = 7.9, 1.7$ Hz, 1H), 7.44 (dd, $J = 7.9, 4.9$ Hz, 1H), 4.57 (t, $J = 6.6$ Hz, 1H), 4.39 (t, $J = 6.5$ Hz, 2H), 3.86-3.77 (m, 2H), 3.50-3.41 (m, 2H), 1.90-1.78 (m, 2H), 1.78-1.72 (m, 4H), 1.50-1.47 (m, 4H); ^{13}C NMR (100 MHz, CDCl_3) δ 164.9, 152.5, 150.1, 137.7, 126.6, 123.5, 98.9, 66.8, 65.4, 62.4, 30.7, 26.2, 25.6, 25.4, 19.6; MS (70 eV) 279 (M^+ , 3), 278 (3), 224 (10), 178 (20), 123 (20), 105 (100), 85 (43), 77 (47), 54 (20); TLC R_f 0.47 (EtOAc/*n*-hexane, 1/9); HR-MS calcd for $\text{C}_{15}\text{H}_{21}\text{NO}_4$: 279.1471, found: 279.1465.

4-(*tert*-Butyl-dimethylsilanyloxy)-butyl 3-phenylpropanoate-10

IR (CCl_4) 2952 (s), 1735 (s), 1604 (w), 1496 (m), 1470 (m), 1454 (M), 1361 (m), 1255 (s), 1161 (s), 1101 (s), 1047 (s); ^1H NMR (400 MHz, CDCl_3) δ 7.30-7.27 (m, 2H), 7.20-7.19 (m, 3H), 4.09 (t, $J = 6.5$ Hz, 2H), 3.62 (t, $J = 6.2$ Hz, 2H), 2.96 (t, $J = 7.6$ Hz, 2H), 2.63 (t, $J = 8.0$ Hz, 2H), 1.69-1.65 (m, 2H), 1.56-1.52 (m, 2H), 0.91 (s, 9H), 0.04 (s, 6H); ^{13}C NMR (100 MHz, CDCl_3) δ 172.9, 140.5, 128.4, 128.2, 126.2, 64.4, 62.5, 35.9, 31.0, 29.1, 25.9, 25.2, 18.3, -5.4; MS (70 eV) 336 (M^+ , 03), 279 (61), 206 (100), 187 (43), 147 (40), 133 (78), 105 (95), 91 (60), 75 (37); TLC R_f 0.57 (EtOAc/*n*-hexane, 2/98); HR-MS calcd for $\text{C}_{19}\text{H}_{32}\text{O}_3\text{Si}$: 336.2121, found: 336.2115.

Diphenylmethyl 3-phenylpropanoate (11)¹³

m.p. 92-93 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.56-7.33 (m, 12H), 7.30-7.25 (m, 3H), 7.01 (s, 1H), 3.09 (t, *J* = 7.8 Hz, 2H), 2.85 (t, *J* = 7.8 Hz, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 171.7, 140.2, 140.1, 128.42, 128.38, 128.2, 127.8, 127.0, 126.2, 76.7, 36.0, 30.8; MS (EI, 70 eV) 316 (M⁺, 5), 184 (20), 167 (100), 166 (60), 152 (17).

1-Phenethyl-but-3-enyl 3-phenylpropanoate (12)¹⁴

¹H NMR (400 MHz, CDCl₃) δ 7.36-7.31 (m, 4H), 7.29-7.18 (m, 6H), 5.81-5.71 (m, 1H), 5.14-5.09 (m, 2H), 5.07-5.01 (m, 1H), 3.01 (t, *J* = 7.6 Hz, 2H), 2.69-2.57 (m, 4H), 2.39 (t, *J* = 7.6 Hz, 2H), 1.98-1.86 (m, 2H); ¹³C NMR (100 MHz, CDCl₃) δ 172.4, 141.4, 140.4, 133.4, 128.4, 128.3, 128.24, 128.22, 126.2, 125.8, 117.7, 72.8, 38.6, 35.9, 35.2, 31.6, 30.9; MS (EI, 70 eV) 308 (M⁺, 10), 223 (30), 158 (65), 116 (100), 90 (90), 77 (30).

2'-Hexanoyloxy-biphenyl-2-yl hexanoate-13

IR (CCl₄) 3067 (m), 2936 (s), 2871 (s), 1947 (w), 1914 (w), 1758 (s), 1604 (w), 1504 (w), 1442 (s), 1416 (s), 1364 (s), 1314 (s), 1272 (s), 1138 (s), 1045 (s), 943 (s), 908 (s); ¹H NMR (400 MHz, CDCl₃) δ 7.38 (ddd, *J* = 9.1, 6.6, 2.6 Hz, 2H), 7.31-7.25 (m, 4H), 7.13 (dd, *J* = 7.8, 0.9 Hz, 2H), 2.28 (t, *J* = 7.5 Hz, 4H), 1.45 (quint, *J* = 7.4 Hz, 4H), 1.27-1.13 (m, 8H), 0.85 (t, *J* = 7.1 Hz, 6H); ¹³C NMR (100 MHz, CDCl₃) δ 172.0, 148.2, 131.2, 130.7, 128.8, 125.7, 122.4, 34.0, 31.0, 24.3, 22.2, 13.8; MS (70 eV) 382 (M⁺, 3), 284 (18), 186 (18), 185 (100), 99 (17), 71 (14); TLC R_f 0.47 (EtOAc/*n*-hexane, 2/98); HR-MS calcd for C₂₄H₃₀O₄: 382.2144, found: 382.2139.

(13) Takahashi, S.; Cohen, L. A.; Miller, H. K.; Peake, E. G. *J. Org. Chem.* **1971**, 36, 1205.

(14) Harada T.; Mukaiyama T. *Chem.Lett.* **1981**, 1109.

2-Cholesteryl oleate: Cholesteryloleatooctadec-9-enoic acid 17-(1,5-dimethyl-hexyl)-10,13-dimethyl-2,3,4,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-1H-cyclopenta[a]phenanthren-3-yl ester (14)¹⁵

m.p. 43-44 °C; ¹H NMR (400 MHz, CDCl₃) δ 5.38- 5.33 (m, 3H), 4.65-4.60 (m, 1H), 2.32-2.26 (m, 4H), 2.01-1.83 (m, 10H), 1.59-1.26 (m, 12H), 1.25-1.01 (m, 23H), 1.01-0.85 (m, 22H), 0.68 (bs, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 173.3, 139.7, 130.0, 129.7, 122.6, 73.7, 56.7, 56.1, 50.0, 42.3, 39.7, 39.5, 38.2, 37.0, 36.6, 36.2, 35.8, 34.7, 31.90, 31.86, 29.8, 29.7, 29.6, 29.5, 29.33, 29.32, 29.2, 29.1, 28.2, 28.0, 27.8, 27.21, 27.15, 25.0, 24.3, 23.8, 22.8, 22.7, 22.6, 21.0, 19.3, 18.7, 14.1, 11.8.

Benzyl 2-hydroxy-2-phenylacetate (15)¹⁶

m.p. 92-93 °C; ¹H NMR (400 MHz, CDCl₃) δ 7.46-7.44 (m, 2H), 7.38-7.33 (m, 6H), 7.24-7.21 (m, 2H), 5.25 (s, 2H), 5.25 (d, *J* = 12.3 Hz, 1H), 5.16 (d, *J* = 12.3 Hz, 1H) 3.45 (s, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 173.4, 138.1, 134.9, 128.5, 128.43, 128.36, 128.3, 127.8, 126.5, 72.9, 67.5; MS (EI, 70 eV) 242 (M⁺, 3), 165 (15), 107 (100), 90 (70), 78 (65), 77 (55).

4-Methoxyphenyl 1-carboxyl-1,2,2-trimethyl-cyclopentane-3-carboxylate-16

IR (CCl₄) 2957 (br m), 1754 (m) 1699 (m), 1505 (m), 1463 (w), 1377 (w), 1282 (m), 1281 (m), 1193 (m), 1141 (m), 1041 (m), 957 (w); ¹H NMR (400 MHz, CDCl₃) δ 7.00 (d, *J* = 9.0, Hz, 2H), 6.88 (d, *J* = 9.0, Hz, 2H), 3.80 (s, 3H), 3.05 (t, *J* = 9.4 Hz, 1H), 2.65-2.56 (m, 1H), 2.35-2.24 (m, 1H), 2.00-1.90 (m, 1H), 1.59 (ddd, *J* = 13.6, 9.6, 3.8 Hz, 1H), 1.42 (s, 3H), 1.32 (s, 3H), 1.02 (s, 3H); ¹³C NMR (100 MHz, CDCl₃) δ 180.8, 172.8,

(15) (a) Hamilton N. O.; Cordes E. H. *J. Biol. Chem.* **1977**, 252, 8071. (b) Weber, N.; Weitkamp, P.; Mukherjee, K. D. *J. Agric. Food Chem.* **2001**, 49, 67.

(16) (a) Khumtaveeporn, K.; Ullmann, A.; Matsumoto, K.; Davis, B. G.; Jones, J. B. *Tetrahedron: Asymmetry* **2001**, 12, 249. (b) Jin, T.-S.; Ma, Y.-R.; Li, Y.; Sun, X.; Li, T.-S. *Synth. Commun.* **2001**, 31, 2051. (c) Zhang, G.-S.; Gong, H. *Synth. Commun.* **1999**, 29, 3149.

157.2, 144.3, 122.3, 114.5, 56.2, 55.6, 52.8, 47.1, 32.3, 23.0, 22.7, 21.6, 21.4; MS (70 eV) 306 (M^+ , 7), 155 (7), 124 (100), 109 (52), 95 (17); TLC R_f 0.45 (EtOAc/*n*-hexane, 3/7); HR-MS calcd for $C_{17}H_{22}O_5 + H$: 307.1540, found: 307.1544.

4-Methoxy-phenyl 7,7-dimethyl-2-oxo-bicyclo[2.2.1]-heptane-1-carboxylate-17

IR (CCl_4) 2958 (s), 1742 (s), 1610 (m), 1597 (m), 1504 (s), 144 (s), 1440 (s), 1391 (s), 1374 (s), 1316 (s), 1181 (s), 1005 (s), 960 (s); 1H NMR (400 MHz, $CDCl_3$) δ 7.04 (d, J = 9.0 Hz, 2H), 6.88 (d, J = 9.0 Hz, 2H), 3.78 (s, 3H), 2.59 (dt, J = 18.3, 3.9, 1H), 2.48 (ddd, J = 14.4, 11.6, 4.0 Hz, 1H), 2.16 (t, J = 4.4 Hz, 1H), 2.12-2.05 (m, 1H), 2.01 (d, J = 18.4 Hz, 1H), 1.92 (ddd, J = 14.2, 9.3, 4.9 Hz, 1H), 1.46 (ddd, J = 12.9, 9.4, 4.0 Hz, 1H), 1.25 (s, 3H), 1.18 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.3, 168.9, 157.2, 144.0, 122.4, 114.3, 68.0, 55.5, 49.4, 44.4, 43.9, 26.4, 26.3, 21.3, 19.8; MS (70 eV) 288 (M^+ , 30), 165 (100), 123 (57), 109 (32), 95 (34), 67 (15); TLC R_f 0.45 (EtOAc/*n*-hexane, 2/98); HR-MS calcd for $C_{17}H_{20}O_4$: 288.1362, found: 288.1356.

Polymer between adipic acid-diethylene glycol-18

IR (CCl_4) 2951 (w), 1738 (s), 1454 (w), 1380 (w), 1262 (m), 1147 (m), 1070 (w); 1H NMR (400 MHz, $CDCl_3$) δ 4.11 (br d, 4H), 3.58 (br d, 4H), 2.20 (br d, 4H), 1.57 (br d, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.9, 68.8, 63.1, 33.4, 24.0; Degree of polymerization $\sim$ 100, $M_n = 1.64 \times 10^4$, $M_w = 2.24 \times 10^4$.

Current Data Parameters

NAME	0916
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20040916
Time	9.49
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	16384
SOLVENT	CDCl3
NS	16
DS	0
SWH	5995.204 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	50.8
DW	83.400 usec
DE	6.50 usec
TE	300.0 K
D1	1.5000000 sec

===== CHANNEL f1 =====

NUC1	1H
P1	10.10 usec
PL1	3.00 dB
SFO1	400.1326008 MHz

F2 - Processing parameters

SI	16384
SF	400.1300088 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

ID NMR plot parameters

CX	20.00 cm
CY	10.50 cm
F1P	10.500 ppm
F1	4201.37 Hz
F2P	-0.500 ppm
F2	-200.05 Hz
PPMCM	0.55000 ppm/cm
HZCM	220.07150 Hz/cm

Current Data Parameters

NAME	0916
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20040916
Time	9.52
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	44
DS	0
SWH	25125.629 Hz
FIDRES	0.383387 Hz
AQ	1.3042154 sec
RG	8192
DW	19.900 usec
DE	6.50 usec
TE	300.0 K
D1	1.2000000 sec
d11	0.0300000 sec
d12	0.0000200 sec

===== CHANNEL f1 =====

NUC1	13C
P1	10.80 usec
PL1	7.00 dB
SFO1	100.6242995 MHz

===== CHANNEL f2 =====

CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	3.00 dB
PL12	21.83 dB
PL13	24.80 dB
SFO2	400.1319000 MHz

F2 - Processing parameters

SI	32768
SF	100.6127821 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

ID NMR plot parameters

CX	20.00 cm
CY	12.50 cm
F1P	220.000 ppm
F1	22134.81 Hz
F2P	-10.390 ppm
F2	-1045.41 Hz
PPMCM	11.51952 ppm/cm
HZCM	1159.01099 Hz/cm

Current Data Parameters
NAME 040917
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040917
Time 10.25
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SMH 5995.204 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 128
DM 63.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.50000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.10 usec
PL1 3.00 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 15384
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.50 cm
F1P 10.500 ppm
F1 4201.37 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME 040917
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040917
Time 10.27
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 43
DS 0
SMH 25125.629 Hz
FIDRES 0.303381 Hz
AQ 1.3042164 sec
RG 8192
DM 19.300 usec
DE 6.50 usec
TE 300.0 K
D1 1.20000005 sec
d11 0.03000000 sec
d12 0.00020000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 10.80 usec
PL1 7.00 dB
SFO1 100.6242995 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.00 dB
PL12 21.83 dB
PL13 24.80 dB
SFO2 400.1319000 MHz

F2 - Processing parameters
SI 32768
SF 100.6127736 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.307 ppm
F2 -1036.97 Hz
PPMCM 11.51533 ppm/cm
HZCM 1158.58911 Hz/cm

Current Data Parameters
NAME 00901
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040901
Time 14.39
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 22.6
DW 83.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.50000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.10 usec
PL1 3.00 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300051 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.50 cm
F1P 10.500 ppm
F1 4201.37 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME 00901
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040901
Time 14.43
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 84
DS 0
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 8192
DW 19.900 usec
DE 6.50 usec
TE 300.0 K
D1 1.20000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.80 usec
PL1 7.00 dB
SFO1 100.6242995 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.00 dB
PL12 21.63 dB
PL13 24.80 dB
SFO2 400.1319000 MHz

F2 - Processing parameters
SI 32768
SF 100.6128020 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 220.000 ppm
F1 22134.82 Hz
F2P -10.588 ppm
F2 -1065.34 Hz
PPMCM 11.52943 ppm/cm
HZCM 1160.09781 Hz/cm

Current Data Parameters
NAME 0915
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040915
Time 10.06
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 30.5
DW 83.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.5000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.10 usec
PL1 3.00 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300084 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.50 cm
F1P 10.500 ppm
F1 4201.37 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME 0915
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040915
Time 10.08
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 55
DS 0
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 8192
DW 19.900 usec
DE 6.50 usec
TE 300.0 K
D1 1.2000000 sec
d11 0.0300000 sec
d12 0.0000200 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 7.00 dB
SFO1 100.6242995 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.00 dB
PL12 21.83 dB
PL13 24.80 dB
SFO2 400.1319000 MHz

F2 - Processing parameters
SI 32768
SF 100.6127813 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 22134.81 Hz
F1 22134.81 Hz
F2P -10.383 ppm
F2 -1044.54 Hz
PPMCM 11.51914 ppm/cm
HZCM 1158.97253 Hz/cm

Current Data Parameters
NAME 0916
EXPNO 11
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040916
Time 15.55
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 50.8
DM 83.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.5000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.10 usec
PL1 3.00 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.50 cm
F1P 10.500 ppm
F1 4201.37 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME 0916
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040916
Time 15.57
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 42
DS 0
SWH 25125.629 Hz
FIDRES 0.383367 Hz
AQ 1.3042164 sec
RG 8192
DM 19.900 usec
DE 6.50 usec
TE 300.0 K
D1 1.20000005 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 10.80 usec
PL1 7.00 dB
SFO1 100.6242995 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.00 dB
PL12 21.83 dB
PL13 24.80 dB
SFO2 400.1319000 MHz

F2 - Processing parameters
SI 32768
SF 100.6127844 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.413 ppm
F2 -1047.70 Hz
PPMCM 11.52066 ppm/cm
HZCM 1159.12585 Hz/cm

Current Data Parameters
NAME 041002
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041002
Time 12.53
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 15
DS 0
SWH 5995.204 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 22.6
DM 83.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.50000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.10 usec
PL1 3.00 dB
SF01 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300080 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.50 cm
F1P 10.500 ppm
F1 4201.37 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PQMCN 0.55000 ppm/cm
HZCN 220.07150 Hz/cm

Current Data Parameters
NAME 041002
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041002
Time 12.55
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 30
DS 0
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 8192
DM 19.900 usec
DE 6.50 usec
TE 300.0 K
D1 1.20000005 sec
D11 0.03000000 sec
D12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 10.80 usec
PL1 7.00 dB
SF01 100.6242995 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.00 dB
PL12 21.83 dB
PL13 24.80 dB
SF02 400.1319000 MHz

F2 - Processing parameters
SI 32768
SF 100.6128051 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 220.000 ppm
F1 22134.82 Hz
F2P -10.519 ppm
F2 -1058.41 Hz
PQMCN 11.53095 ppm/cm
HZCN 1160.16125 Hz/cm

Current Data Parameters
NAME 041018
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041018
Time 14.47
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 22.6
DM 83.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.50000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.10 usec
PL1 3.00 dB
SF01 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300050 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.50 cm
F1P 10.500 ppm
F1 4201.37 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.55000 ppm/
HZCM 220.07150 Hz/c

Current Data Parameters
NAME 041018
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041018
Time 14.50
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 141
DS 0
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 8192
DM 19.900 usec
DE 6.50 usec
TE 300.0 K
D1 1.20000005 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 10.80 usec
PL1 7.00 dB
SF01 100.6242993 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.00 dB
PL12 21.83 dB
PL13 24.80 dB
SF02 400.1319000 MHz

F2 - Processing parameters
SI 32768
SF 100.6127997 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 220.000 ppm
F1 22134.82 Hz
F2P -10.565 ppm
F2 -1063.04 Hz
PPMCM 11.52828 ppm/cm

Current Data Parameters
NAME 040920
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040920
Time 15.15
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 80.6
DM 83.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.50000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.10 usec
PL1 3.00 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.50 cm
F1P 10.500 ppm
F1 4201.37 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME 040920
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040920
Time 15.18
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 21
DS 0
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 8192
DM 19.900 usec
DE 6.50 usec
TE 300.0 K
D1 1.20000005 sec
d1t 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 7.00 dB
SFO1 100.6242995 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.00 dB
PL12 21.83 dB
PL13 24.80 dB
SFO2 400.1319000 MHz

F2 - Processing parameters
SI 32768
SF 100.6127798 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.368 ppm
F2 -1043.11 Hz
PPMCM 11.51838 ppm/cm
HZCM 1158.89587 Hz/cm

Current Data Parameters
NAME 0916
EXPNO 9
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040916
Time 13.55
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 22.5
DE 83.400 usec
TE 300.0 K
D1 1.50000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.10 usec
PL1 3.00 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.50 cm
F1P 10.500 ppm
F1 4201.37 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME 0916
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040916
Time 13.57
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 51
DS 0
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 8192
DE 19.900 usec
TE 300.0 K
D1 1.20000005 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.80 usec
PL1 7.00 dB
SFO1 100.6242995 MHz

===== CHANNEL f2 =====
CPOPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.00 dB
PL12 21.83 dB
PL13 24.80 dB
SFO2 400.1319000 MHz

F2 - Processing parameters
SI 32768
SF 100.6127913 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.482 ppm
F2 -1054.61 Hz
PPMCM 11.52405 ppm/cm
HZCM 1159.47107 Hz/cm

Current Data Parameters

NAME	041009
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20041009
Time	10.14
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zg30
TD	16384
SOLVENT	CDCl3
NS	16
DS	0
SWH	5005.204 Hz
FIDRES	0.365918 Hz
AQ	1.3664756 sec
RG	322.5
DM	83.400 usec
DE	5.50 usec
TE	300.0 K
D1	1.5000000 sec

===== CHANNEL f1 =====

NUC1	1H
P1	10.10 usec
PL1	3.00 dB
SFO1	400.1326008 MHz

F2 - Processing parameters

SI	16384
SF	400.1300095 MHz
WDW	EM
SSB	0
LB	0.10 Hz
GB	0
PC	1.00

1D NMR plot parameters

CX	20.00 cm
CY	10.00 cm
F1P	10.500 cm
F1	4201.37 Hz
F2P	-1.015 ppm
F2	-406.25 Hz
PPMCM	0.57577 ppm/cm
HZCM	230.30004 Hz/cm

Current Data Parameters

NAME	050009
EXPNO	2
PROCNO	1

F2 - Acquisition Parameters

Date_	20050808
Time	17.27
INSTRUM	spect
PROBHD	5 mm BBO BB-1H
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1368
DS	0
SWH	25175.409 Hz
FIDRES	0.383087 Hz
AQ	1.3042164 sec
RG	8192
DM	19.500 usec
DE	8.50 usec
TE	300.0 K
D1	1.2000000 sec
D11	0.0300000 sec
D12	0.0000000 sec

===== CHANNEL f1 =====

NUC1	13C
P1	12.00 usec
PL1	7.00 dB
SFO1	100.6242895 MHz

===== CHANNEL f2 =====

CPDPRG2	zgpg1215
NUC2	1H
PCPD2	90.00 usec
PL2	3.00 dB
PL12	21.83 dB
PL13	24.80 dB
SFO2	400.1300095 MHz

F2 - Processing parameters

SI	32768
SF	100.6127721 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

1D NMR plot parameters

CX	20.00 cm
CY	10.00 cm
F1P	200.800 ppm
F1	20134.81 Hz
F2P	-18.201 ppm
F2	-1025.44 Hz
PPMCM	51.51457 ppm/cm
HZCM	1158.51833 Hz/cm

Current Data Parameters
NAME 041007
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041007
Time 12.45
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 80.6
DM 83.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.50000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.10 usec
PL1 3.00 dB
SFO1 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300050 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.50 cm
F1P 10.500 ppm
F1 4201.37 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.55000 ppm/ci
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME 041007
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041007
Time 12.48
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 54
DS 0
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042154 sec
RG 8132
DM 19.900 usec
DE 6.50 usec
TE 300.0 K
D1 1.20000005 sec
d11 0.03000000 sec
d12 0.00002000 sec

***** CHANNEL f1 *****
NUC1 13C
P1 10.80 usec
PL1 7.00 dB
SFO1 100.6242995 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.00 dB
PL12 21.63 dB
PL13 24.60 dB
SFO2 400.1319000 MHz

F2 - Processing parameters
SI 32768
SF 100.6127752 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.322 ppm
F2 -1038.50 Hz
PPMCM 11.51609 ppm/cm
HZCM 1158.66577 Hz/cm

Current Data Parameters
 NAME 041010
 EXPNO 1
 PROCNO 1
 F2 - Acquisition Parameters
 Date_ 20041010
 Time 12.32
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TO 16384
 SOLVENT CDCl3
 NS 16
 DS 4
 SWH 5985.204 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 40.3
 OW 83.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.50000000 sec

***** CHANNEL f1 *****
 NUC1 1H
 P1 10.10 usec
 PL1 3.00 dB
 SF01 400.1326008 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300050 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 10.50 cm
 F1P 10.500 ppm
 F1 4201.37 Hz
 F2P -0.500 ppm
 F2 -200.06 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 220.07150 Hz/cm

Current Data Parameters
 NAME 041010
 EXPNO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20041010
 Time 12.34
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TO 65536
 SOLVENT CDCl3
 NS 36
 DS 0
 SWH 25125.629 Hz
 FIDRES 0.383387 Hz
 AQ 1.3042164 sec
 RG 8192
 OW 19.900 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.20000005 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

***** CHANNEL f1 *****
 NUC1 13C
 P1 10.80 usec
 PL1 7.00 dB
 SF01 100.6242995 MHz

***** CHANNEL f2 *****
 CROPRG2 waltz16
 NUC2 1H
 PCPRG2 90.00 usec
 PL2 3.00 dB
 PL12 21.83 dB
 PL13 24.80 dB
 SF02 400.1319000 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127905 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -10.474 Hz

Current Data Parameters
NAME 041020
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041020
Time 8.37
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 22.6
DW 83.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.5000000 sec

***** CHANNEL f1 *****
NUC1 1H
P1 10.10 usec
PL1 3.00 dB
SF01 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300050 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.50 cm
F1P 10.500 ppm
F1 4201.37 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME 041020
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041020
Time 8.40
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 8192
DW 19.900 usec
DE 6.50 usec
TE 300.0 K
D1 1.2000000 sec
d11 0.0300000 sec
d12 0.0000200 sec

***** CHANNEL f1 *****
NUC1 13C
P1 10.80 usec
PL1 7.00 dB
SF01 100.6242995 MHz

***** CHANNEL f2 *****
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.00 dB
PL12 21.83 dB
PL13 24.80 dB
SF02 400.1319000 MHz

F2 - Processing parameters
SI 32768
SF 100.6127836 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.406 ppm
F2 -1046.94 Hz
PPMCM 11.52028 ppm/cm
HZCM 1159.08765 Hz/cm

Current Data Parameters
 NAME 041002
 EXPNO 3
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20041002
 Time 13.01
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zg30
 TD 16384
 SOLVENT CDCl3
 NS 16
 DS 0
 SWH 5995.204 Hz
 FIDRES 0.365918 Hz
 AQ 1.3664756 sec
 RG 45.3
 DM 83.400 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.50000000 sec

===== CHANNEL f1 =====
 NUC1 1H
 P1 10.10 usec
 PL1 3.00 dB
 SFO1 400.1326008 MHz

F2 - Processing parameters
 SI 16384
 SF 400.1300095 MHz
 WDW EM
 SSB 0
 LB 0.10 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 CY 10.50 cm
 F1P 10.500 ppm
 F1 4201.37 Hz
 F2P -0.500 ppm
 F2 -200.06 Hz
 PPMCM 0.55000 ppm/cm
 HZCM 220.07150 Hz/cm

Current Data Parameters
 NAME 041002
 EXPNO 4
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20041002
 Time 13.03
 INSTRUM spect
 PROBHD 5 mm BBO BB-1H
 PULPROG zgpg30
 TD 65536
 SOLVENT CDCl3
 NS 34
 DS 0
 SWH 25125.629 Hz
 FIDRES 0.383387 Hz
 AQ 1.3042164 sec
 RG 8192
 DM 19.900 usec
 DE 6.50 usec
 TE 300.0 K
 D1 1.20000000 sec
 d11 0.03000000 sec
 d12 0.00002000 sec

===== CHANNEL f1 =====
 NUC1 13C
 P1 10.80 usec
 PL1 7.00 dB
 SFO1 100.6242995 MHz

===== CHANNEL f2 =====
 CPDPRG2 waltz16
 NUC2 1H
 PCPD2 90.00 usec
 PL2 3.00 dB
 PL12 21.83 dB
 PL13 24.80 dB
 SFO2 400.1319000 MHz

F2 - Processing parameters
 SI 32768
 SF 100.6127867 MHz
 WDW EM
 SSB 0
 LB 1.00 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 CY 12.50 cm
 F1P 220.000 ppm
 F1 22134.81 Hz
 F2P -10.436 ppm
 F2 -1050.01 Hz
 PPMCM 11.52180 ppm/cm
 HZCM 1159.24097 Hz/cm

Current Data Parameters
NAME 041022
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041022
Time 10.05
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 5995.204 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 64
DW 83.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.50000000 sec

===== CHANNEL f1 =====
NUC1 1H
P1 10.10 usec
PL1 3.00 dB
SF01 400.1326008 MHz

F2 - Processing parameters
SI 16384
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 10.50 cm
F1P 10.500 ppm
F1 4201.37 Hz
F2P -0.500 ppm
F2 -200.06 Hz
PPMCM 0.55000 ppm/cm
HZCM 220.07150 Hz/cm

Current Data Parameters
NAME 041022
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041022
Time 10.10
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 80
DS 0
SWH 25125.629 Hz
FIDRES 0.383387 Hz
AQ 1.3042164 sec
RG 8192
DW 19.900 usec
DE 6.50 usec
TE 300.0 K
D1 1.20000005 sec
d11 0.03000000 sec
d12 0.00002000 sec

===== CHANNEL f1 =====
NUC1 13C
P1 10.80 usec
PL1 7.00 dB
SF01 100.6242995 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 3.00 dB
PL12 21.83 dB
PL13 24.80 dB
SF02 400.1319000 MHz

F2 - Processing parameters
SI 32768
SF 100.6127775 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 12.50 cm
F1P 220.000 ppm
F1 22134.81 Hz
F2P -10.345 ppm
F2 -1040.80 Hz
PPMCM 11.51723 ppm/cm
HZCM 1158.78088 Hz/cm