Supporting Information

Selective Oxoammonium Salt Oxidations of Alcohols to Aldehydes and Aldehydes to Carboxylic Acids

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Experimental Section

General. Compound numbers and Tables refer to those contained in the main body of the paper. The majority of alcohols and aldehydes used in this study were commercially available; 2,2,6,6-tetramethylpiperidine-1-oxoammonium tetrafluoroborate (**1**) was prepared following the procedure of Bobbitt and Merbough.¹ Literature procedures, incorporating some minor modifications, were followed for the preparation of 3-(4-hydroxymethylphenyl)-1-propanol² and 3-(4-hydroxymethylphenoxy)-propan-1-ol.³

Relative Rates of Oxidation of Primary Alcohols to Aldehydes (Table 1).

Slurries consisting of equimolar quantities (typically 2 mmol) of two alcohols and one molar equivalent of **1** in CH₂Cl₂ (typically 25 mL) were allowed to stand at room temperature until the the yellow slurry became white and the solution gave a negative starch-KI test. An aliquot was then filtered through a cotton plug into an NMR tube, a sealed capillary containing CDCl₃ was inserted in the NMR tube to serve as an external reference and lock, and spectra were recorded with suppression of the CH₂Cl₂ resonance at δ 5.30 ppm. Multiple reaction mixtures, involving various combinations of alcohols, were run to completion in this way and integration of the carbinol CH₂ resonance in the alcohols and the CHO resonance in the aldehyde products were used to obtain the relative rate data reported in Table 1. The propagated experimental error of the measurements was ~5 %.

(1) Bobbitt, J. M.; Merbough, N. *Organic Syntheses*; Wiley: New York, **2009**; Collect. Vol. 11, p 93.

(2) Tanaka, K.; Shibata Y.; Suda, T.; Hagiwara Y.; Hirano M. *Org. Lett.* **2007**, 9, 1215.

(3) Linares, M. L.; Sánchez, N.; Alajarin, R.; Vaquero, J. J.; Alvarez-Builla, J. *Synthesis* **2001**, 382.

Relative Rates of Oxidation of Aldehydes to Carboxylic Acids (Table 3).

Approximately 0.17 M solutions of equimolar quantities (typically 2 mmol) of two aldehydes and one molar equivalent of **1** in CH₃CN – D₂O (9:1 by vol) were allowed to stand at room temperature for 24 h. An aliquot was then removed, a sealed capillary containing CDCl₃ was inserted in the NMR tube to serve as an external reference, and spectra were recorded with suppression of the CH₃CN resonance at δ 2.06 ppm. Multiple reaction mixtures, involving various combinations of aldehydes, were run to completion in this way and integration of the CHO resonance in the residual aldehydes were used to obtain the relative rate data reported in Table 3. The propagated experimental error of the measurements was ~6 %.

General Procedure for the Oxidation of Alcohols to Carboxylic Acids (Table 2). An approximately 0.25 M solution of a primary alcohol in CH₃CN – H₂O (9:1 by vol) was allowed to sit at room temperature (typically overnight for unhindered aliphatic alcohols) until the orange-brown solution faded to clear orange-yellow. The reaction mixture was then diluted to twice its volume with deionized water and extracted with five portions of Et₂O. The combined organic layers were washed sequentially with 5 mL of 10% aq. HCl (to remove any nitroxide) and 10 mL of brine and then dried (MgSO₄). Concentration by rotary evaporation afforded essentially pure carboxylic acids; yields are reported in Table 2. The acids prepared in this way displayed the following properties.

Octanoic Acid. oil: ¹H NMR (400 MHz, CDCl₃) δ 11.09 (br s, 1 H), 2.35 (t, 2 H, J = 5.0 Hz), 1.62 (q, 2 H, J = 5.0 Hz), 1.32 - 1.28 (m, 8 H), 0.88 (t, 3 H, J = 5.0 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 180.6, 34.2, 31.7, 29.1, 29.0, 24.8, 22.7, 14.1.

Decanoic Acid. semi-solid; ^1H NMR (400 MHz, CDCl_3) δ 11.9 (br s, 1 H), 2.32 (t, 2 H, $J = 7.0$ Hz), 1.65 - 1.58 (m, 2 H), 1.29 - 1.25 (m, 12 H), 0.86 (t, 3 H, $J = 7.0$ Hz); δ 180.3, 34.3, 32.0, 29.5, 29.3, 29.2 (2 C), 24.8, 22.8, 14.2.

2-Methylpentanoic Acid. oil; ^1H NMR (400 MHz, CDCl_3) δ 10.75 (br s, 1H), 2.47 (m, 1H), 1.67 (m, 1H), 1.52 - 1.26 (m, 3H), 1.18 (d, 3H, $J = 6.0$ Hz), 0.92 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 183.3, 39.3, 35.7, 20.4, 16.9, 14.0.

Cyclohexanecarboxylic Acid. semi-solid; ^1H NMR (400 MHz, CDCl_3) δ 11.31 (br s 1 H), 2.33 (t, 1H, $J = 11.2$ Hz), 1.93 (d, 2H, $J = 12.4$ Hz), 1.76 (d, 2H, $J = 11.6$ Hz), 1.65 (d, 1H, $J = 8.0$ Hz), 1.45 (q, 2H, $J = 10.8$ Hz), 1.37 - 1.14 (m, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 182.8, 43.0, 28.9, 25.5, 25.2.

Pivalic Acid. semi-solid; ^1H NMR (300 MHz, CDCl_3) δ 11.79 (s, 1H), 1.23 (s, 9H); ^{13}C NMR (75 MHz, CDCl_3) δ 186.0, 38.8, 27.2.

***p*-Methoxyphenylacetic Acid.** solid, mp 79-81 C (lit.⁴ mp 77-79 °C); ^1H NMR (400 MHz, CDCl_3) δ 9.49 (br s, 1H), 7.20 (d, 2H, $J = 8.3$ Hz), 6.87 (d, 2H, $J = 8.3$ Hz), 3.80 (s, 3H), 3.59 (s, 2H); ^{13}C NMR (100 MHz, CDCl_3) δ 178.2, 159.0, 130.6, 125.6, 114.3, 55.4, 40.3.

9-Decenoic Acid. oil; ^1H NMR (400 MHz, CDCl_3) δ 11.60 (br s, 1H), 5.73 - 5.83 (m, 1H), 4.88 - 5.00 (m, 2H), 2.32 (t, 2H, $J = 7.5$ Hz), 2.00 - 2.07 (m, 2H), 1.57 - 1.68 (m, 2H), 1.24 - 1.43 (m, 8H); ^{13}C NMR (100 MHz, CDCl_3) δ 180.4, 138.8, 114.1, 34.0, 33.7, 29.04, 29.0, 28.9, 28.8, 24.6.

(4) Laird, T.; Williams, H. *J. Chem. Soc. C* **1971**, 1863.

11-Bromoundecanoic Acid. solid, mp 45-48 °C (lit.⁵ mp 44-47 °C); ¹H NMR (500 MHz, CDCl₃) δ 9.78 (br s, 1H), 3.42 (t, 2H, J = 6.9 Hz), 2.36 (t, 2H, J = 7.5 Hz), 1.82-1.91 (m, 4H), 1.60 - 1.70 (m, 4H), 1.39 - 1.48 (m, 4H), 1.28 - 1.34 (m, 4H); ¹³C NMR (125 MHz, CDCl₃) δ 180.1, 34.0, 34.0, 32.8, 29.3, 29.3, 29.2, 29.0, 28.7, 28.1, 24.6.

Decanedioic Acid. solid, mp 131-133 °C (lit.⁶ mp 129-131 °C); ¹H NMR (500 MHz, DMSO) δ 11.9 (br s, 2H), 2.19 (t, 4H, J = 7.4 Hz), 1.44 - 1.56 (m, 8H), 1.20 - 1.30 (m, 4H); ¹³C NMR (125 MHz, DMSO) δ 174.9, 34.1, 29.1, 29.0, 24.9.

3-Pentynoic Acid. solid, mp 102-104 °C (lit.⁷ mp 103.5-104.5 °C); ¹H NMR (500 MHz, CDCl₃) δ 10.3 (br s, 1H), 3.32 (q, 2H, J = 2.6 Hz), 1.84 (t, 3H, J = 2.6 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 175.3, 79.9, 69.7, 25.8, 3.50.

Oxidation of a Mixture of Benzyl Alcohol and 1-Octanol (Scheme 4). A solution of 8.33 g (27.7 mmol) of **1**, 1.00 g (9.25 mmol) of benzyl alcohol and 1.20 g (9.25 mmol) of 1-octanol in 35 mL of CH₃CN – H₂O (9:1 by vol) was stirred at room temperature for 8 h. The reaction mixture was diluted with 15 mL of water and extracted with three 20-mL portions of Et₂O. The combined ether extract was extracted three 15-mL portions of sat., aq. Na₂CO₃. The organic layer was washed with 10 mL of water and 10 mL of brine, dried (MgSO₄) and concentrated to give 0.88 g (90%) of benzaldehyde as a colorless liquid: ¹H NMR (CDCl₃, 400 MHz) δ 10.05 (s, 1H), 8.06 (d, J = 4.00 Hz, 2H), 7.88-7.87 (m, 1H), 7.54 - 7.44 (m, 2H); ¹³C NMR (CDCl₃, 100 MHz) δ 192.6, 136.6, 134.6, 129.9, 129.2. The combined aqueous extract was acidified with 10% aq. HCl and

(5) Kling, M. R; Easton C. J; Poulos, A *J. Chem. Soc., Perkin Trans. I* **1993**, 10, 1183.

(6) Ansell, M. F; Shepherd I. S; Weedon, B. C. L *J. Chem. Soc. C* **1971**, 1840.

(7) Bigley, D. B; Weatherhead R. H. *J. Chem. Soc., Perkin Trans. 2* **1976**, 592.

extracted with three 10-mL portions of Et₂O. The combined ether extract was washed with 10 mL of water and 10 mL of brine, dried (MgSO₄) and concentrated to give 1.01 g (91%) of octanoic acid as a colorless liquid: ¹H NMR (CDCl₃, 400 MHz) δ 11.90 (bs, 1H), 2.33 (t, J = 8.00 Hz, 2H), 1.64 - 1.59 (m, 2H), 1.30 - 1.27 (m, 8H), 0.89 - 0.85 (m, 3H); ¹³C NMR (CDCl₃, 100 MHz) δ 180.9, 34.3, 31.8, 29.2, 29.1, 24.9, 22.8, 4.2.

Oxidation of 3-(4-Hydroxymethylphenyl)-1-propanol (Scheme 4). A solution of 920 mg (3.07 mmol) of **1** and 170 mg (1.02 mmol) of 3-(4-hydroxymethylphenyl)-1-propanol² in 3.8 mL of CH₃CN – H₂O (9:1 by vol) was stirred at room temperature for 8 h. The reaction mixture was diluted with 1.5 mL of water and extracted with three 2-mL portions of Et₂O. The organic layer was washed with 1.5 mL of water and one 1.0 mL of brine, dried (MgSO₄) and concentrated to give 150 mg (83%) of 3-(formylphenyl)propionic acid as white solid: mp 134-138 °C [lit.⁸ mp 131-134 °C]; ¹H NMR (DMSO, 400 MHz) δ 12.2 (bs, 1H), 9.96 (s, 1H), 7.86 - 7.81 (m, 2H), 7.46 (d, J = 8.00 Hz, 2H), 2.92 (t, J = 7.60 Hz, 2H), 2.59 (t, J = 7.60 Hz, 2H); ¹³C NMR (DMSO, 100 MHz) δ 192.6, 173.4, 129.5, 129.3, 129.1, 128.4, 34.5, 30.4.

4-(3-Hydropropoxy)benzaldehyde (Scheme 5). A slurry of 547 mg (3.00 mmol) of 3-(4-hydroxymethylphenoxy)-propan-1-ol³ and 985 mg (3.28 mmol) of **1** in 25 mL of CH₂Cl₂ was stirred at room temperature for 2 h at which time the yellow slurry had become white. The slurry was filtered and the filter cake was rinsed with CH₂Cl₂. The filtrate was passed through a short column of silica gel and concentrated to give 537 mg (98%) of the known⁹ title compound as a clear, slightly yellow liquid: ¹H NMR (300

(8) Hoffmann-LaRoche. U.S. Patent 4038289, 1977; *Chem. Abstr.* **1977**, 87, 152001.

(9) Gaud, O.; Granet, R.; Kaouadji, M.; Krausz, P.; Blais, J.; Bolbach, G. *Can. J. Chem.* **1996**, 74, 481.

MHz, CDCl₃) δ 9.83 (s, 1H), 7.79 (d, 2H, J = 8.3 Hz), 6.97 (d, 2H, J = 8.5 Hz), 4.18 (t, 2H, J = 6.1 Hz), 3.85 (t, 2H, J = 5.9 Hz), 2.21 (br s, 1H), 2.06 (quintet, 2H, J = 6.0 Hz); ¹³C NMR (400 MHz, CDCl₃) δ 191.1, 164.2, 132.2, 130.0, 114.9, 65.7, 59.7, 32.0.

4-(3-Oxopropoxy)benzaldehyde (Scheme 5). A slurry of 518 mg (2.88 mmol) of 4-(3-hydropropoxy)benzaldehyde, 985 g (3.28 mmol) of **1** and 2.12 g of silica gel in 25 mL of CH₂Cl₂ was stirred for 4 d at which time the slurry had become white. The mixture was filtered, the filter cake was rinsed with CH₂Cl₂, the filtrate was passed through a short column of silica gel, and concentrated to give 499 mg (97%) of the known¹⁰ title dialdehyde as a clear liquid: ¹H NMR (300 MHz, CDCl₃) δ 9.88 (s, 2H), 7.83 (d, 2H, J = 8.7 Hz), 7.00 (d, 2H, J = 8.6 Hz), 4.39 (t, 2H, J = 6.1 Hz), 2.97 (t, 2H, J = 6.0 Hz); ¹³C NMR (100 MHz, CDCl₃) δ 199.5, 191.1, 163.6, 132.2, 130.3, 114.9, 61.9, 43.1.

3-(4-Formylphenoxy)propanoic Acid (Scheme 5). A solution of 469 mg (2.63 mmol) of 4-(3-oxopropoxy)benzaldehyde and 868 mg (2.89 mmol) of **1** in 10.5 mL of CH₃CN – H₂O (9:1 by vol) was stirred for 6 d and then diluted with 9 mL of water. The resulting solution was extracted sequentially with two 10-mL portions of Et₂O and four 5-mL portions of Et₂O. The combined ether extracts were washed with 10 mL of 10% aq. HCl and then extracted with 10 mL of saturated, aq. NaHCO₃. The aqueous extract was made acidic to litmus paper by addition of conc. HCl and extracted with four 10-mL portions of Et₂O. The ether extracts were washed with 10 mL of brine, dried (MgSO₄), and concentrated to give 483 mg (95%) of the title acid as a white solid: mp 128.5–129.5

(10) Kommreddy, A.; Bowsher, M. S.; Gunna, M. R.; Botha, K.; Vinod, T. K. *Tetrahedron Lett.* **2008**, 49, 4378.

°C (lit.¹¹ mp 127–128 °C); ¹H NMR (300 MHz, DMSO-d₆) δ 10.5 (br s, 1 H), 9.87 (s, 1H), 7.85 (d, 2H, J = 9 Hz), 7.12 (d, 2H, J = 9 Hz), 4.28 (t, 2H, J = 6 Hz), 2.74 (t, 2 H, J = 6 Hz); ¹³C NMR (75 MHz, CDCl₃) δ 191.3, 172.0, 163.3, 131.8, 129.7, 114.9, 64.2, 33.9.

(11) Gresham, T. L.; Jansen, J. E.; Shaver, F. W.; Bankert, R. A.; Beears, W. L.; Prendergast, M. G. *J. Am. Chem. Soc.* **1949**, *71*, 661.

11.09

Table 2, entry 1

2.36
2.35
2.33
1.65
1.63
1.62
1.60
1.32
1.31
1.28
0.90
0.88
0.86

Current Data Parameters
NAME Octanoic Acid
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100127
Time 20.34
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 2
SWH 5592.841 Hz
FIDRES 0.170680 Hz
AQ 2.9295092 sec
RG 57
DW 89.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.50000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.20 usec
PL1 -2.00 dB
SFO1 400.1464009 MHz

F2 - Processing parameters
SI 32768
SF 400.1440086 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

180.59

Table 2, entry 1

34.24
31.72
29.10
28.98
24.77
22.67
14.11

Current Data Parameters
NAME Octanoic Acid
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100127
Time 20.45
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 68
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 322.5
DW 20.850 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 0 dB
SFO1 100.6263500 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 110.00 usec
PL2 -2.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 400.1456010 MHz

F2 - Processing parameters
SI 32768
SF 100.6162804 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

S10

Table 2, entry 2

2.36
2.34
2.32
1.65
1.63
1.61
1.30
1.27
0.89
0.88
0.86

Current Data Parameters
NAME Decanoic Acid
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100916
Time 14.14
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 32
DS 2
SWH 5592.841 Hz
FIDRES 0.170680 Hz
AQ 2.9295092 sec
RG 57
DW 89.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.50000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.20 usec
PL1 -2.00 dB
SFO1 400.1464009 MHz

F2 - Processing parameters
SI 32768
SF 400.1440085 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

180.33

Table 2, entry 2

34.26
31.96
29.49
29.34
29.17
24.80
22.75
14.17

Current Data Parameters
NAME Decanoic Acid
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100916
Time 14.36
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 324
DS 4
SWH 23980.814 Hz
FIDRES 0.365918 Hz
AQ 1.3664756 sec
RG 32768
DW 20.850 usec
DE 6.00 usec
TE 300.2 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 0 dB
SFO1 100.6263500 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 110.00 usec
PL2 -2.00 dB
PL12 19.00 dB
PL13 19.00 dB
SFO2 400.1456010 MHz

F2 - Processing parameters
SI 32768
SF 100.6162789 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

S12

Table 2, entry 3

2.49
2.47
2.45
1.69
1.67
1.65
1.50
1.48
1.46
1.43
1.40
1.38
1.36
1.33
1.26
1.19
1.17
0.94
0.90

Current Data Parameters
NAME 2-Methylpentanoic Acid
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100917
Time 19.30
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4496.403 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 90.5
DW 111.200 usec
DE 6.00 usec
TE 300.2 K
D1 1.50000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.20 usec
PL1 0 dB
SFO1 300.1318008 MHz

F2 - Processing parameters
SI 32768
SF 300.1300062 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Current Data Parameters
NAME 2-Methylpentanoic Acid
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100917
Time 20.10
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 332
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.50 usec
TE 300.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 0 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -2.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677449 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

183.34
183.20

Table 2, entry 3

39.24
35.74

20.37
16.86
13.96

200 180 160 140 120 100 80 60 40 20 0 ppm

S14

11.31

Table 2, entry 4

2.35
2.34
2.33
2.31
2.30
1.95
1.91
1.77
1.74
1.66
1.64
1.49
1.47
1.44
1.41
1.37
1.33
1.30
1.27
1.25
1.21
1.18

Current Data Parameters
NAME Cyclohexanecarboxylic Acid
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100904
Time 15.59
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg30
TD 32768
SOLVENT CDC13
NS 16
DS 2
SWH 5592.841 Hz
FIDRES 0.170680 Hz
AQ 2.9295092 sec
RG 406.4
DW 89.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.50000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 9.20 usec
PL1 -2.00 dB
SFO1 400.1464009 MHz

F2 - Processing parameters
SI 32768
SF 400.1440087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

12 11 10 9 8 7 6 5 4 3 2 1 ppm

0.87

0.90
1.86
1.88
1.13
2.00
3.24

182.93

Table 2, entry 4

43.14

28.95

25.88

25.51

Current Data Parameters
NAME Cyclohexanecarboxylic Acid
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100904
Time 19.22
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.50 usec
TE 300.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 0 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -2.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677334 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

S16

11.79

Table 2, entry 5

1.23

Current Data Parameters
NAME Pivalic Acid
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110218
Time 18.11
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 3906.250 Hz
FIDRES 0.059605 Hz
AQ 8.3886576 sec
RG 32
DW 128.000 usec
DE 6.50 usec
TE 300.2 K
D1 1.50000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.20 usec
PL1 0 dB
SFO1 300.1326507 MHz

F2 - Processing parameters
SI 32768
SF 300.1310086 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1.03

9.00

185.98

Table 2, entry 5

38.82

27.16

Current Data Parameters
NAME Pivalic Acid
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110218
Time 18.19
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 17
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 32768
DW 27.800 usec
DE 6.50 usec
TE 300.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 0 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -2.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4679825 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Table 2, entry 6

Current Data Parameters
NAME 4-Methoxyphenylacetic Acid
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100917
Time 18.57
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 4496.403 Hz
FIDRES 0.137219 Hz
AQ 3.6438515 sec
RG 71.8
DW 111.200 usec
DE 6.00 usec
TE 300.2 K
D1 1.50000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.20 usec
PL1 0 dB
SFO1 300.1318008 MHz

F2 - Processing parameters
SI 32768
SF 300.1300060 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Current Data Parameters
NAME 4-Methoxyphenylacetic Acid
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100917
Time 19.22
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 100
DS 4
SWH 17985.611 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 114
DW 27.800 usec
DE 6.50 usec
TE 300.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 7.50 usec
PL1 0 dB
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -2.00 dB
PL12 15.00 dB
PL13 15.00 dB
SFO2 300.1312005 MHz

F2 - Processing parameters
SI 32768
SF 75.4677371 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Table 2, entry 6

Table 2, entry 7

Current Data Parameters
NAME NBB842011_9decanoicacid
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20110804
Time 13.46
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 74
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912244 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 300.2 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 6.25 usec
PL1 0 dB
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -1.00 dB
PL12 13.76 dB
PL13 70.00 dB
SFO2 500.1320005 MHz

F2 - Processing parameters
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Table 2, entry 7

Table 2, entry 8

NAME NBB852011_11bromoundecanoicacid2
EXPNO 1
PROCNO 1
Date_ 20110805
Time 13.44
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860696 sec
RG 35.9
DW 48.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 -1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

180.11

Table 2, entry 8

34.07
33.98
32.81
29.33
29.27
29.16
29.01
28.71
28.14
24.65

NAME NBB852011_11bromoundecanoicacid2
EXPNO 2
PROCNO 1
Date_ 20110805
Time 13.47
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 25
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 300.2 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 6.25 usec
PL1 0.00 dB
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -1.00 dB
PL12 13.76 dB
PL13 70.00 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Table 2, entry 9

NAME NBB852011_decanedioicacid
EXPNO 1
PROCNO 1
Date_ 20110805
Time 13.36
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT DMSO
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860696 sec
RG 90.5
DW 48.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 -1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

NAME NBB852011_decanedioicacid
EXPNO 2
PROCNO 1
Date_ 20110805
Time 13.39
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 17
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 32768
DW 16.650 usec
DE 6.50 usec
TE 300.2 K
D1 1.50000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 6.25 usec
PL1 0.00 dB
SFO1 125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 70.00 usec
PL2 -1.00 dB
PL12 13.76 dB
PL13 70.00 dB
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

Table 2, entry 9

NAME NBB852011_3pentynoicacid
EXPNO 1
PROCNO 1
Date_ 20110805
Time 11.12
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.315264 Hz
AQ 1.5860696 sec
RG 71.8
DW 48.400 usec
DE 6.00 usec
TE 300.2 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 -1.00 dB
SFO1 500.1330885 MHz
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Table 2, entry 10

Table 2, entry 10

S28

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NAME      NBB852011_3pentynoicacid
EXPNO     2
PROCNO    1
Date_     20110805
Time      11.16
INSTRUM    spect
PROBHD     5 mm BBO BB-1H
PULPROG    zgpg30
TD         65536
SOLVENT    CDC13
NS         18
DS         4
SWH        30030.029 Hz
FIDRES     0.458222 Hz
AQ         1.0912410 sec
RG         32768
DW         16.650 usec
DE         6.50 usec
TE         300.2 K
D1         1.50000000 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         6.25 usec
PL1        0.00 dB
SFO1       125.7703643 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      70.00 usec
PL2        -1.00 dB
PL12       13.76 dB
PL13       70.00 dB
SFO2       500.1320005 MHz
SI         32768
SF         125.7577890 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.00
  
```