Supporting Information

Alkylation of H-Phosphinate Esters under Basic Conditions

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LIST OF CONTENTS:

	PAGE
General Chemistry	S2
Preparation of H-phosphinate Ester Starting Materials	S2
Spectral Data	S2-S14
Table 2, entry 1	S2
Table 3, entries 1-15c	S3-S10
Scheme 1	S10
Equation 5	S10
Scheme 2	S10-S12
Scheme 3	S12-S13
Scheme 4	S14
References	S15
Spectra	S16-98

General. All reactions were conducted in oven- or flame-dried glassware, under nitrogen. Organic solutions of products were dried over MgSO_4 , and filtered. Lithium hexamethyldisilazide (LiHMDS , 1 M in THF) and butyllithium (1.6 M in hexanes) were purchased and used as received. Tetrahydrofuran (THF) was distilled under N_2 from sodium benzophenone ketyl, and used immediately. Chemical shifts for ^1H NMR spectra are reported (in parts per million) relative to internal tetramethylsilane (Me_4Si , $\delta = 0.00$ ppm) with CDCl_3 as solvent. ^{13}C NMR spectra were recorded at 75 MHz. Chemical shifts for ^{13}C NMR spectra are reported (in parts per million) relative to CDCl_3 ($\delta = 77.0$ ppm). ^{31}P NMR at 121 MHz and/or at 36 MHz, and chemical shifts reported (in parts per million) relative to external 85% phosphoric acid ($\delta = 0.0$ ppm). All ^{31}P -NMR spectra are reported as ^1H -decoupled. Radial chromatography was carried out using 1, 2, or 4 mm layers of silica gel 60 PF_{254} containing gypsum. Silica gel (200-300 mesh) was used for flash chromatography. Ethyl acetate/hexanes mixtures were used as the eluent for chromatographic purifications. TLC plates were visualized by immersion in anisaldehyde stain (by volume: 93% ethanol, 3.5% sulfuric acid, 1% acetic acid, and 2.5% anisaldehyde) followed by heating.

Preparation of H-Phosphinate Esters Starting Materials. The starting materials used in this studies were prepared using our previously described methodologies.¹

Methyl-phenyl-phosphinic acid ethyl ester (Table 2, entry 1)²

Yield: 98 %; RN : [51103-74-3]. ^1H NMR (CDCl_3 , 300 MHz) δ 1.29 (t, $J = 7.0$ Hz, 3 H), 1.67 (d, $J_{\text{HP}} = 14.4$ Hz, 3 H), 4.83 & 4.08 (m, 2 H), 7.45-7.60 (m, 3 H), 7.76-7.85 (m, 2H).

Allyl-(1,1-dieethoxy-ethyl)-phosphinic acid ethyl ester (Table 3, entry 1a)^{1b}

Yield: 68 %, RN : [173781-44-7]. ¹H NMR (CDCl₃, 300 MHz) δ 1.21 (t, J = 7.0 Hz, 6 H), 1.31 (t, J = 7.0 Hz, 3 H), 1.52 (d, J_{HP} = 11.4 Hz, 3 H), 2.65 (dqt, J_{HP} = 14.6 Hz, J = 7.9 Hz, 2 H), 3.61-3.81 (m, 4 H), 4.09-4.27 (m, 2 H), 5.18-5.26 (m, 2 H), 5.80 (m, 1 H). ³¹P NMR (CDCl₃, 121.47 MHz) δ 46.04 (s).

(1,1-Dieethoxy-ethyl)-pent-4-enyl-phosphinic acid ethyl ester (Table 3, entry 1b)

Yield: 66 %. ¹H NMR (CDCl₃, 300 MHz) δ 1.21 (t, J = 7.0 Hz, 6 H), 1.32 (t, J = 7.0 Hz, 3 H), 1.49 (d, J_{HP} = 11.1 Hz, 3 H), 1.69-1.79 (m, 4 H), 2.15 (dm, J_{HP} = 11.1 Hz, 2 H), 3.71 (m, 4 H), 4.21 (tdd, J = 6.4 Hz, J = 10.2 Hz, 2 H), 5.18-5.26 (m, 2 H), 5.80 (m, 1 H). ¹³C {¹H} NMR (CDCl₃, 75.45 MHz) δ 15.5, 15.7, 16.9 (d, J_{POCC} = 5.5 Hz), 20.4 (d, J_{PCC} = 4.9 Hz), 20.7 (d, J_{PCC} = 12.7 Hz), 25.0 (d, J_{PC} = 87 Hz), 34.5, 35.0, 57.8 (d, J_{PCC} = 7.2 Hz), 58.3 (d, J_{PCC} = 4.6 Hz), 61.8 (d, J_{PCC} = 6.9 Hz), 101.4 (d, J_{PCC} = 137.6 Hz), 115.7, 137.8. ³¹P NMR (CDCl₃, 121.47 MHz) δ 50.68 (s). HRMS (CI, M⁺H): calc. for C₁₃H₂₇O₄P 279.1725, obs. 279.1718.

4-[(1,1-Dieethoxy-ethyl)-ethoxy-phosphinoyl]-butyric acid ethyl ester (Table 3, entry 2)

Yield: 62 %. ¹H NMR (CDCl₃, 300 MHz) δ 1.20 & 1.21 (2 $\times$ t, J = 7.3 Hz, 6 H), 1.25 (t, J = 7.0 Hz, 3 H), 1.32 (t, J = 7.0 Hz, 3 H), 1.49 (d, J_{HP} = 11.1 Hz, 3 H), 1.70-2.09 (m, 4 H), 2.42 & 2.44 (2 $\times$ d, J_{HP} = 7.0 Hz, 2 H), 3.59-3.81 (m, 4 H), 4.25 (qt, J = 7.0 Hz, 2 H), 4.13-4.29 (m, 2 H). ¹³C {¹H} NMR (CDCl₃, 75.45 MHz) δ 13.2, 14.2, 14.5, 15.6 (d, J_{POCC} = 5.5 Hz), 15.8 (d, J_{POCC} = 4.6 Hz), 19.4 (d, J_{POCC} = 12.4 Hz), 23.1 (d, J_{PC} = 86.6 Hz), 33.8 (d, J_{PCC} = 13.8 Hz), 56.6 (d, J_{POC} = 7.1 Hz), 57.1 (d, J_{POC} = 4.6 Hz), 59.3, 60.7 (d, J_{POC} =

5.8 Hz), 100.0 (d, $J_{\text{PC}} = 139.4$ Hz). ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 49.34 (s). HRMS (M^+H ion by direct probe) : calc. for $\text{C}_{14}\text{H}_{30}\text{O}_6\text{P}$ 325.1780, obs. 325.1774.

(1,1-Diethoxy-ethyl)-(1,3-dioxo-1,3-dihydro-isoindol-2-ylmethyl)phosphinic acid ethyl ester (Table 3, entry 3)

Yield: 70 %. ^1H NMR (CDCl_3 , 300 MHz) δ 1.20 & 1.21 (2xt, $J = 7.0$ Hz, 6 H), 1.34 (t, $J = 7.0$ Hz, 3 H), 1.62 (d, $J_{\text{HP}} = 11.1$ Hz, 3 H), 3.59-3.81 (m, 4 H), 4.07 (dt, $J_{\text{HP}} = 15.5$ Hz, $J = 7.0$ Hz, 2 H), 4.13-4.36 (m, 2 H), 7.69-7.76 & 7.84-7.90 (2xm, 2x2 H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) δ 15.5 (d, $J_{\text{POCC}} = 13$ Hz), 16.8 (d, $J_{\text{PCC}} = 5.2$ Hz), 20.1 (d, $J_{\text{POCC}} = 13$ Hz), 34.6 (d, $J_{\text{PCC}} = 87.2$ Hz), 58.2 (d, $J_{\text{POC}} = 7.5$ Hz), 58.7 (d, $J_{\text{POC}} = 5.5$ Hz), 62.6 (d, $J_{\text{POC}} = 7.2$ Hz), 101.2 (d, $J_{\text{PC}} = 141.3$ Hz), 123.6, 132.3, 134.2, 167.4. ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 38.74 (s). HRMS (MW + Na by Electrospray) : calc. for $\text{C}_{17}\text{H}_{24}\text{O}_6\text{PNa}$ 392.1239, obs. 392.1232.

2-[(1,1-Diethoxy-ethyl)-ethoxy-phosphinoylethyl]-acrylic acid ethyl ester (Table 3, entry 4)

Yield: 48 %. ^1H NMR (CDCl_3 , 300 MHz) δ 1.20 & 1.21 (2xt, $J = 7.3$ Hz, 6 H), 1.27 (t, $J = 7.0$ Hz, 3 H), 1.31 (t, $J = 7.0$ Hz, 3 H), 1.53 (d, $J_{\text{HP}} = 11.4$ Hz, 3 H), 2.98 (d, $J_{\text{HP}} = 14.9$ Hz, 2 H), 3.60-3.81 (m, 4 H), 4.09-4.26 (m, 4 H), 5.88 (d, $J = 4.7$ Hz, 1 H), 6.35 (d, $J = 4.7$ Hz, 1 H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) δ 14.3, 15.5 (d, $J_{\text{POCC}} = 21.3$ Hz), 16.8 (d, $J_{\text{PCC}} = 5.2$ Hz), 20.6 (d, $J_{\text{POCC}} = 12.7$ Hz), 28.6 (d, $J_{\text{PC}} = 81.7$ Hz), 57.8 (d, $J_{\text{POC}} = 7.2$ Hz), 58.3 (d, $J_{\text{POC}} = 4.9$ Hz), 61.3, 62.1 (d, $J_{\text{POC}} = 7.2$ Hz), 101.0 (d, $J_{\text{PC}} = 141$ Hz), 128.8 (d, $J_{\text{PCCC}} = 9.8$ Hz), 131.1 (d, $J_{\text{PCC}} = 8.0$ Hz), 166.6 (d, $J_{\text{PC}} = 10$ Hz). ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 44.81 (s). HRMS (MW + Na by Electrospray) : calc. for $\text{C}_{14}\text{H}_{27}\text{O}_6\text{PNa}$ 345.1443, obs. 345.1435.

2,2-Dimethyl-propionic acid (1,1-diethoxy-ethyl)-ethoxyphosphinoyl methyl ester (Table 3, entry 5a)

Yield: 50 %. ^1H NMR (CDCl_3 , 300 MHz) δ 1.21 (t, $J = 7.0$ Hz, 6 H), 1.23 (s, 9 H), 1.34 (t, $J = 7.0$ Hz, 3 H), 1.54 (d, $J_{\text{HP}} = 11.4$ Hz, 3 H), 3.65-3.81 (m, 4 H), 4.25 (dqt, $J_{\text{HP}} = 2.6$ Hz, $J = 7.0$ Hz, 2 H), 4.40 & 4.53 (ABXsyst, $J_{\text{AB}} = 14.4$ Hz, $J_{\text{BX}} = 5.9$ Hz, $J_{\text{AX}} = 4.1$ Hz, 2 H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) δ 15.6 (d, $J_{\text{POCC}} = 11.2$ Hz), 16.8 (d, $J_{\text{PCC}} = 5.2$ Hz), 20.6 (d, $J_{\text{POCC}} = 12.1$ Hz), 27.3, 57.1, 58.0 (d, $J_{\text{POC}} = 7.5$ Hz), 58.5 (d, $J_{\text{POC}} = 4.9$ Hz), 101.0 (d, $J_{\text{PC}} = 146$ Hz). ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 39.01 (s). HRMS (MW + Na by Electrospray) : calc. for $\text{C}_{14}\text{H}_{29}\text{O}_6\text{PNa}$ 347.1599, obs. 347.1592.

Benzyloxymethyl-(1,1-diethoxy-ethyl)-phosphinic acid ethyl ester (Table 3, entry 5b)

Yield: 70 %. ^1H NMR (CDCl_3 , 300 MHz) δ 1.18 (t, $J = 7.0$ Hz, 6 H), 1.34 (t, $J = 7.0$ Hz, 3 H), 1.56 (d, $J_{\text{HP}} = 11.4$ Hz, 3 H), 3.60-3.78 (m, 4 H), 3.84 (dd, $J_{\text{HP}} = 5.9$ Hz, $J = 2.0$ Hz, 2 H), 4.25 (qt, $J = 7.0$ Hz, 2 H), 4.65 (s, 2 H), 7.29-7.38 (m, 5 H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) δ 15.6 (d, $J_{\text{POCC}} = 14.7$ Hz), 16.8 (d, $J_{\text{PCC}} = 5.2$ Hz), 20.8 (d, $J_{\text{POCC}} = 11.5$ Hz), 58.0 (d, $J_{\text{POC}} = 7.2$ Hz), 58.4 (d, $J_{\text{POC}} = 5.5$ Hz), 62.2 (d, $J_{\text{POC}} = 7.2$ Hz), 64.7 (d, $J_{\text{PC}} = 102$ Hz), 75.4 (d, $J_{\text{PCOC}} = 10.4$ Hz), 101.2 (d, $J_{\text{PC}} = 140$ Hz), 128.2, 128.3, 128.6, 137.3. ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 41.42 (s). HRMS (MW + Na by Electrospray) : calc. for $\text{C}_{16}\text{H}_{27}\text{O}_5\text{PNa}$ 353.1494, obs. 353.1502.

(1,1-Diethoxy-ethyl)-pyridin-4-ylmethyl-phosphinic acid ethyl ester (Table 3, entry 6a)

Yield: 48 %. ^1H NMR (CDCl_3 , 300 MHz) δ 1.12-1.29 (m, 9 H), 1.49 (d, $J_{\text{HP}} = 11.4$ Hz, 3 H), 3.12 & 3.21 (ABXsyst, $J_{\text{AB}} = 14.6$ Hz, $J_{\text{BX}} = 12.6$ Hz, $J_{\text{AX}} = 11.1$ Hz, 2 H), 3.59-3.81 (m, 4 H), 4.08 (qt, $J = 7.3$ Hz, 2 H), 7.27-7.29 (m, 2 H), 8.53 (d, $J = 4.7$ Hz, 2 H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) δ 15.4 (d, J_{POCC}

= 20.7 Hz), 16.5 (d, $J_{\text{PCC}} = 5.2$ Hz), 20.6 (d, $J_{\text{POCC}} = 12.4$ Hz), 32.4 (d, $J_{\text{PC}} = 78$ Hz), 57.7 (d, $J_{\text{POC}} = 7.5$ Hz), 58.5 (d, $J_{\text{POC}} = 4.6$ Hz), 62.2 (d, $J_{\text{POC}} = 7.2$ Hz), 101.5 (d, $J_{\text{PC}} = 142$ Hz), 125.5 (d, $J_{\text{PCC}} = 5.2$ Hz), 141.3 (d, $J_{\text{PCCC}} = 8.6$ Hz), 148.3. ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 43.85 (s). HRMS (M^+H ion by direct probe) : calc. for $\text{C}_{14}\text{H}_{25}\text{O}_4\text{P}$ 302.1521, obs. 302.1518.

(1,1-Diethoxy-ethyl)-pyridin-2-ylmethyl-phosphinic acid ethyl ester (Table 3, entry 6c)

Yield: 62 %. ^1H NMR (CDCl_3 , 300 MHz) δ 1.14 (t, $J = 7.0$ Hz, 6 H), 1.19 (t, $J = 7.0$ Hz, 3 H), 1.54 (d, $J_{\text{HP}} = 11.7$ Hz, 3 H), 3.40 & 3.48 (ABXsyst, $J_{\text{AB}} = 15.8$ Hz, $J_{\text{BX}} = 14.3$ Hz, $J_{\text{AX}} = 14.0$ Hz, 2 H), 3.58-3.87 (m, 4 H), 4.08 (dq, $J_{\text{HP}} = 0.9$ Hz, $J = 7.0$ Hz, 2 H), 7.18 (~t, $J = 6.1$ Hz, 1 H), 7.34 (~d, $J = 7.9$ Hz, 1 H), 7.63 (dt, $J = 7.6$ Hz, $J = 1.7$ Hz, 1 H), 8.53 (~d, $J = 4.5$ Hz, 1 H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) δ 15.6 (d, $J_{\text{POCC}} = 20.7$ Hz), 16.7 (d, $J_{\text{PCC}} = 5.2$ Hz), 20.6 (d, $J_{\text{POCC}} = 12.4$ Hz), 30.0 (d, $J_{\text{PC}} = 78$ Hz), 57.9 (d, $J_{\text{POC}} = 7.8$ Hz), 58.7 (d, $J_{\text{POC}} = 4.6$ Hz), 62.3 (d, $J_{\text{POC}} = 6.9$ Hz), 101.5 (d, $J_{\text{PC}} = 142$ Hz), 122.1 (d, $J_{\text{PCCNC}} = 3.2$ Hz), 124.7 (d, $J_{\text{PCCCC}} = 4.9$ Hz), 137.0 (d, $J_{\text{PCCC}} = 2.3$ Hz), 149.7 (d, $J_{\text{PCCC}} = 2.6$ Hz), 153.4 (d, $J_{\text{PCC}} = 6.9$ Hz). ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 44.09 (s). HRMS (M^+H ion by direct probe) : calc. for $\text{C}_{14}\text{H}_{25}\text{O}_4\text{P}$ 302.1521, obs. 302.1520.

(2-Benzyloxycarbonylamino-ethyl)-(1,1-diethoxy-ethyl)-phosphinic acid ethyl ester (Table 3, entry 7)

Yield: 58 %. ^1H NMR (CDCl_3 , 300 MHz) δ 1.21 (t, $J = 7.0$ Hz, 6 H), 1.34 (t, $J = 7.0$ Hz, 3 H), 1.62 (d, $J_{\text{HP}} = 11.4$ Hz, 3 H), 1.89-2.14 (m, 2 H), 3.59-3.81 (m, 6 H), 4.11-4.27 (m, 2 H), 5.09 (s, 2 H), 5.68 (brs, 1 H), 7.27-7.35 (m, 5 H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) δ 15.6 (d, $J_{\text{POCC}} = 15$ Hz), 16.9 (d, $J_{\text{PCC}} = 5.2$ Hz), 20.2 (d, $J_{\text{POCC}} = 12.4$ Hz), 26.4 (d, $J_{\text{PC}} = 84.3$ Hz), 34.9, 58.0 (d, $J_{\text{POC}} = 7.2$ Hz), 58.6 (d, $J_{\text{POC}} = 4.9$ Hz), 61.9 (d, $J_{\text{POC}} = 6.9$ Hz), 101.2 (d, $J_{\text{PC}} =$

141.3 Hz), 128.2, 128.7, 136.8, 156.5. ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 49.52 (s). HRMS (M^+H ion by direct probe) : calc. for $\text{C}_{18}\text{H}_{31}\text{O}_6\text{PN}$ 388.1889, obs. 388.1893.

Difluoromethyl-octyl-phosphinic acid butyl ester (Table 3, entry 10)

Yield: 78 %. ^1H NMR (CDCl_3 , 300 MHz) : δ 0.88 (t, $J = 7.0$ Hz, 3H), 0.94 (t, $J = 7.2$ Hz, 3H), 1.25-1.47 (m, 10H), 1.61-1.90 (m, 8H), 4.05-4.27 (m, 2H), 5.90 (dt, $J_{\text{HF}} = 23.2$ Hz, $J_{\text{HF}} = 48.9$ Hz, 1H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) : $\delta = 13.7, 14.3, 18.8, 20.5$ (d, $J_{\text{PCCCC}} = 4.9$ Hz), 23.8, 24.5 (d, $J_{\text{PC}} = 96$ Hz), 29.1, 29.2, 30.9 (d, $J_{\text{PCCC}} = 14.9$ Hz), 31.9, 32.7 (d, $J_{\text{POCC}} = 5.2$ Hz), 66.5 (d, $J_{\text{POC}} = 7.2$ Hz), 113.2 (dd, $J_{\text{PCC}} = 129$ Hz). ^{31}P NMR (CDCl_3 , 121.47 MHz) : $\delta = 39.81$ (t, $J_{\text{PF}} = 77.1$ Hz). ^{19}F NMR (CDCl_3 , 282.30 MHz) δ -135.31 (ddd, $J_{\text{FH}} = 23.2$ Hz, $J_{\text{FP}} = 77.1$ Hz, $J_{\text{FF}} = 49.5$ Hz). HRMS (MW by direct probe EI) : calc. for $\text{C}_{13}\text{H}_{27}\text{O}_2\text{PF}_2$ 284.1717, obs. 284.1719.

Isopropyl-octyl-phosphinic acid butyl ester (Table 3, entry 11)

Yield: 73 %. ^1H NMR (CDCl_3 , 300 MHz) δ 0.88 (t, $J = 6.7$ Hz, 3 H), 0.93 (t, $J = 7.3$ Hz, 3 H), 1.14 and 1.19 (2xd, $J = 7.0$ Hz, 2x3 H), 1.27-1.46 (m, 12 H), 1.55-1.73 (m, 6 H), 1.92 (sept, $J = 7.0$ Hz, 1 H), 4.98 (qd, $J = 6.7$ Hz, 2 H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) δ 13.9, 14.3, 15.8 & 15.9 (2xd, $J_{\text{PCC}} = 2.6$ Hz), 19.1, 21.9 (d, $J_{\text{POCC}} = 4.3$ Hz), 22.8, 25.5 (d, $J_{\text{PC}} = 87$ Hz), 25.5 (d, $J_{\text{PC}} = 92$ Hz), 29.3 (d, $J_{\text{PCC}} = 3.2$ Hz), 31.2 (d, $J_{\text{POC}} = 14.7$ Hz), 32.0, 33.1 (d, $J_{\text{PCC}} = 5.5$ Hz), 64.1 (d, $J_{\text{POC}} = 6.9$ Hz). ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 62.13 (s). HRMS (EI^+) : calc. for $\text{C}_{15}\text{H}_{33}\text{O}_2\text{P}$ 276.2218, obs. 276.2217.

Pent-4-enyl-(1-propyl-pent-1-enyl)-phosphinic acid butyl ester (Table 3, entry 12)

Yield: 66 %. ^1H NMR (CDCl_3 , 300 MHz) δ 0.93 & 0.95 (2xt, $J = 7.3$ Hz, 6 H), 1.29 (t, $J = 7.0$ Hz, 3 H), 1.36-1.77 (m, 8 H), 2.05-2.22 (m, 6 H), 3.98 &

4.11 (tdd, $J_{\text{HP}} = 6.5$ Hz, $J = 10$ Hz, $J = 8.5$ Hz, 2 H), 5.00-5.06 (m, 2 H), 5.74 (dq, $J = 6.7$ Hz, $J = 10.3$ Hz, 1 H), 6.54 (td, $J = 7.3$ Hz, $J = 20.8$ Hz, 1 H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) δ 14.1, 14.5, 16.6 (d, $J_{\text{POCC}} = 6.3$ Hz), 21.0 (d, $J_{\text{PCCCC}} = 2.9$ Hz), 21.2 (d, $J_{\text{PCCCC}} = 1.6$ Hz), 23.0, 27.2 (d, $J_{\text{PC}} = 97.5$ Hz), 29.5 (d, $J_{\text{PCCC}} = 12.0$ Hz), 31.0 (d, $J_{\text{PCCC}} = 15.1$ Hz), 34.7 (d, $J_{\text{PCCC}} = 16.1$ Hz), 60.4 (d, $J_{\text{POC}} = 6.3$ Hz), 115.9, 129.6 (d, $J_{\text{PC}} = 149.8$ Hz), 131.7, 148.4 (d, $J_{\text{PCC}} = 8.5$ Hz). ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 49.88 (s). HRMS (EI^+) : calc. for $\text{C}_{15}\text{H}_{29}\text{O}_2\text{P}$ 272.1905, obs. 272.1908.

Allyl-methyl-phosphinic acid butyl ester (Table 3, entry 13)⁴

Yield: 95 %; RN : [69695-69-8]. ^1H NMR (CDCl_3 , 300 MHz) δ 0.93 (t, $J = 7.3$ Hz, 3 H), 1.40 (sext, $J = 7.3$ Hz, 2 H), 1.50 (d, $J_{\text{HP}} = 14.1$ Hz, 3 H), 1.63 (sext, $J = 6.7$ Hz, 2 H), 2.61-2.73 (m, 2 H), 4.04 (qd, $J = 6.7$ Hz, 2 H), 5.20-5.27 (m, 2 H), 5.84 (m, 1 H). ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 53.58 (s).

Difluoromethyl-(3,7,11-trimethyl-dodeca-2,6,10-trienyl)-phosphinic acid butyl ester (Table 3, entry 14)

Yield: 72 %. ^1H NMR (CDCl_3 , 300 MHz) : δ 0.94 (t, $J = 7.3$ Hz, 3H), 1.51 (sex, $J = 7.3$ Hz, 2H), 1.59 (s, 3H), 1.63-1.75 (m, 12H), 1.94-2.13 (m, 7H), 2.55-2.66 (dd, $J_{\text{HP}} = 18.2$ Hz, $J = 7.9$ Hz, 2H), 4.09-4.24 (m, 2H), 5.05-5.16 (m, 3H), 7.04 (dt, $J_{\text{HF}} = 24.6$ Hz, $J_{\text{HF}} = 48.9$ Hz, 1H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) : $\delta = 13.8, 16.2, 16.8$ (d, $J_{\text{PCCCC}} = 3.2$ Hz), 17.9, 19.0, 25.9, 26.6 (d, $J_{\text{PCCCC}} = 3.7$ Hz), 26.9, 30.2 (d, $J_{\text{PC}} = 91.6$ Hz), 32.6 (d, $J_{\text{POCC}} = 6$ Hz), 39.8 (m), 39.9, 66.6 (d, $J_{\text{POC}} = 7.2$ Hz), 110.6 (d, $J_{\text{PCC}} = 9.2$ Hz), 123.9, 124.5, 131.6, 135.7, 142.2 (d, $J_{\text{PCCC}} = 14.0$ Hz). ^{31}P NMR (CDCl_3 , 121.47 MHz) : $\delta = 38.36$ (t, $J_{\text{PH}} = 76.7$ Hz). ^{19}F NMR (CDCl_3 , 282.30 MHz) δ -135.31 (ddd, $J_{\text{FH}} = 15.8$ Hz, $J_{\text{FP}} = 75.3$ Hz, $J = 49.5$ Hz). HRMS (MW by direct probe EI) : calc. for $\text{C}_{20}\text{H}_{35}\text{O}_2\text{PF}_2$ 376.2343, obs. 376.2341.

Methyl-pyridin-4-ylmethyl-phosphinic acid butyl ester (Table 3, entry 15a)

Yield: 50 %. ^1H NMR (CDCl_3 , 300 MHz) δ 0.91 (t, $J = 7.3$ Hz, 3 H), 1.36 (sext, $J = 7.3$ Hz, 2 H), 1.43 (d, $J_{\text{HP}} = 13.8$ Hz, 3 H), 1.61 (sext, $J = 7.3$ Hz, 2 H), 3.15 (d, $J_{\text{HP}} = 17.6$ Hz, 2 H), 3.95 (dq, $J_{\text{HP}} = 2.3$ Hz, $J = 7.3$ Hz, 2 H), 7.26 (m, 2 H), 8.56 (d, $J = 5.9$ Hz, 2 H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) δ 13.6 (d, $J_{\text{PC}} = 95.3$ Hz), 13.6, 18.8, 32.6 (d, $J_{\text{PCC}} = 5.7$ Hz), 37.7 (d, $J_{\text{PC}} = 86.4$ Hz), 64.6 (d, $J_{\text{POC}} = 6.9$ Hz), 125.0 (d, $J_{\text{PCCCC}} = 5.2$ Hz), 142.0 (d, $J_{\text{PCC}} = 7.2$ Hz), 149.5 (d, $J_{\text{PCCC}} = 2.6$ Hz). ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 49.48 (s). HRMS (EI^+) : calc. for $\text{C}_{11}\text{H}_{19}\text{O}_2\text{P}$ 227.1075, obs. 227.1071.

Methyl-pyridin-3-ylmethyl-phosphinic acid butyl ester (Table 3, entry 15b)

Yield: 62 %. ^1H NMR (CDCl_3 , 300 MHz) δ 0.91 (t, $J = 7.3$ Hz, 3 H), 1.34 (sext, $J = 7.3$ Hz, 2 H), 1.41 (d, $J_{\text{HP}} = 13.8$ Hz, 3 H), 1.62 (sext, $J = 7.3$ Hz, 2 H), 3.13 (d, $J_{\text{HP}} = 17.0$ Hz, 2 H), 4.00 (dq, $J_{\text{HP}} = 0.9$ Hz, $J = 7.3$ Hz, 2 H), 7.25 (m, 1 H), 7.69 (~dd, $J = 7.9$ Hz, $J = 1.8$ Hz, 1 H), 8.49-8.53 (m, 2 H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) δ 13.6 (d, $J_{\text{PC}} = 94.7$ Hz), 13.8, 18.9, 32.8 (d, $J_{\text{PCC}} = 5.7$ Hz), 35.3 (d, $J_{\text{PC}} = 85.7$ Hz), 64.7 (d, $J_{\text{POC}} = 6.9$ Hz), 123.8, 128.4 (d, $J_{\text{PCC}} = 6.9$ Hz), 137.4 (d, $J_{\text{PCCCC}} = 5.2$ Hz), 148.5 (d, $J_{\text{PCCC}} = 3.1$ Hz), 150.6 (d, $J_{\text{PCCCC}} = 6.6$ Hz). ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 50.36 (s). HRMS (EI^+) : calc. for $\text{C}_{11}\text{H}_{18}\text{O}_2\text{P}$ 227.1075, obs. 227.1075.

Methyl-pyridin-2-ylmethyl-phosphinic acid butyl ester (Table 3, entry 15c)

Yield: 68 %. ^1H NMR (CDCl_3 , 300 MHz) δ 0.91 (t, $J = 7.3$ Hz, 3 H), 1.34 (sext, $J = 7.3$ Hz, 2 H), 1.48 (d, $J_{\text{HP}} = 11.4$ Hz, 3 H), 1.63 (sext, $J = 7.3$ Hz, 2 H), 3.39 (dd, $J_{\text{HP}} = 17.9$ Hz, $J = 1.5$ Hz, 2 H), 4.00 (dq, $J_{\text{HP}} = 1.2$ Hz, $J = 7.3$ Hz, 2 H), 7.18 (~t, $J = 7.3$ Hz, 1 H), 7.34 (~d, $J = 6.5$ Hz, 1 H), 7.66 (dt, $J =$

7.3 Hz, $J = 1.5$ Hz, 1 H), 8.53 (~d, $J = 5.0$ Hz, 1 H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) δ 13.8, 13.9 (d, $J_{\text{PC}} = 95.3$ Hz), 18.9, 32.8 (d, $J_{\text{PCC}} = 6.4$ Hz), 40.8 (d, $J_{\text{PC}} = 85.5$ Hz), 64.5 (d, $J_{\text{POC}} = 6.6$ Hz), 122.1 (d, $J_{\text{PCCNC}} = 3.2$ Hz), 124.7 (d, $J_{\text{PCCCC}} = 4.9$ Hz), 137.0 (d, $J_{\text{PCCC}} = 2.3$ Hz), 149.7 (d, $J_{\text{PCCC}} = 2.6$ Hz), 153.4 (d, $J_{\text{PCC}} = 6.9$ Hz). ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 51.43 (s). HRMS (M^+H ion by direct probe) : calc. for $\text{C}_{11}\text{H}_{19}\text{O}_2\text{P}$ 228.1153, obs. 228.1155.

(2-Hydroxy-cyclohexyl)-octyl-phosphinic acid butyl ester 1 (Scheme 1)

Yield: 82 %. ^1H NMR (CDCl_3 , 300 MHz) δ 0.88 (t, $J = 6.7$ Hz, 3 H), 0.95 (dt, $J = 7.3$ Hz, $J_{\text{HP}} = 2.3$ Hz, 3 H), 1.14-1.45 (m, 16 H), 1.55-1.90 (m, 11 H), 2.05 (m, 1H), 3.37 (m, 1 H), 4.00-4.36 (m, 2 H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) δ 13.8, 14.3, 19.1, 21.4 (d, $J_{\text{POCC}} = 4.7$ Hz), 21.7 (d, $J_{\text{POCC}} = 3.8$ Hz), 23.8, 24.4 (d, $J = 7.8$ Hz), 24.8, 25.3, 25.6, 25.7, 25.8, 25.9, 26.0, 28.6 (d, $J_{\text{PC}} = 93$ Hz), 31.1 (d, $J_{\text{PCCC}} = 10.1$ Hz), 31.2 (d, $J_{\text{PCCC}} = 10.1$ Hz), 32.0, 33.0 (d, $J_{\text{PCC}} = 5.2$ Hz), 33.1 (d, $J_{\text{PCC}} = 5.2$ Hz), 35.1, 35.3, 44.4 (d, $J_{\text{PC}} = 85$ Hz), 44.5 (d, $J_{\text{PC}} = 85$ Hz), 64.2 (d, $J_{\text{POC}} = 6.6$ Hz), 64.8 (d, $J_{\text{POC}} = 6.9$ Hz), 69.8 (d, $J_{\text{POC}} = 6.9$ Hz), 70.2 (d, $J_{\text{POC}} = 6.6$ Hz). ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 62.46 & 61.92 (s). HRMS (EI^+) : calc. for $\text{C}_{19}\text{H}_{37}\text{O}_3\text{P}$ 332.2480, obs. 332.2478.

1-Butoxy-phosphinane-1-oxide 3 (Equation 5)⁵

Yield: 71 %; RN : [34299-24-6]. ^1H NMR (CDCl_3 , 300 MHz) δ 0.95 (t, $J = 7.6$ Hz, 3 H), 1.25-1.48 (m, 4 H), 1.62-1.99 (m, 10 H), 4.04 (qd, $J = 6.7$ Hz, 2 H). ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 50.67 (s).

(3-Benzyloxycarbonylamino-propyl)-H-phosphinic acid benzyl ester 4 (Scheme 2)

To a solution of $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$ (5 mmol) and the corresponding alkene (5 mmol) in methanol (10 ml) was added Et_3B (1.0 M, 2 ml, 2 mmol), at rt in a reaction vessel open to air. The solution was stirred at rt for 6h. The

reaction was concentrated a rotary evaporator, and the residue was partitioned between aqueous KHSO_4 and EtOAc. The aqueous layer was extracted twice with EtOAc, and the combined organic layers were dried and concentrated to afford the crude phosphinic acid. This was diluted with toluene (10ml), and esterified, using $(\text{BnO})_4\text{Si}$ (1eq), at reflux for 24h. The crude mixture was concentrated under vacuum and purified by column chromatography (silica, EtOAc : Hexanes (50:50)) to afford the desired product **4** (60%). ^1H NMR (CDCl_3 , 300 MHz) δ 1.81-1.82 (m, 4 H), 3.26 (qd, $J = 6.4$ Hz, 2 H), 4.99-5.29 (m, 5 H), 7.13 (d, $J_{\text{HP}} = 536$ Hz, 1 H), 7.29-7.37 (m, 10 H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) δ 22.7, 28.4 (d, $J_{\text{PC}} = 89.5$ Hz), 41.5, 65.9 (d, $J_{\text{POC}} = 7.0$ Hz), 66.9, 128.1, 128.3, 128.6, 128.7, 128.9, 136.8, 136.9, 137.1, 156.7. ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 39.04 (d, $J_{\text{PH}} = 536$ Hz).

(3-Benzoyloxycarbonylamino-propyl)-butyl-phosphinic acid benzyl ester 5 (Scheme 2)

In a dry two-neck flask, neat H-phosphinate **4** (1.056 g, 3 mmol) was placed under vacuum for 10 min before use. Anhydrous THF (10 mL) was then added under N_2 . The flask was cooled to -78°C and deoxygenated under vacuum for 5 min. The reaction flask was back-filled with nitrogen, then LiHMDS (1.0 M in THF, 3 mL, 3 mmol) was added at -78°C . After 10 min, butyl iodide (300 μL , 2.6 mmol) was added under N_2 . After addition, the temperature of the solution was slowly allowed to warm up to rt. After 2 hours at rt, the reaction mixture was quenched with a saturated solution of NH_4Cl /brine, and extracted with ethyl acetate (3 X) then dried over anhydrous MgSO_4 and concentrated in vacuo. The resulting oil was purified by column chromatography (silica, EtOAc:Hexanes 80:20) to afford the desired product **5** (76%). ^1H NMR (CDCl_3 , 300 MHz) δ 0.87 (t, J

= 7.3 Hz, 3 H), 1.34 (sext, $J = 7.3$ Hz, 2 H), 1.50 (sext, $J = 7.3$ Hz, 2 H), 1.64-1.78 (m, 6 H), 3.15-3.24 (m, 2 H), 4.99 (d, $J_{\text{HP}} = 9.3$ Hz, 2 H), 5.08 (s, 2 H), 5.27 (brs, 1 H), 7.29-7.37 (m, 10 H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) δ 13.8, 22.7, 24.1 (d, $J_{\text{PCC}} = 5.5$ Hz), 24.2 (d, $J_{\text{PCC}} = 6.3$ Hz), 25.7 (d, $J_{\text{PC}} = 90.1$ Hz), 28.4 (d, $J_{\text{PC}} = 89.5$ Hz), 41.5, 41.7, 65.9 (d, $J_{\text{POC}} = 7.0$ Hz), 66.9, 128.1, 128.3, 128.6, 128.7, 128.9, 136.8, 136.9, 137.1, 156.7. ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 59.86 (s). HRMS (EI^+) : calc. for $\text{C}_{22}\text{H}_{30}\text{O}_4\text{PN}$ 403.1912, obs. 403.1913.

(3-Amino-propyl)-butyl-phosphinic acid 6 (CGP36742, Scheme 2)⁶

Pd/C (10 %, 110 mg) then a mixture of solvent $\text{H}_2\text{O}:\text{THF}$ (1:2, 1.5 mL), were placed under N_2 , then a solution of benzyl ester **5** (0.800 g, 2 mmol) in the same solvent mixture (3.5 mL) was added. Then reaction flask was shaken in a Parr apparatus, at rt under hydrogen pressure (52 psi) for 40 h. The mixture was filtered through celite and the aqueous layer was concentrated under vacuum to give a white solid (98 %). ^1H NMR (D_2O , 300 MHz) δ 1.05 (t, $J = 5.0$ Hz, 3 H), 1.50-1.73 (m, 8 H), 1.88-2.00 (m, 2 H), 3.14 (t, $J = 7.5$ Hz, 2 H). ^{31}P NMR (D_2O , 121.47 MHz) δ 45.9 (s).

2-Benzyloxycarbonylamino-3-(ethoxy-phenyl-phosphinoyl)-propionic acid methyl ester 7 (Scheme 3)

Neat ethyl phenyl-H-phosphinate (0.510 g, 3 mmol) was placed under vacuum in a dry two-neck flask, during 10 min before use. Anhydrous THF (10 mL) was then added under N_2 . The flask was placed at -78°C and deoxygenated under vacuum for 5 min. The reaction flask was back-filled with nitrogen, then LiHMDS (1.0 M in THF, 3 mL, 3 mmol) was added at -78°C . After 10 min, the alkyl iodide (1.089 g, 3 mmol) dissolved in anhydrous THF (6 mL) was added under N_2 . After addition, the

temperature of the solution was slowly allowed to warm up to rt, then stirred overnight. The reaction mixture was then quenched with brine, and extracted with ethyl acetate (3 X), then dried over anhydrous MgSO_4 , and concentrated in vacuo. The resulting oil was purified by column chromatography (silica, EtOAc:Hexanes 80:20) to afford product **7** (62%, mixture of 2 diastereoisomers). ^1H NMR (CDCl_3 , 300 MHz) δ (2 diastereoisomers) 1.26 (t, $J = 7.0$ Hz, 3 H), 2.40-2.58 (m, 2 H), 3.41 & 3.74 (2xs, 3 H), 3.82 (m, 1 H), 4.04 (m, 1 H), 4.50 (m, 1 H), 4.98-5.12 (m, 2 H), 6.19 & 6.32 (2xd, $J = 7.0$ Hz, $J = 8.2$ Hz, 1 H), 7.27-7.58 (m, 8 H), 7.71-7.79 (m, 2 H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) δ (2 diastereoisomers) 16.4, 16.5, 31.2 (d, $J_{\text{PC}} = 89.5$ Hz), 31.4 (d, $J_{\text{PC}} = 90$ Hz), 48.8, 49.9, 52.6, 52.9, 61.1, 61.2, 67.1, 128.2, 128.3, 128.7, 128.8, 128.9, 130.0, 129.1, 130.4 (d, $J_{\text{PC}} = 128$ Hz), 130.6 (d, $J_{\text{PC}} = 128$ Hz), 131.7 (d, $J_{\text{PCC}} = 10$ Hz), 132.1 (d, $J_{\text{PCC}} = 10$ Hz), 132.9, 133.0, 136.4, 136.5, 155.9, 156.0, 170.9, 171.0, 171.5. ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 40.6 & 40.7 (2xs). HRMS (EI^+) : calc. for $\text{C}_{20}\text{H}_{24}\text{O}_6\text{P}$ 405.1341, obs. 405.1336.

2-Amino-3-(hydroxy-phenyl-phosphinoyl)-propionic acid **8** **(Scheme 3)⁷**

The above intermediate (0.287 g) was dissolved in aqueous HCl (6 N, 1.5 mL) and refluxed for 24h. After cooling to rt, the mixture was extracted 3 times with CH_2Cl_2 and the aqueous layer concentrated in vacuo. The resulting solid was dissolved in MeOH (2 mL) and propylene oxide (1 mL) was added dropwise. After 6h, the solution was concentrated in vacuo. The solid was redissolved in water and washed 3 times with CH_2Cl_2 , then concentrated in vacuo to afford 2-amino-3-(hydroxy-phenyl-phosphinoyl)-propionic acid (0.129 g, 69 %) as a solid. ^1H NMR (D_2O , 300

MHz) δ 2.3 (m, 2 H), 4.0 (m, 1 H), 7.6 (m, 5 H). ^{31}P NMR (D_2O , 121.49 MHz) δ 30.19 (s).

2-*tert*-Butoxycarbonylamino-4-(butoxy-methyl-phosphinoyl)-butyric acid methyl ester 10 (Scheme 4)

Neat H-phosphinate **9** (0.272 g, 2 mmol) was placed under vacuum in a dry two-neck flask, during 10 min before use. Anhydrous THF (7 mL) was then added under nitrogen. The flask was placed at -78°C and deoxygenated under vacuum for 5 min. The reaction flask was back-filled with nitrogen, then LiHMDS (1.0 M in THF, 2 mL, 2 mmol) was added at -78°C . After 10 min, the alkyl bromide (0.592 g, 2 mmol) dissolved in anhydrous THF (3 mL) was added under N_2 . After addition, the temperature of the solution was slowly allowed to warm up to rt in 10 min. After 5 hours at rt, the reaction mixture was quenched with brine, and extracted with ethyl acetate (3 X) then dried over anhydrous MgSO_4 and concentrated in vacuo. The resulting oil was purified by column chromatography (silica, EtOAc 100%) to afford the desired product **10** (40%). ^1H NMR (CDCl_3 , 300 MHz) δ 0.93 (t, $J = 7.3$ Hz, 3 H), 1.27-1.86 (m, 17 H), 2.18-2.37 (m, 2 H), 3.54 (d, $J_{\text{HP}} = 14.1$ Hz, 3 H), 3.60-3.81 (m, 3 H), 4.50 (m, 1 H), 5.02 (d, $J = 9.5$ Hz, 1 H). ^{13}C $\{^1\text{H}\}$ NMR (CDCl_3 , 75.45 MHz) δ 12.3 (d, $J_{\text{PC}} = 90.7$ Hz), 13.9, 28.5, 29.3 (d, $J_{\text{PC}} = 84.3$ Hz), 30.7, 32.9, 33.0, 51.6, 51.8, 55.8 (d, $J_{\text{POC}} = 13.2$ Hz), 56.2 (d, $J_{\text{POC}} = 15.8$ Hz), 65.5, 65.6, 65.7, 65.8, 67.6 (d, $J_{\text{POC}} = 11.2$ Hz), 68.2 (d, $J_{\text{POC}} = 11.2$ Hz), 79.9, 80.5, 155.2, 188.1. ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 48.0 & 44.41 (s). HRMS (M^+H ion by direct probe) : calc. for $\text{C}_{15}\text{H}_{31}\text{O}_6\text{P}$ 352.1889, obs. 352.1884.

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Sample directory:
File: PROTON
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Pulse Sequence: s2pul

Solvent: CDCl3

Ambient temperature
Mercury-300BB "mercuryplus300"

Relax. delay 1.000
Pulse 45 0 degrees

Acq. time 1.994 sec

width 4003.1 Hz
8 repetitions[illegible]

CHIA FRUGLESS	IT size 32768
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Total time 0 min, 43 sec

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2	2350.821	7.835	16.2	41	1227.739	4.082	13.4
3	2349.355	7.830	8.1	42	1224.807	4.082	12.1
4	2346.424	7.820	5.2	43	1220.703	4.068	9.4
5	2344.371	7.813	19.6	44	1217.772	4.059	16.8
6	2342.613	7.807	19.2	45	1210.443	4.034	12.1
7	2340.267	7.800	12.5	46	1203.407	4.011	3.5
8	2338.802	7.795	17.0	47	1169.401	3.897	3.3
9	2337.336	7.790	8.6	48	1162.365	3.874	10.7
10	2334.404	7.780	5.1	49	1155.329	3.850	11.9
11	2332.352	7.773	19.9	50	1154.743	3.848	11.3
12	2330.593	7.767	18.9	51	1152.398	3.841	8.1
13	2278.118	7.592	4.0	52	1147.707	3.825	10.0
14	2276.652	7.588	4.6	53	1145.362	3.817	8.4
15	2270.496	7.567	10.4	54	1144.482	3.814	9.5
16	2269.030	7.562	11.0	55	1140.378	3.801	3.3
17	2266.685	7.554	3.9	56	1137.447	3.791	8.6
18	2265.219	7.549	7.4	57	617.680	2.059	3.2
19	2263.460	7.544	18.1	58	509.506	1.698	162.0
20	2261.995	7.539	17.1	59	495.141	1.650	149.2
21	2260.236	7.533	6.3	60	423.904	1.413	3.7
22	2259.063	7.529	4.4	61	394.588	1.315	68.2
23	2257.890	7.525	12.2	62	387.553	1.292	134.3
24	2257.011	7.522	18.0	63	382.862	1.276	5.8
25	2255.838	7.518	12.0	64	380.517	1.268	63.3
26	2254.666	7.514	13.1	65	378.172	1.260	3.6
27	2253.786	7.511	18.6	66	375.826	1.253	10.9
28	2252.614	7.507	9.8	67	368.791	1.229	9.3
29	2251.441	7.504	11.9	68	3.225	0.011	11.6
30	2249.975	7.499	19.3	69	0.000	0.000	320.6
31	2248.216	7.493	15.4	70	-3.225	-0.011	7.1
32	2246.750	7.488	18.3				
33	2244.698	7.481	7.9				
34	2242.939	7.475	7.4				
35	2241.180	7.469	7.7				
36	2239.715	7.464	8.3				
37	2237.956	7.459	5.1				
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677

exp1 s2pul

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np	not used	19158	il	20.000	20.000
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ss	4	dp	hs		n
ss	1.000	hs			y
d1	8	fn			nm
nt					
ct					
TRANSMITTER			PROCESSING		
tn	H1	sp	fn	not used	DISPLAY
sfreq	300.053	wp			-60.0
tof	300.9	rfl			3060.5
tpwr	55	rffp			598.3
ppw	8.600	rp			0
DECOUPLER			lp		-116.5
dn	C13	1p			-75.3
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dmm	c	vs			0
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dpmf	13100	ai			3
		cdc			ph

677

exp1 s2pul

SAMPLE		SPECIAL	
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fb	14800	il	n
bs	64	in	n
ss	4	dp	y
di	1.000	hs	nn
nt	15	lb	2.00
ct	15	fn	not used
TRANSMITTER		DISPLAY	
tn	P31	sp	0
sfrq	121.474	wp	10010.0
tof	10608.2	rfl	2437.3
tpwr	55	rfl	0
pw	7.117	rp	130.6
DECOUPLER		lp	-288.8
dn	H1	PLOT	
dof	0	wc	250
dm	VVY	w	0
dmm	W	sc	0
dwr	35	vs	10
dmf	6700	th	4
		ai	no ph

cs 105

exp1 s2pul

SAMPLE		SPECIAL	
date	Sep 13 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
rp	19158	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	8	PROCESSING	not used
ct	8	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-60.0
sfrq	300.053	wp	3060.5
tof	300.9	rfl	595.7
tpwr	55	rfp	-100.5
pw	8.600	lp	-70.9
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	337
dmm	c	th	3
dpwr	45	ai	cdc
dmf	13100	ph

cs 102 6hr
exp1 s2pu1

SAMPLE		SPECIAL	
date	Aug 14 2006	temp	not used
solvent	THF	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
di	1.000	hs	nn
nt	16	PROCESSING 2.00	
ct	16	lb	not used
TRANSMITTER		fn	DISPLAY
tn	P31	0	
sfrq	121.474	sp	10010.0
tof	10608.2	wp	2437.3
tpwr	55	rfl	-72.9
pw	7.117	rfp	-288.8
DECOUPLER		rp	PL0T
dn	H1	lp	250
dof	0	yy	0
dm	yy	wc	21
dmm	w	sc	4
dpwr	35	vs	
dmf	6700	th	
		ai	no ph

INDEX FREQUENCY HEIGHT
1 6631.443 54.597 107.7

CS 110

exp1 s2pu1

SAMPLE		SPECIAL	
date	Nov 10 2006	temp	not used
solvent	CDC13	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alpha	20.000
np	19158	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
di	1.000	hs	nn
nt	8	PROCESSING	
ct	8	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-60.0
sfrq	300.053	wp	3060.5
tof	300.9	rfl	593.1
tpwr	55	rfp	0
pw	8.600	rp	-100.7
DECOUPLER		lp	-74.4
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	277
dpwr	45	th	2
dmf	13100	ai	cdc
		ph

604 after column

exp1 s2pul

SAMPLE		SPECIAL	
date	Sep 2 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	8	PROCESSING	
ct	8	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-60.0
sfrq	300.053	wp	3060.5
tof	300.8	rfl	594.8
tpwr	55	rff	0
pw	8.600	rp	-108.8
DECOUPLER		lp	-71.0
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	C	vs	128
dpwr	45	th	3
dmf	13100	ai	cdc
		ph

604 after column

exp1 s2pu1

SAMPLE		SPECIAL	
date	Sep 2 2005	temp	not used
solvent	CDCl3	gain	not used
file		spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	l1	n
bs	64	in	n
ss	4	dp	y
di	1.000	hs	nn
nt	10	PROCESSING	
ct	10	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-168.1
dn	H1	lp	-288.8
dof	0	PLOT	
dm	YVY	wc	250
dmm	w	sc	0
dpwr	35	vs	36
dmf	6700	th	10
		al	no ph

exp1 s2pul

SAMPLE		SPECIAL	
date	9 2006	temp	not used
solvent	May CDC13	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	6	PROCESSING	
ct	6	lb	2.00
tn	P31	fn	not used
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-144.1
dn	H1	lp	-288.8
dof	0	PLOT	
dm	VVY	wc	250
dmm	W	sc	0
dpwr	35	vs	8
dmf	6700	th	2
		ai	no ph

100% 40 0.00

exp1 s2pul

SAMPLE		SPECIAL	
date	May 9 2006	temp	not used
solvent	CDC13	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	18867.9	pw90	18.500
at	1.815	alfa	20.000
np	68492	FLAGS	
fb	10400	il	n
bs	64	in	n
ss	4	dp	y
di	1.000	hs	nn
nt	512	PROCESSING	
ct	469	lb	1.00
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	75.456	sp	-150.9
tof	737.2	wp	15844.1
tpwr	58	rfl	1134.7
pw	9.250	rff	0
DECOUPLER		rp	131.5
dn	H1	lp	-363.9
dof	0	PLOT	
dm	yyv	wc	250
dmm	w	sc	0
dpwr	35	vs	202
dmf	6700	th	14
		ai	no ph

576 pure

expl s2pul

SAMPLE		SPECIAL	
date	Aug 13 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	8	PROCESSING	
ct	8	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-60.0
sfrq	300.053	wp	3060.5
tof	300.9	rfl	594.5
tpwr	55	rff	0
pw	8.600	rp	-111.9
DECOUPLER		lp	-70.2
dn	C13	PLOT	
dof	0	wc	250
dm	nn	sc	0
dmm	c	vs	121
dpwr	45	tn	2
dmf	13100	ai	cdc ph

3.85

INDEX 1 FREQUENCY 5993.348 PPM 49.343 HEIGHT 126.0

49.343

INDEX	FREQUENCY	PPM	HEIGHT
1	12965.076	171.841	21.3
2	7624.788	101.060	19.2
3	7485.444	99.213	18.9
4	5771.565	76.497	76.3
5	5739.608	76.074	78.5
6	5707.651	75.650	76.9
7	4566.562	60.791	8.2
8	4580.804	60.715	33.2
9	4573.894	60.623	34.7
10	4480.038	59.379	54.6
11	4313.343	57.170	41.6
12	4308.737	57.109	42.2
13	4276.204	56.678	39.7
14	4269.006	56.582	40.7
15	2561.749	33.954	45.2
16	2547.930	33.771	52.3
17	2535.550	33.607	8.7
18	1835.085	24.323	40.8
19	1748.427	23.174	40.3
20	1743.820	23.113	8.3
21	1471.178	19.499	47.6
22	1458.798	19.335	46.9
23	1309.665	17.359	7.5
24	1201.702	15.928	52.7
25	1197.095	15.866	51.8
26	1183.852	15.691	35.6
27	1178.382	15.618	35.7
28	1093.163	14.489	74.8
29	1076.176	14.264	73.1
30	996.428	13.207	59.9
31	-0.000	-0.000	14.5

SPECIAL not used
 temp gain not used
 spin 20
 hst 0.008
 pw90 18.500
 alfa 20.000
 FLAGS
 11 n
 64 n
 4 y
 1.000 nn
 512 1.00
 288 not used
 PROCESSING
 tn C13
 sf1q 75.456 SP -1134.4
 tof 737.2 WP 18867.6
 tpwr 58 rfl 1226.2
 pw 9.250 rfp 92.1
 DECOUPLER H1 0 -258.9
 dn 0
 dof 0
 dm VV WC 250
 dmm W SC 0
 dpwr 35 VS 161
 dmf 6700 TH 7
 at no ph

exp1 s2pu1

SAMPLE

date	solvent	CDCl ₃	exp	spin	gain	temp	not used
Jan 19 2006	CDCl ₃	exp	spin	gain	temp	not used	
ACQUISITION	sw	4803.1	hst	hw	17.200	0.008	
at	1.994	19158	alfa	alfa	20.000	20.000	
np	not used						FLAGS
fb	16						n
bs	4						n
ss	4						y
d1	1.000						nn
nt	8						
ct	8						PROCESSING
tn	H1						not used
strq	300.053						DISPLAY
tof	300.9						-60.0
tpwr	55						3080.5
pw	8.600						591.3
DECOUPLER							0
dn	C13						-109.3
dof	0						-71.4
dm	nmn						PLOT
dmm	C						250
dmn	45						0
dpmr	13100						208
dmf	at						2
	cdc						ph

ap4 carbone

exp1 s2pul

SAMPLE		SPECIAL	
date	Jan 19 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	18867.9	pw90	18.500
at	1.815	alfa	20.000
np	68492	FLAGS	
fb	10400	l1	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	512	PROCESSING	
ct	354	lb	1.00
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	75.456	sp	-150.9
tof	737.2	wp	15844.1
tpwr	58	rfl	1134.7
pw	9.250	rffp	117.9
DECOUPLER		lp	-304.7
dn	H1	PLOT	
dof	0	wc	250
dm	YVY	w	0
dmm	W	sc	0
dpwr	35	vs	153
dmf	6700	th	13
		ai	no
			ph

9

exp1 s2pul

SAMPLE
date May 17 2006
solvent CDCl3
file CDC13

SPECIAL

temp not used

gain not used

spin 20

hst 0.008

pw90 17.200

alpha 20.000

FLAGS

il n

in n

dp y

hs nn

PROCESSING

not used

fn

DISPLAY

tn H1 sp

sfrq 300.053 wp -60.0

tof 300.9 rfl 3060.5

tpwr 55 rfp 593.3

pw 8.600 rp -103.3

DECOUPLER C13 lp -70.1

dn C13 PLOT

dof 0 wc 250

dm nnn sc 0

dmm c vs 340

dpwr 45 th cdc

dmf 13100 ai ph 3

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0.00

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0.02

0.43

exp1 s2pu1

SAMPLE May 17 2006 SPECIAL
 date May 17 2006 temp not used
 solvent CDC13 gain not used
 file exp 20
 ACQUISITION hst 0.008
 sw 26738.0 pw90 18.300
 at 1.598 alfa 20.000
 np 85476
 fb 14800
 bs 64
 ss 4
 di 1.000
 nt 6
 ct 6

TRANSMITTER P31
 tn 121.474 sp
 sfrq 10608.2 wp 10010.0
 tof 55 rfl 2437.3
 tpwr 7.117 rfp 0
 pw DECOUPLER H1 lp -126.6
 dn 0 -288.8
 dof 0
 dm YVY WC 250
 dmm W SC 0
 dpwr 35 VS 10
 dmf 6700 th no ph 3

PROCESSING 2.00
 DISPLAY not used

FLAGS
 ll in n
 64 in n
 4 dp y
 hs nn
 lb fn 2.00
 not used

PLOT

exp1 s2pul

SAMPLE
 date May 17 2006
 solvent CDC13
 file exp
 ACQUISITION
 sw 18867.9
 at 1815
 np 68482
 fb 10400
 bs 64
 ss 4
 d1 1.000
 nt 512
 ct 415
 TRANSMITTER C13
 tn 75.456
 sfrq 737.2
 tof 58
 tpwr 9.250
 pw DECOUPLER
 dn H1
 dof 0
 dm YY
 dmm W
 dpwr 35
 dmf 6700

SPECIAL
 temp not used
 gain not used
 spin 20
 hst 0.008
 pw90 18.500
 alfa 20.000
 FLAGS
 ll n
 ln n
 dp y
 hs nn
 PROCESSING
 lb 1.00
 fn not used
 DISPLAY
 sp -150.9
 wp 15844.1
 rfl 1134.7
 rfp 0
 rp 120.5
 lp -342.5
 PLOT
 wc 250
 sc 0
 vs 201
 th 12
 ai no ph

21

exp1 s2pul1

SAMPLE		SPECIAL	
date	Mar 8 2006	temp	not used
solvent	CDC13	gain	not used
file	exp	spn	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
fb	19158	FLAGS	
nt	not used	il	n
bs	16	in	n
ss	4	dp	y
di	1.000	hs	nn
nt	8	fn	not used
ct	8	PROCESSING	
TRANSMITTER		DISPLAY	
tn	H1	sp	-60.0
sfrq	300.053	wp	3060.5
tof	300.9	rfl	592.2
tpwr	55	rfp	-105.5
pw	8.600	rp	-89.9
DECOUPLER		lp	PLOT
dn	G13	wc	250
dof	0	nn	0
dm	nn	sc	0
dmm	c	vs	185
dpwr	45	th	2
dmf	13100	ai	cdc ph

615

exp1 s2pu1

SAMPLE		SPECIAL	
date	Sep 14 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	10	PROCESSING	
ct	10	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	126.7
dn	H1	lp	-288.8
dof	0	PLOT	
dm	YVY	wc	250
dmm	W	sc	0
dpwr	35	vs	19
dmf	6700	th	4
		ai	no ph

INDEX	FREQUENCY	PPM	HEIGHT
1	7695.826	102.002	18.6
2	7549.572	100.063	13.5
3	7113.113	94.278	14.7
4	5861.893	77.695	80.7
5	5829.935	77.271	84.5
6	5797.978	76.847	86.1
7	4707.407	62.393	28.1
8	4700.209	62.297	29.5
9	4421.233	58.600	31.7
10	4415.475	58.524	34.4
11	4410.868	58.462	42.4
12	4385.821	58.130	34.4
13	4378.335	58.031	33.6
14	4311.254	57.142	42.0
15	2948.903	39.085	22.9
16	2064.757	27.367	252.4
17	1560.641	20.685	39.8
18	1548.550	20.525	36.9
19	1274.179	16.888	28.0
20	1268.997	16.820	25.8
21	1182.051	15.667	59.6
22	1170.823	15.518	54.9

330
1542303

bomalkylation
exp1 s2pul
SAMPLE
date Sep 12 2005
solvent CDCl3
file exp
ACQUISITION
sw 4803.1
at 1.994
np 19158
fb not used
bs 16
ss 4
d1 1.000
nt 8
ct 8
TRANSMITTER H1
tn 300.053
sfrq 300.3
tof 55
tpwr 8.600
pw DECOUPLER C13
dn 0
dof 250
dm nnn
dmn SC
dmr C
dpwr 45
dmf 13100
SPECIAL
temp not used
gain not used
spin 20
hst 0.008
pw90 17.200
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn not used
DISPLAY
-60.0
3080.5
588.3
0
-112.3
-67.8
PLOT
wc 250
sc 0
vs 156
th 3
cdc ph

bomalkylation

exp1 s2pu]

SAMPLE		SPECIAL	
date	Sep 12 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
nt	1.000	hs	nn
ct	8	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rpf	0
DECOUPLER		rp	-127.3
dn	H1	lp	-288.8
dof	0	PLOT	
dm	YVY	wc	250
dmm	w	sc	0
dpwr	35	vs	14
dmf	6700	th	4
		ai	no
		ph

40

19

exp1 s2pul

SAMPLE		SPECIAL	
date	Mar 8 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	18867.9	pw90	18.500
at	1.815	alfa	20.000
np	68492	FLAGS	
fb	10400	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	512	PROCESSING	
ct	512	lb	1.00
TRANSMITTER C13		fn	not used
tn	75.456	DISPLAY	
sfrq	737.2	sp	-150.9
tof	58	wp	15844.1
tpwr	9.250	rfl	1134.7
pw	DECOUPLER	rfp	0
dn	H1	rp	52.2
dof	0	lp	-268.7
dm	yyv	wc	250
dmm	w	sc	0
dpwr	35	vs	182
dmf	6700	th	10
		ai	no
		ph

gibapyr3

exp1 s2pu1

SAMPLE		SPECIAL	
date	Nov 1 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
di	1.000	hs	nn
nt	8	fn	not used
ct	8	PROCESSING	
TRANSMITTER		DISPLAY	
tn	H1	sp	-60.0
sfrq	300.053	wp	3060.5
tof	300.9	rfl	595.7
tpwr	55	rfp	-116.7
pw	8.600	lp	-65.1
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	nnn	0
dm	nmn	sc	213
dmm	c	vs	3
dpwr	45	th	
dmf	13100	ai	cdc
		ph

gibapyr3

exp1 s2pu1

SAMPLE		SPECIAL	
date	Nov 1 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
di	1.000	hs	nn
ct	10	PROCESSING	
tn	10	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rffp	0
DECOUPLER		rp	-148.5
dn	H1	lp	-288.8
dof	0	PLOT	
dm	YVY	wc	250
dmm	w	sc	0
dpwr	35	vs	16
dmf	6700	th	4
		ai	no
		ph

33

682

exp1 s2pul

SAMPLE		SPECIAL	
date	Nov 1 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	18867.9	pw90	18.500
at	1.815	alfa	20.000
np	68492	FLAGS	
fb	10400	il	n
bs	64	in	n
ss	4	dp	y
dl	1.000	hs	nn
nt	512	PROCESSING	
ct	512	lb	1.00
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	75.456	sp	-150.9
tof	737.2	wp	15844.1
tpwr	58	rfl	1151.3
pw	9.250	rfp	0
DECOUPLER		rp	99.9
dn	H1	lp	-322.1
dof	0	PLOT	
dm	YVY	wc	250
dmm	W	sc	0
dpwr	35	vs	211
dmf	6700	th	7
		ai	no
		ph

```

exp1 s2pul
SAMPLE
date Oct 20 2005
solvent CDCl3
file exp
ACQUISITION
sw 4803.1 pw30
at 1.994 alpha
fb 19158
bs not used il
ss 16 in
d1 4 dp
nt 1.000 hs
ct 8 fn
TRANSMITTER
tn H1 sp
sfrq 300.053 wp
tof 300.9 rfl
tpwr 55 rfp
pw 8.600 rp
DECOUPLER C13 lp
dn 0 wc
dof 0 sc
dm 0 vs
dmm 131
dpwr 45 th
dmf 13100 ai cdc ph
SPECIAL
temp not used
gain not used
spin 20
hst 0.008
17.200
20.000
FLAGS
n
n
y
nn
PROCESSING
not used
DISPLAY
-60.0
3060.5
583.4
0
-108.7
-73.7
PLOT
250
0
131
2
7.71
8.99
-0.03
0.70
32.40
49.27
0.41
0.56

```

INDEX	FREQUENCY	PPM	HEIGHT
1	11404.288	151.155	31.6
2	11398.242	151.074	31.2
3	11266.095	149.323	25.7
4	11229.532	148.838	20.7
5	11184.619	148.243	31.4
6	11181.452	148.201	29.1
7	10402.102	137.871	27.3
8	10397.496	137.810	28.6
9	10040.498	133.079	20.8
10	9316.425	123.482	28.6
11	5863.332	77.714	103.7
12	5831.375	77.290	103.9
13	5799.418	76.867	100.8
14	4704.528	62.355	22.3
15	4426.991	58.676	30.0
16	4422.384	58.615	25.7
17	4372.001	57.947	24.4
18	4364.228	57.844	24.6
19	2382.601	31.579	30.0
20	2302.276	30.515	32.4
21	1564.960	20.742	27.9
22	1552.580	20.578	20.0
23	1266.694	16.789	21.2
24	1187.521	15.740	41.6
25	1166.504	15.461	44.8

581 pure

exp1 s2pu1

SAMPLE		SPECIAL	
date	Aug 13 2005	temp	not used
solvent	CDCl3	gain	not used
file		spin	20
ACQUISITION	exp	hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	8	fn	not used
ct	8	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-60.0
sfrq	300.053	wp	3060.5
tof	300.9	rfl	596.0
tpwr	55	rflp	0
pw	8.600	rp	-109.9
dn	DECOUPLER	lp	-77.8
dof	C13	pl	PLOT
dm	0	wc	250
dmm	nnn	sc	0
dpwr	c	vs	133
dmf	45	th	3
	13100	ai	cdc
		ph

581 pure

expl s2pu1

SAMPLE		SPECIAL	
date	Aug 13 2005	temp	not used
solvent	CDCl3	gain	not used
file		spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alifa	20.000
np	85476	FLAGS	
bs	14800	il	n
ss	64	in	n
d1	4	dp	y
nt	1.000	hs	nn
ct	6	PROCESSING	
tn	TRANSMITTER P31	lb	2.00
sfreq	121.474	fn	not used
tof	10608.2	sp	DISPLAY
tpwr	55	wp	0
pw	7.117	rfl	10010.0
DECOUPLER		rfp	2437.3
dn	H1	rp	0
dof	0	lp	-156.5
dm	YVY	PLOT	
dmm	w	wc	-288.8
dpwr	35	sc	250
dmf	6700	vs	0
		th	17
		ai	5
		no	
		ph

¹H, ¹³C

ap5

exp1 s2pul

SAMPLE

date Jan 19 2006

solvent CDC13

file exp

ACQUISITION

sw 4803.1

at 1.994

np not used

bs 16

ss 4

d1 1.000

nt 8

ct 8

tn TRANSMITTER

sftrq H1

tof 300.053

tpwr 300.9

pw 55

DECOUPLER

C13

dn 0

dof 0

dm 250

dmm 0

dpwr 287

dmf 2

SPECIAL

temp not used

gain not used

spin 20

hst 0.008

pw80 17.200

alfa 20.000

FLAGS

il n

in n

dp y

hs nn

PROCESSING

fn not used

DISPLAY

-60.0

3060.5

593.6

0

-109.6

-68.5

PLOT

250

0

287

2

SPECIAL

temp not used

gain not used

spin 20

hst 0.008

pw80 17.200

alfa 20.000

FLAGS

il n

in n

dp y

hs nn

PROCESSING

fn not used

DISPLAY

-60.0

3060.5

593.6

0

-109.6

-68.5

PLOT

250

0

287

2

SPECIAL

temp not used

gain not used

spin 20

hst 0.008

pw80 17.200

alfa 20.000

FLAGS

il n

in n

dp y

hs nn

PROCESSING

fn not used

DISPLAY

-60.0

3060.5

593.6

0

-109.6

-68.5

PLOT

250

0

287

2

SPECIAL

temp not used

gain not used

spin 20

hst 0.008

pw80 17.200

alfa 20.000

FLAGS

il n

in n

dp y

hs nn

PROCESSING

fn not used

DISPLAY

-60.0

3060.5

593.6

0

-109.6

-68.5

PLOT

250

0

287

2

SPECIAL

temp not used

gain not used

spin 20

hst 0.008

pw80 17.200

alfa 20.000

FLAGS

il n

in n

dp y

hs nn

PROCESSING

fn not used

DISPLAY

-60.0

3060.5

593.6

0

-109.6

-68.5

PLOT

250

0

287

2

SPECIAL

temp not used

gain not used

spin 20

hst 0.008

pw80 17.200

alfa 20.000

FLAGS

il n

in n

dp y

hs nn

PROCESSING

fn not used

DISPLAY

-60.0

3060.5

593.6

0

-109.6

-68.5

PLOT

250

0

287

2

SPECIAL

temp not used

gain not used

spin 20

hst 0.008

pw80 17.200

alfa 20.000

FLAGS

il n

in n

dp y

hs nn

PROCESSING

fn not used

DISPLAY

-60.0

3060.5

593.6

0

-109.6

-68.5

PLOT

250

0

287

2

SPECIAL

temp not used

gain not used

spin 20

hst 0.008

pw80 17.200

alfa 20.000

FLAGS

il n

in n

dp y

hs nn

PROCESSING

fn not used

DISPLAY

-60.0

3060.5

593.6

0

-109.6

-68.5

PLOT

250

0

287

2

SPECIAL

temp not used

gain not used

spin 20

hst 0.008

pw80 17.200

alfa 20.000

FLAGS

il n

in n

dp y

hs nn

PROCESSING

fn not used

DISPLAY

-60.0

3060.5

593.6

0

-109.6

-68.5

PLOT

250

0

287

2

SPECIAL

temp not used

gain not used

spin 20

hst 0.008

pw80 17.200

alfa 20.000

FLAGS

il n

in n

dp y

hs nn

PROCESSING

fn not used

DISPLAY

-60.0

3060.5

593.6

0

-109.6

-68.5

PLOT

250

0

287

2

SPECIAL

temp not used

gain not used

spin 20

hst 0.008

pw80 17.200

alfa 20.000

FLAGS

il n

in n

dp y

hs nn

PROCESSING

fn not used

DISPLAY

-60.0

3060.5

593.6

0

-109.6

-68.5

PLOT

250

0

287

2

SPECIAL

temp not used

gain not used

spin 20

hst 0.008

pw80 17.200

alfa 20.000

FLAGS

il n

in n

dp y

hs nn

PROCESSING

fn not used

DISPLAY

-60.0

3060.5

593.6

0

-109.6

-68.5

PLOT

250

0

287</

601 after column

exp1 s2pul

SAMPLE		SPECIAL	
date	Sep 2.2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	10	PROCESSING	
ct	10	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	131.9
dn	H1	lp	-288.8
dof	0	PLOT	
dm	YVY	wc	250
dmm	4	sc	0
dpwr	35	vs	24
dmf	6700	tn	11
		ai	no ph

ap5

exp1 s2pul

SAMPLE		SPECIAL	
date	Jan 19 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	18867.9	pw90	18.500
at	1.815	alfa	20.000
np	68492	FLAGS	
fb	10400	il	n
bs	64	tn	n
ss	4	dp	y
d1	1.000	hs	nn
nt	512	PROCESSING	
ct	512	lb	1.00
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	75.456	sp	-150.9
tof	737.2	wp	15844.1
tpwr	58	rfl	1134.7
pw	9.250	rffp	0
DECOUPLER		rp	74.4
dn	H1	lp	-283.5
dof	0	PLOT	
dm	YVY	wc	250
dmm	4	sc	0
dpwr	35	vs	211
dmf	6700	tn	11
		ai	no ph

584b pure

exp1 s2pul

SAMPLE		SPECIAL	
date	Aug 19 2005	temp	not used
solvent	CDCl3	gain	not used
file		spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	8	PROCESSING	
ct	not used	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-60.0
sfrq	300.053	wp	3060.5
tof	300.9	rfl	598.0
tpwr	55	rfp	0
pw	8.600	rp	-115.1
DECOUPLER		lp	-63.5
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	269
dpwr	45	th	3
dmf	13100	ai	cdc
		ph

52

584b pure

exp1 s2pul

SAMPLE		SPECIAL	
date	Aug 19 2005	temp	not used
solvent	CDC13	gain	not used
file		spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	il	n
fb	14800	in	n
bs	64	dp	y
ss	4	hs	nn
d1	1.000	hs	nn
nt	15	PROCESSING	
ct	15	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfreq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfl	0
DECOUPLER		rp	108.9
dn	H1	lp	-115.8
dof	0	PLOT	
dm	ynn	wc	250
dmm	w	sc	0
dpwr	35	vs	37
dmf	6700	th	19
		ai	no
			ph

53

INDEX

FREQUENCY PPM

HEIGHT

INDEX

FREQUENCY PPM

HEIGHT

std

exp1 s2pul

SAMPLE		SPECIAL	
date	Mar 21 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
dl	1.000	hs	nn
nt	6	PROCESSING	
ct	6	lb	not used
TRANSMITTER		fn	DISPLAY
tn	P31	0	
sfrq	121.474	sp	10010.0
tof	10608.2	wp	2437.3
tpwr	55	rfl	0
pw	7.117	rfp	152.3
DECOUPLER		rp	58.2
dn	H1	lp	PLOT
dof	0	wc	250
dm	YVY	w	0
dmm	W	sc	0
dwr	35	vs	26
dmf	6700	th	11
		ai	no
		ph

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2181.670	7.271	30.8	40	505.402	1.684	26.4
2	1840.143	6.133	14.4	41	504.522	1.681	26.8
3	1816.983	6.056	15.3	42	501.004	1.670	21.0
4	1791.185	5.970	31.4	43	498.952	1.663	35.4
5	1768.026	5.892	31.8	44	494.555	1.648	16.3
6	1742.228	5.806	16.4	45	492.503	1.641	19.9
7	1719.069	5.729	15.4	46	488.985	1.630	7.5
8	1283.145	4.276	3.9	47	486.933	1.623	8.2
9	1276.696	4.255	9.2	48	484.881	1.616	7.2
10	1273.178	4.243	7.4	49	478.138	1.594	4.3
11	1269.367	4.231	10.3	50	443.546	1.478	7.4
12	1266.728	4.222	15.9	51	437.976	1.460	6.7
13	1262.624	4.208	5.3	52	435.924	1.453	22.5
14	1259.107	4.196	15.9	53	430.354	1.434	20.1
15	1252.657	4.175	7.3	54	428.595	1.428	34.9
16	1247.674	4.158	7.1	55	423.025	1.410	25.5
17	1241.224	4.137	20.0	56	420.679	1.402	40.0
18	1237.706	4.125	4.7	57	415.696	1.385	16.9
19	1234.481	4.114	20.4	58	413.644	1.379	26.9
20	1231.257	4.103	11.7	59	408.953	1.363	7.7
21	1227.739	4.092	7.5	60	406.315	1.354	11.4
22	1224.514	4.081	11.6	61	382.276	1.274	85.7
23	1217.772	4.059	4.1	62	377.292	1.257	35.0
24	572.534	1.908	6.8	63	291.691	0.972	77.4
25	571.362	1.904	6.9	64	284.362	0.948	162.0
26	565.499	1.885	11.7	65	281.723	0.939	4.3
27	562.860	1.876	8.7	66	277.033	0.923	63.1
28	561.981	1.873	8.7	67	271.170	0.904	26.3
29	560.515	1.868	8.0	68	264.720	0.882	96.5
30	559.636	1.865	8.2	69	257.684	0.859	31.2
31	555.824	1.852	19.8	70	2.932	0.010	3.3
32	552.307	1.841	9.4	71	2.052	0.007	3.0
33	548.202	1.827	9.2	72	1.466	0.005	4.9
34	542.339	1.807	11.0	73	-0.000	-0.000	107.1
35	541.460	1.805	11.9				
36	520.646	1.735	10.3				
37	513.610	1.712	27.1				
38	510.972	1.703	14.7				
39	506.867	1.689	32.6

octfluoro

exp1 s2pul

SAMPLE
date Mar 16 2006
solvent CDC13
file exp
ACQUISITION
sw 26738.0
at 1.598
np 85476
fb 14800
bs 64
ss 4
d1 1.000
nt 10
ct 10
TRANSMITTER P31
tn 121.474
sfrq 10608.2
tof 55
tpwr 7.117
pw DECOUPLER H1
dn 0
dof YYV
dm W
dmm 35
dpwr 6700
dmf th
SPECIAL
temp not used
gain not used
spin 20
hst 0.008
pw90 18.300
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 2.00
fn not used
DISPLAY
sp 0
wp 10010.0
rf1 2437.3
rfp 0
rp -162.4
lp -288.8
PLOT
WC 250
SC 0
VS 56
TH 12
ai no ph

exp1 s2pul

SAMPLE
date Apr 25 2006
solvent CDCl3
file
ACQUISITION
sw 4803.1
at 1.994
np not used
bs 16
ss 4
d1 1.000
nt 8
ct 8
tn TRANSMITTER H1
sfrq 300.053
tof 300.9
tpwr 55
pw 8.600
dn DECOUPLER C13
dof 0
dm nnn
dmn C
dpwr 45
dmf 13100
ai cdc
ph

SPECIAL
temp not used
gain not used
spin 20
hst 0.008
pw90 17.200
alpha 20.000
FLAGS

PROCESSING
nn
fn not used
DISPLAY

H1 sp -60.0
wp 3060.5
rf1 590.1
rfl 0
rff 0
lp -106.6
lp -63.6
WC 250
SC 0
VS 350
th 2

59

exp1 s2pul

SAMPLE date Apr 25 2006
solvent CDC13
file
ACQUISITION exp
sw 26738.0
at 1.598
np 85476
fb 14800
bs 64
ss 4
d1 1.000
nt 6
ct 6
TRANSMITTER P31
tn 121.474
sfrq 10608.2
tpwr 55
pw 7.117
DECOUPLER H1
dn 0
dof 0
dm YVW
dmm W
dpwr 35
dmf 6700
SPECIAL temp not used
gain not used
spin 20
hst 0.008
pw90 18.300
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING 2.00
lb fn not used
fn DISPLAY
sp 0
wp 10010.0
rfl 2437.3
rfp 0
rp -177.9
lp -288.8
PLOT
wc 250
w 0
sc 12
vs 4
th no
ai ph

60

Isa #6

exp1 s2pul

SAMPLE		SPECIAL	
date	May 17 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19156	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nm
nt	8	fn	not used
ct	8	PROCESSING	
TRANSMITTER		DISPLAY	
tn	H1	sp	-60.0
sfrq	300.053	wp	3060.5
tof	300.9	rfl	592.2
tpwr	55	rpf	0
pw	8.600	rp	-105.4
dn	C13	lp	-60.6
dof	0	wc	250
dm	nmn	sc	0
dmm	c	vs	255
dpwr	45	th	3
dmf	13100	ai	cdc
		ph

TRANSMITTER

62

INDEX	FREQUENCY	PPM	HEIGHT
1	6078.618	50.045	3.4
2	6059.034	49.884	126.0

Index
1
2

100.000
100.000
100.000

100.000
100.000
100.000

100.000
100.000
100.000

630

Isa #6

```

exp1 s2pul1
SAMPLE
date May 17 2006
solvent CDCl3
file ACQUISITION
sw 20746.9
at 1815
np 75312
fb 11400
bs 64
ss 4
d1 1.000
nt 512
ct 241
tn TRANSMITTER C13
sfrq 75.456
tof 925.8
tpwr 58
pw 9.250
dn DECOUPLER H1
dof 0
dm yyy
dmw w
dpwr 35
dmf 6700
SPECIAL
temp not used
gain not used
spin 20
hst 0.008
pw90 18.500
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn not used
DISPLAY
sp -150.9
wp 15844.1
rfl 1885.6
rfp 0
rp 14.5
lp -276.8
PLOT
wc 250
sc 0
vs 133
th 12
ai no
ph

```

100% C13
 100% C13
 100% C13

629

exp1 s2pu1

SAMPLE		SPECIAL	
date	Sep 23 2005	temp	not used
solvent	CDC13	gain	not used
file		spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	8	fn	not used
ct	8	PROCESSING	
TRANSMITTER		DISPLAY	
tn	H1	sp	-60.0
sfrq	300.053	wp	3060.5
tof	300.9	rfl	595.4
tpwr	55	rff	0
pwr	8.600	rp	-111.7
DECOUPLER	1p	lp	-67.1
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	152
dpwr	45	th	2
dmf	13100	ai	cdc
		ph

629

exp1 s2pul

SAMPLE		SPECIAL	
date	temp	temp	not used
Sep 23 2005			
solvent CDC13	gain	gain	not used
file	spn	spn	20
ACQUISITION	nst	nst	0.008
sw 26738.0	pw90	pw90	18.300
at 1.598	alpha	alpha	20.000
np 85476	il	il	n
fb 14800	in	in	n
bs 64	dp	dp	y
ss 4	hs	hs	nn
d1 1.000	5	5	2.00
nt	lb	lb	not used
Ct	fn	fn	DISPLAY
TRANSMITTER	p31	p31	0
tn	121.474	sp	10010.0
sfrq	10608.2	wp	2437.3
tof	55	rfl	0
tpwr	7.117	rfl	135.2
pw	DECOUPLER	rp	-288.8
dn	H1	lp	PLOT
dof	0	wc	250
dm	yy	w	0
dmm	35	sc	31
dpwr	6700	th	10
dmf	ai	no	ph

15c

17 3/p 06 15c

std
exp1 s2pu1

SAMPLE
date Mar 21 2006
solvent CDCl3
file exp
ACQUISITION
sw 4803.1
at 1.994
np 19158
fb not used
bs 16
ss 4
di 1.000
nt 8
ct 8

TRANSMITTER
tn H1
sfrq 300.053
tof 300.9
tpwr 55
pw 8.600

DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 45
dmf 13100

SPECIAL
temp not used
gain not used
spin 20
hst 0.008
pw90 17.200
alpha 20.000

FLAGS
il n
in n
dp y
hs nn
fn not used

PROCESSING
DISPLAY
-60.0
3060.5
598.3
-103.6
-77.2

PLOT
wc 250
sc 0
vs 175
tn 3
ai cdc
ph

std

exp1 s2pul

SAMPLE		SPECIAL	
date	Mar 21 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
dl	1.000	hs	nn
nt	10	PROCESSING	
ct	10	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rffp	0
DECOUPLER		rp	-127.9
dn	H1	lp	-288.8
dof	0	PLOT	
dm	YV	wc	250
dmm	W	sc	0
dwr	35	vs	30
dmf	6700	th	17
		al	no
		ph

farnesyl fluoro

exp1 s2pul

date	Mar 21 2006	temp	not used	SPECIAL
solvent	CDCl3	gain	not used	
file	exp	spin	20	
	18867.9	hst	0.008	
sw	18867.9	pw90	18.500	
at	1.815	alfa	20.000	
tp	68492			FLAGS
fb	10400	il		n
bs	64	in		n
ss	4	dp		y
d1	1.000	hs		nn
nt	512			
ct	512	lb	1.00	
tn	TRANSMITTER C13	fn	not used	
sfrq	75.456	sp	-150.9	
tof	737.2	wp	15844.1	
tpwr	58	rfl	1134.7	
pw	9.250	rfp	0	
	DECOUPLER	H1	48.6	
dn		lp	-203.8	
dof	0			PLOT
dm	yy	wc	250	
dmm	w	sc	0	
dpwr	35	vs	213	
dmf	6700	th	17	
		ai	no	ph

butapyr3

exp1 s2pul

SAMPLE		SPECIAL	
date	Nov 1 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	il	n
fb	14800	in	n
bs	64	dp	y
ss	4	hs	nn
d1	1.000	hs	nn
nt	10	PROCESSING	2.00
ct	10	lb	not used
TRANSMITTER P31		fn	DISPLAY
tn	121.474	sp	0
sfrq	10608.2	wp	10010.0
tof	55	rfl	2437.3
tpwr	7.117	rfl	0
pw	DECOUPLER	rp	-136.3
dn	H1	lp	-288.8
dof	0	PLOT	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	9
dmf	6700	th	3
		ai	no ph

71

20

681

exp1 s2pu1

SAMPLE		SPECIAL	
date	Nov 2 2005	temp	not used
solvent	CDC13	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	18867.9	pw90	18.500
at	1.815	alfa	20.000
np	68492	FLAGS	
fb	10400	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nm
nt	512	PROCESSING	
ct	512	lb	1.00
TRANSMITTER C13		fn	not used
tn	75.456	sp	-150.9
sfrq	737.2	wp	15844.1
tof	58	rfl	1150.5
tpwr	9.250	rfp	0
pw	DECOUPLER	rp	132.4
dn	H1	lp	-316.7
dof	0	PLOT	
dm	VVY	wc	250
dmm	w	sc	0
dpwr	35	vs	209
dmf	6700	tn	8
		ai	no ph

INDEX FREQUENCY PPM HEIGHT
1 6117.785 50.368 110.8

50.368

64 62 60 58 56 54 52 50 48 46 44 42 40 38 ppm

663

exp1 s2pul

SAMPLE		SPECIAL	
date	Oct 17 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	18867.9	pw90	18.500
at	1.815	alfa	20.000
np	68492	FLAGS	
fb	10400	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	512	PROCESSING	
ct	512	lb	1.00
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	75.456	sp	-150.9
tof	737.2	wp	15844.1
tpwr	58	rfl	1134.7
pw	9.250	rff	0
DECOUPLER		rp	76.4
dn	H1	lp	-241.6
dof	0	PLOT	
dm	YVY	wc	250
dmm	W	sc	0
dpwr	35	vs	171
dmt	6700	th	13
		ai	no
			ph

662

exp1 s2pu1

SAMPLE		SPECIAL	
date	Oct 15 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	8	PROCESSING	
ct	8	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-60.0
sfrq	300.053	wp	3060.5
tof	300.9	rfl	597.5
tpwr	55	rfp	0
pw	8.600	rp	-114.7
DECOUPLER		lp	-73.1
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	211
dpr	45	th	3
dmf	13100	ai	cdc
		ph

INDEX 1 FREQUENCY 6246.709 PPM 51.429 HEIGHT 158.3

51.429

ppm

20

30

40

50

60

70

80

90

662

exp1 s2pu1

SAMPLE		SPECIAL	
date	Oct 17 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	18867.9	pw80	18.500
at	1.615	alfa	20.000
np	68492	il	n
fb	10400	in	n
bs	64	dp	y
ss	4	hs	nn
d1	1.000	ps	1.00
nt	512	lb	not used
ct	512	fn	not used
tn	TRANSMITTER C13	sp	DISPLAY
sfrq	75.456	wp	-150.9
tof	737.2	rfl	15844.1
tpwr	58	rfl	1134.7
pw	9.250	rfl	0
DECOUPLER		rp	68.0
dn	H1	lp	-270.0
dof	0	plot	PLOT
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	213
dmf	6700	th	13
	ai	no	ph

8

exp1 s2pu1

SAMPLE		SPECIAL	
date	May 17 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw90	17.200
at	1.994	alfa	20.000
np	19158	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
dl	1.000	hs	nn
nt	8	fn	not used
ct	8	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-60.0
sfrq	300.053	wp	3060.5
tof	300.9	rfl	595.4
tpwr	55	rff	0
pw	8.600	rp	-100.9
DECOUPLER		lp	-72.9
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	453
dpwr	45	th	3
dmf	13100	ai	cdc
		ph

8

exp1 s2pul

SAMPLE		SPECIAL	
date	May 17 2006	temp	not used
solvent	CDC13	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
rp	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
dl	1.000	hs	nn
nt	14	lb	2.00
ct	14	fn	not used
TRANSMITTER		DISPLAY	
tn	P31	sp	0
sfrq	121.474	wp	10010.0
tof	10608.2	rfl	2437.3
tpwr	55	rff	0
pw	7.117	rp	169.7
DECOUPLER		lp	-288.8
dn	H1	PLOT	
dof	0	vc	250
dm	VVW	sc	0
dmm	W	vs	22
dpr	35	th	6
dmf	6700	ai	no
		ph

80

8

exp1 s2pu1

SAMPLE		SPECIAL	
date	May 17 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	18867.9	pw90	18.500
at	1.815	alfa	20.000
np	68492	FLAGS	
fb	10400	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	256	PROCESSING	
ct	211	lb	1.00
TRANSMITTER C13		fn	not used
tn	C13	DISPLAY	
sfrq	75.456	sp	-150.9
tof	737.2	wp	15844.1
tpwr	58	rfl	1134.7
pw	9.250	rfp	0
DECOUPLER		rp	64.3
dn	H1	lp	-127.7
dof	0	PLOT	
dm	YVY	wc	250
dmm	W	sc	0
dpwr	35	vs	146
dmf	6700	tn	18
		ai	no ph

IA #7

exp1 s2pul

SAMPLE

date May 17 2006

solvent CDC13

file exp

sw 4803.1

at 1.994

np 19158

fb not used

bs 16

ss 4

d1 1.000

nt 8

ct 8

tn H1

sfrq 300.053

tof 300.8

tpwr 55

pw 8.600

DECOUPLER C13

dn 0

dof 250

dm 0

dmm C

dpwr 45

dmf 13100

SPECIAL

temp not used

gain not used

spin 20

hst 0.008

pw90 17.200

alfa 20.000

FLAGS

il n

in n

dp y

hs nn

PROCESSING

not used

fn

DISPLAY

-60.0

3080.5

595.7

0

-102.7

-69.1

PLOT

250

0

251

2

0.066
8.48
14.71
46.04
2.12

825

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT	INTEGRATION	AREA	AREA%
1	5574.751	45.897	126.0	1	5574.751	45.897	126.0	1.000	126.0	100.0
2	5529.056	45.521	101.2	2	5531.855	45.891	101.2	1.000	101.2	100.0

IA #7

exp1 s2pul

SAMPLE		SPECIAL	
date	May 17 2006	temp	not used
solvent	CDC13	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	18867.9	pw90	18.500
at	1.815	alfa	20.000
np	68492	FLAGS	
fb	10400	il	n
bs	64	tn	n
ss	4	dp	y
d1	1.000	hs	nn
nt	512	PROCESSING	
ct	344	lb	1.00
TRANSMITTER C13		fn	not used
tn	75.456	DISPLAY	
sfrq	737.2	sp	-150.9
tof	56	wp	15844.1
tpwr	9.250	rfl	1134.7
pw	0	rff	72.0
DECOUPLER		rp	-246.4
dn	H1	lp	
dof	0	PLOT	
dm	VVY	wc	250
dmm	W	sc	0
dpwr	35	vs	173
dmf	6700	th	17
		ai	no
			ph

60

1

expl s2pul

SAMPLE Nov 18 2005 temp not used
solvent CDCl3 gain not used
file 20
ACQUISITION exp spin
4803.1 pw90 0.008
1.994 alfa 17.200
13158 20.000
at not used il n
fb 16 in n
bs 4 dp y
ss 1.000 hs mn
nt 8 fn not used
ct TRANSMITTER H1 sp
tn 300.053 wp -60.0
sfrq 300.9 rfl 3060.5
tof 55 rfp 596.0
tpwr 8.600 rp -115.2
pw DECOUPLER lp -66.3
dn C13 PLOT
dof 0 wc 250
dm nnn sc 0
dmm c vs 183
dpwr 45 th
dmf 13100 ai cdc ph

85

exp1 s2pul

SAMPLE		SPECIAL	
date	Feb 7 2005	temp	not used
solvent	CDCl3	gain	not used
file		spin	20
ACQUISITION		hst	0.008
sw	4803.1	pw80	17.200
at	1.984	alifa	20.000
np	19158	FLAGS	
fb	not used	il	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	8	fn	not used
ct	8	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-60.0
sfrq	300.053	wp	3060.5
tof	300.9	rfl	599.8
tpwr	55	rfl	0
pw	8.600	rp	-106.8
DECOUPLER		lp	-59.6
dn	C13	PLOT	
dof	0	wc	250
dm	nmn	sc	0
dmm	c	vs	138
dpwr	45	th	3
dmf	13100	ai	cdc ph

exp1 s2pu1

SAMPLE		SPECIAL	
date	Feb 7 2005	temp	not used
solvent	CDC13	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alpha	20.000
np	85476	il	n
fb	14800	in	n
bs	64	dp	y
ss	4	hs	nm
d1	1.000	PROCESSING	
nt	10	lb	2.00
ct	10	fn	not used
TRANSMITTER		DISPLAY	
tn	p31	sp	0
sfrq	121.474	wp	10010.0
tof	10608.2	rfl	2437.3
tpwr	55	rfp	0
pw	7.117	rp	172.4
dn	H1	lp	-288.8
dof	0	PLOT	
dm	yyv	wc	250
dmm	w	sc	0
dpwr	35	vs	28
dmf	6700	th	11
		ai	no
			ph

87

1

exp1 s2pu1

SAMPLE		SPECIAL	
date	Nov 18 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		nst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nm
nt	8	PROCESSING	
ct	8	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	167.5
dn	H1	lp	-286.8
dof	0	PLOT	
dm	yy	wc	250
dmm	w	sc	0
dpwr	35	vs	46
dmf	6700	th	11
ai	no	ph

483

exp1 s2pul

SAMPLE
date May 25 2005 temp not used
solvent CDC13 gain not used
file exp 20
ACQUISITION
sw 4803.1 pw90 0.008
at 1.994 alfa 17.200
np 19158 20.000
fb not used il n
bs ib in n
ss 4 dp y
d1 1.000 hs nn
nt 8
ct 8 fn not used
TRANSMITTER H1 sp
tn -60.0
sfrq 300.053 wp 3080.5
tof 300.9 rfl 599.2
tpwr 55 rfp 0
pw 8.600 rp -117.7
DECOUPLER C13 lp -66.5
dn dof 250
dm nnn SC 0
dmm C VS 530
dpwr 45 th cdc
dmf 13100 al ph 2

SPECIAL

FLAGS

PROCESSING

not used

DISPLAY

PLOT

880

INDEX FREQUENCY PPM HEIGHT
1 7270.761 59.860 95.9

59.860

ap17

exp1 s2pul

SAMPLE		SPECIAL	
date	Jan 19 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	18867.9	pw90	18.500
at	1.815	alfa	20.000
np	68492	FLAGS	
fb	10400	il	n
bs	64	in	n
ss	4	dp	y
dl	1.000	hs	nn
nt	512	PROCESSING	
ct	512	lb	1.00
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	75.456	sp	-150.9
tof	737.2	wp	15844.1
tpwr	58	rfl	1134.7
pw	9.250	rff	0
dn	DECOUPLER	rp	63.5
dof	H1	lp	-260.6
dm	0	PLOT	
dmm	VVY	wc	250
dpr	W	sc	0
dmf	35	vs	208
	6700	th	19
		ai	no
			ph

std

exp1 s2pul

SAMPLE
date May 26 2005 temp not used
solvent D2O gain not used
file exp hst 20
ACQUISITION
sw 4803.1 pw90 0.008
at 1.934 alfa 17.200
np 19158
fb not used il n
bs 16 in n
ss 4 dp y
d1 1.000 hs
nt 8
ct 8
PROCESSING
not used
fn
DISPLAY
-60.0
tn H1 sp
sfrq 300.054 wp 3060.5
tof 300.9 rfl 601.2
tpwr 55 rfp 0
pw 8.600 rp -105.6
DECOUPLER C13 lp -77.9
PLOT
dn C13 250
dof 0 wc
dm nnn sc 0
dmm c vs 1172
dpwr 45 th 8
dmf 13100 al cdc ph

exp1 s2pu1

SAMPLE		SPECIAL	
date	May 26 2005	temp	not used
solvent	D2O	gain	not used
file	exp	spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	10	PROCESSING	
ct	10	lb	2.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	-100.5
dn	H1	lp	-288.8
dof	0	PLOT	
dm	YVY	wc	250
dmm	W	sc	0
dpwr	35	vs	18
dmf	6700	th	7
		ai	no ph

INDEX	FREQUENCY	PPM	HEIGHT
1	5098.628	41.977	47.9
2	5050.485	41.581	72.1

exp2 s2pu1

date Jan 22 2007
 solvent CDCl3
 file exp
 sw 4803.1
 at 1.994
 np 19158
 bs not used
 ss 11
 d1 16
 nt 4
 ct 1.000
 SPECIAL
 temp not used
 gain not used
 spin 20
 hst 0.008
 pw90 17.200
 alfa 20.000
 FLAGS
 n
 n
 y
 nm
 not used
 DISPLAY
 tn H1 sp -60.0
 sfrq 300.053 wp 3060.5
 tof 300.9 rf1 600.4
 tpwr 55 rfp 0
 pw 8.600 rp -102.1
 DECOUPLER lp -71.5
 PLOT
 dn 0 wc 250
 dof 0
 dm nnn sc 0
 dnm c vs 681
 dnr 45 th 6
 dmf 13100 ai cdc ph

CS 98

exp1 s2pu1

SAMPLE		SPECIAL	
date	Aug 31 2006	temp	not used
solvent	D2O	gain	not used
file		spin	20
ACQUISITION		exp	0.008
sw	4803.1	pw30	17.200
at	1.984	alfa	20.000
np	not used	il	n
fb	16	in	n
bs	4	dp	y
ss	1.000	hs	nn
d1	8	fn	not used
nt	8	fn	not used
ct	8	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-60.0
sfrq	300.054	wp	3060.5
tof	300.9	rfl	601.2
tpwr	55	rfl	0
pw	8.600	rp	-90.9
dn	C13	lp	-83.9
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	853
dpwr	45	th	2
dmf	13100	ai	cdc
		ph

INDEX 1 FREQUENCY 3691.505 PPM 30.392 HEIGHT 24.0