Supporting Information

Palladium-Catalyzed Dehydrative Allylation of Hypophosphorous Acid with Allylic Alcohols

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General Chemistry. ^1H NMR spectra were recorded on a 300 MHz spectrometer. 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 spectra were recorded 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). Mass spectrometry was provided by the Mass Spectrometry Facility of the University of South Carolina. 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. Organic solutions of products were dried over anhydrous MgSO_4 .

Reagents and Solvents. Aqueous hypophosphorous acid (50 wt. %), was obtained from Aldrich and used as received. Concentrated hypophosphorous acid was obtained by concentrating the 50 wt. % aqueous solution in vacuo on a rotary evaporator, at room temperature for 20-30 min before reaction. DMF was dried over 4A molecular sieves or used as received.

Gas Chromatography. Gas chromatographic analyses were conducted on a gas chromatograph equipped with capillary GC and FID detection, and with mesitylene as an internal standard. The column was a 30 m HP5 (crosslinked 5% PH ME siloxane) capillary column ($0.25\ \mu\text{m}$, phase ratio 320, ID 0.32 mm). Conditions: oven $60\ ^\circ\text{C}$, initial time: 1 min, rate1: $5\ ^\circ\text{C}/\text{min}$, final temperature1: $160\ ^\circ\text{C}$, final time 1: 0 min, then rate2: $25\ ^\circ\text{C}/\text{min}$, final temperature2: $280\ ^\circ\text{C}$, final time 2: 20 min. Inlet $200\ ^\circ\text{C}$, detector $280\ ^\circ\text{C}$, split mode with constant make-up. For GC analysis, 3 drops of sample are diluted in 1 mL of diethyl ether. The solution is washed with 1 mL of 2N NaOH, and $1\ \mu\text{L}$ of the organic solution is injected in the GC. For each sampling, the area of the different peaks are normalized with the area of the mesitylene peak.

^{31}P NMR Yield Measurements. The NMR yields are determined by integration of all the resonances in the ^{31}P NMR spectra, an approach which is valid if no phosphorus-containing gas (ie. PH_3) evolves, or if the precipitate in a heterogeneous mixture does not contain phosphorus. The yields determined by NMR are generally accurate within $\sim 10\%$ of the value indicated, and are reproducible. Isolated yields are sometimes significantly lower because of the H-phosphinate esters are polar compounds and hydrolytically labile.

General Procedure for the Cross-Coupling of Allylic Alcohols with H_3PO_2 .

Concentrated or 50% aqueous H_3PO_2 (0.132 g, 2-6 mmol) and allylic alcohol (2 mmol), were dissolved in DMF (10 ml). The palladium catalyst and the ligand (0.05-2.0 mol % relative to the allylic alcohol) were added at room temperature and the reaction was heated at $85\ ^\circ\text{C}$, either open to air or under N_2 until completion of the reaction (NMR monitoring on a sample of the crude reaction mixture). The reaction mixture was then concentrated under reduced pressure to evaporate the DMF. The residue was diluted with EtOAc and washed with 2 M aqueous HCl and then brine. Drying on MgSO_4 and concentration afforded the product in more than 95% purity.

Representative Experimental Procedures.

Representative Procedure for the Cross-Coupling of Allylic Alcohols with H_3PO_2 using $\text{Pd}_2\text{dba}_3/\text{xantphos}$: Preparation of Cinnamylphosphinic acid (Table 1, entry 1a).

To a solution of concentrated H_3PO_2 (0.132 g, 2 mmol) in dried DMF (10 mL, 0.2 M) was added cinnamyl alcohol (0.274 g, 2 mmol) followed by Pd_2dba_3 (0.0046 g, 0.005 mmol) and xantphos (0.0058 g, 0.01 mmol), at room temperature. The reaction mixture was heated at 85 °C under N_2 for 1.5 h. After cooling to room temperature, ^{31}P NMR analysis showed the product at 26.9 ppm (100%). The mixture was then concentrated under high vacuum. The residue was diluted with EtOAc and washed successively with 2 M aqueous HCl (1 x). The aqueous phase was extracted with EtOAc (2 x) and the combined organic fractions were washed with brine. Drying and concentration afforded the pure product as a light yellow solid (0.346 g, 95%).

Representative Procedure for One-Pot, Two-Steps: Cross-Coupling–Esterification: Preparation of Butyl Allyl Phosphinate (Table 1, entry 9).^{1,2}

A solution of concentrated H_3PO_2 (1.19 g, 18 mmol) in dried DMF (10 mL) was placed in a pressure tube. Allyl alcohol (0.41 mL, 6 mmol), Pd_2dba_3 (0.0550 g, 0.06 mmol) and xantphos (0.0692 g, 0.12 mmol) were then added in that order. The mixture was heated at 85 °C for 3 h. To the reaction mixture was added $(\text{BuO})_4\text{Si}$ (5.77 g, 18 mmol) at room temperature. The resulting mixture was heated at 85 °C for 14 h. After cooling to room temperature, ^{31}P NMR analysis showed the product at 37.2 ppm (63%). The solution was diluted with EtOAc and washed with 2 M aqueous HCl (1 x). The aqueous phase was extracted with EtOAc (2 x) and the combined organic fractions were washed with saturated aqueous NaHCO_3 and brine. Drying over MgSO_4 , concentration and purification by column chromatography over silica gel (hexanes/EtOAc 1:1, v/v, EtOAc) afforded the product as a colorless oil (0.418 g, 43%).

Representative Procedure for the Cross-Coupling of Allylic Alcohols with H_3PO_2 using $\text{Pd}_2\text{dba}_3/\text{Polystyrene-supported nixantphos 6}$.³

Multi-run reaction. To a solution of concentrated H_3PO_2 (0.264 g, 4 mmol) in DMF (10 mL) was added cinnamyl alcohol (0.274 g, 2 mmol), Pd_2dba_3 (0.0092 g, 0.01 mmol) and supported nixantphos³ (0.18 mmol/g, 0.111 g, 0.02 mmol). The reaction mixture was heated at 85 °C under N_2 for 4 h. The mixture was suction-filtered in order to separate the resin from the filtrate. The resin was washed with DMF and kept apart to be used in the next run without further precautions. The filtrates were collected and stored in a stoppered flask. The procedure above was repeated for four more times. The resin was filtered, washed and reused each time for the next run. At the end of the sequence, all the filtrates were combined and concentrated under high vacuum. The residue was diluted with EtOAc and washed with 2 M aqueous HCl (1x). The aqueous phase was extracted twice with EtOAc and the combined organic fractions were washed with brine. After drying over MgSO_4 and concentration, the product was obtained as a white solid (1.67 g, 92% over 5 runs).

Representative Procedure for the Rearrangement of Allylic Hypophosphite Esters: Preparation of Butyl Cinnamyl Phosphinate by Rearrangement of Cinnamyl Hypophosphite.

To a solution of concentrated H_3PO_2 (0.132 g, 2 mmol) in dried DMF (10 mL) was added $(\text{PhO})_4\text{Si}^4$ (0.800 g, 2 mmol) and cinnamyl alcohol (0.514 mL, 4 mmol). The solution was

heated at 85 °C for 2 h. After cooling to room temperature, ^{31}P NMR analysis showed the cinnamyl hypophosphite ester at 14.32 ppm (76%). To the reaction mixture was added Pd_2dba_3 (0.0092 g, 0.01 mmol) and xantphos (0.0115 g, 0.02 mmol). The resulting mixture was heated at 85 °C for 2 h or stirred for 4 h at room temperature. The solution was then concentrated in vacuo and the residue was diluted with EtOAc and washed with 2 M aqueous HCl (1 x). The aqueous phase was extracted with EtOAc (2 x) and the combined organic fractions were washed with brine, dried over MgSO_4 and concentrated. The residue was dissolved in toluene (10 mL), followed by addition of $(\text{BuO})_4\text{Si}$ (0.641 g, 2 mmol). The resulting mixture was stirred at reflux for 12 h, then diluted with EtOAc and washed with brine. Drying, concentration and purification by radial chromatography (4 mm thickness, hexanes/EtOAc 5:1, v/v, EtOAc), afforded the product, butyl cinnamyl phosphinate^{2,5} as a yellow oil (0.286 g, 60%).

Spectral Data

Cinnamyl phosphinic acid (Table 1, entry 1). m.p. = 64 °C; ^1H NMR (CDCl_3 , 300 MHz) δ 11.26 (bs, 1H), 7.15 – 7.43 (m, 5H), 7.01 (d, J_{HP} = 558 Hz, 1H), 6.51 (dd, J = 16 Hz, J = 5 Hz, 1H), 6.03 – 6.19 (m, 1H), 2.75 (dd, J_{HP} = 19 Hz, J = 7 Hz, 2H); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 136.7 (d, J_{PCCCC} = 4 Hz), 136.2 (d, J_{PCCC} = 14 Hz), 128.8 (2C), 128.1, 126.6 (d, J_{PCCCCC} = 2 Hz, 2C), 117.0 (d, J_{PCC} = 10 Hz), 34.9 (d, J_{PC} = 90 Hz); ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 35.32 (dm, J_{PH} = 558 Hz); HRMS (EI^+) calcd. for $\text{C}_9\text{H}_{11}\text{O}_2\text{P}$, ($[\text{M}]^+$) 182.0495, found 182.0497.

(trans-Hexen-2-yl) phosphinic acid (Table 1, entries 2a, 2b). ^1H NMR (CDCl_3 , 300 MHz) δ 11.15 (bs, 1H), 6.94 (d, J_{HP} = 554 Hz, 1H), 5.64 (dt, J = 22 Hz, J = 8 Hz, 1H), 5.35 (dt, J = 22 Hz, J = 8 Hz, 1H), 2.57 (dd, J_{HP} = 19 Hz, J = 7 Hz, 2H), 2.03 (t, J = 6 Hz, 2H), 1.39 (sext., J = 7 Hz, 2H), 0.89 (t, J = 7 Hz, 3H); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 137.6 (d, J_{PCCC} = 14 Hz), 116.8 (d, J_{PCC} = 9 Hz), 34.7 (d, J_{PCCCC} = 3 Hz), 34.0 (d, J_{PC} = 91 Hz), 22.3 (d, J_{PCCCCC} = 4 Hz), 13.6; ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 37.15 (dm, J_{PH} = 554 Hz); HRMS (EI^+) calcd. for $\text{C}_6\text{H}_{13}\text{O}_2\text{P}$, ($[\text{M}]^+$) 148.0653, found 148.0657.

(trans-Buten-2-yl) phosphinic acid (Table 1, entry 2c). ^1H NMR (CDCl_3 , 300 MHz) δ 12.13 (bs, 1H), 6.95 (d, J_{HP} = 553 Hz, 1H), 5.64 (dt, J = 15 Hz, J = 6 Hz, 1H), 5.38 (dq, J = 15 Hz, J = 6 Hz, 1H), 2.56 (dd, J_{HP} = 19 Hz, J = 7 Hz, 2H), 1.72 (td, J = 6 Hz, 3H); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 132.1 (d, J_{PCCC} = 14 Hz), 117.9 (d, J_{PCC} = 9 Hz), 33.1 (d, J_{PC} = 92 Hz), 18.2 (d, J_{PCCCC} = 3 Hz); ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 37.78 (dm, J_{PH} = 553 Hz); HRMS (EI^+) calcd. for $\text{C}_4\text{H}_9\text{O}_2\text{P}$, ($[\text{M}]^+$) 120.0340, found 120.0339.

(3-Methyl-buten-2-yl) phosphinic acid (Table 1, entries 3a, 8a). ^1H NMR (CDCl_3 , 300 MHz) δ 10.91 (bs, 1H), 6.94 (d, J_{HP} = 550 Hz, 1H), 5.13 (qq, J = 7 Hz, J = 2 Hz, 1H), 2.57 (dd, J_{HP} = 19 Hz, J = 8 Hz, 2H), 1.77 (d, J = 6 Hz, 3H), 1.66 (d, J = 4 Hz, 3H); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 138.4 (d, J_{PCCC} = 14 Hz), 110.6 (d, J_{PCC} = 9 Hz), 30.1 (d, J_{PC} = 92 Hz), 25.8 (d, J_{PCCCC} = 3 Hz), 18.2 (d, J_{PCCCCC} = 3 Hz); ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 35.86 (dm, J_{PH} = 550 Hz); HRMS (EI^+) calcd. for $\text{C}_5\text{H}_{11}\text{O}_2\text{P}$, ($[\text{M}]^+$) 134.0497, found 134.0494.

(E)-Geranyl phosphinic acid (Table 1, entry 3b). ^1H NMR (CDCl_3 , 300 MHz) δ 11.39 (bs, 1H), 6.94 (dt, J_{HP} = 552 Hz, J = 2 Hz, 1H), 5.0 – 5.21 (m, 2H), 2.60 (dd, J_{HP} = 19 Hz, J = 8 Hz, 2H), 2.0 – 2.18 (m, 4H), 1.68 (s, 3H), 1.66 (d, J = 4 Hz, 3H), 1.60 (s, 3H); ^{13}C NMR (CDCl_3 ,

75.45 MHz) δ 142.3 (d, $J_{\text{PCCC}} = 14$ Hz), 132.0, 124.0, 110.6 (d, $J_{\text{PCC}} = 9$ Hz), 39.9 (d, $J_{\text{PCCCC}} = 3$ Hz), 30.2 (d, $J_{\text{PC}} = 92$ Hz), 26.7 (d, $J_{\text{PCCCC}} = 4$ Hz), 25.9, 17.9, 16.7 (d, $J_{\text{PCCCC}} = 3$ Hz); ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 37.10 (dm, $J_{\text{PH}} = 552$ Hz); HRMS (EI^+) calcd. for $\text{C}_{10}\text{H}_{19}\text{O}_2\text{P}$, ($[\text{M}]^+$) 202.1123, found 202.1122.

(*E, E*)-Farnesyl phosphinic acid (Table 1, entry 3c). ^1H NMR (CDCl_3 , 300 MHz) δ 10.92 (bs, 1H), 6.94 (d, $J_{\text{HP}} = 548$ Hz, 1H), 5.0 – 5.22 (m, 3H), 2.59 (dd, $J_{\text{HP}} = 19$ Hz, $J = 7$ Hz, 2H), 1.93 – 2.16 (m, 9H), 1.63 – 1.73 (m, 6H), 1.57 – 1.63 (m, 5H); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 142.0 (d, $J_{\text{PCCC}} = 14$ Hz), 135.4, 131.3, 124.3, 123.7, 110.4 (d, $J_{\text{PCC}} = 9$ Hz), 39.8 (d, $J_{\text{PCCCC}} = 3$ Hz), 39.7, 30.2 (d, $J_{\text{PC}} = 92$ Hz), 26.7, 26.4 (d, $J_{\text{PCCCC}} = 4$ Hz), 25.7, 17.7, 16.6 (d, $J_{\text{PCCCC}} = 3$ Hz), 16.0; ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 36.72 (dm, $J_{\text{PH}} = 548$ Hz); HRMS (EI^+) calcd. for $\text{C}_{15}\text{H}_{27}\text{O}_2\text{P}$, ($[\text{M}]^+$) 270.1754, found 270.1749.

(*E*)-2-Methyl-3-phenyl-propen-2-yl phosphinic acid (Table 1, entry 4). m.p. = 62 °C; ^1H NMR (CDCl_3 , 300 MHz) δ 11.15 (bs, 1H), 7.16 – 7.36 (m, 5H), 7.11 (d, $J_{\text{HP}} = 558$ Hz, 1H), 6.39 (d, $J = 6$ Hz, 1H), 2.73 (d, $J_{\text{HP}} = 19$ Hz, 2H), 1.98 (dd, $J = 4$ Hz, $J = 2$ Hz, 3H); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 137.3 (d, $J_{\text{PCCCC}} = 4$ Hz), 130.4 (d, $J_{\text{PCC}} = 12$ Hz), 128.9 (d, $J_{\text{PCCCC}} = 3$ Hz, 2C), 128.1, 127.6 (d, $J_{\text{PCCC}} = 11$ Hz), 126.6 (2C), 41.6 (d, $J_{\text{PC}} = 89$ Hz), 19.6 (d, $J_{\text{PCCC}} = 3$ Hz); ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 37.87 (dm, $J_{\text{PH}} = 558$ Hz); HRMS (EI^+) calcd. for $\text{C}_{10}\text{H}_{13}\text{O}_2\text{P}$, ($[\text{M}]^+$) 196.0653, found 196.0654.

6,6-Dimethyl bicyclo(3.1.1)hept-2-ene-2-(methyl phosphinic acid) (Myrtenyl phosphinic acid) (Table 1, entry 5). ^1H NMR (CDCl_3 , 300 MHz) δ 11.61 (bs, 1H), 6.96 (d, $J_{\text{HP}} = 553$ Hz, 1H), 5.40 – 5.52 (m, 1H), 2.57 (d, $J_{\text{HP}} = 19$ Hz, 2H), 2.34 – 2.44 (m, 1H), 2.20 – 2.34 (m, 2H), 2.16 (t, $J = 6$ Hz, 1H), 2.06 – 2.13 (m, 2H), 1.28 (s, 3H), 0.87 (s, 3H); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 136.9 (d, $J_{\text{PCC}} = 10$ Hz), 122.9 (d, $J_{\text{PCCC}} = 14$ Hz), 46.7 (d, $J_{\text{PCCC}} = 3$ Hz), 40.2, 38.5 (d, $J_{\text{PC}} = 91$ Hz), 38.2 (d, $J_{\text{PCCCC}} = 3$ Hz), 31.6 (d, $J_{\text{PCCCC}} = 3$ Hz), 31.5 (d, $J_{\text{PCCCC}} = 4$ Hz), 26.2, 21.2; ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 36.59 (dm, $J_{\text{PH}} = 553$ Hz); HRMS (EI^+) calcd. for $\text{C}_{10}\text{H}_{17}\text{O}_2\text{P}$, ($[\text{M}]^+$) 200.0966, found 200.0964.

(*trans*-Penten-2-yl) phosphinic acid (Table 1, entry 6a). ^1H NMR (CDCl_3 , 300 MHz) δ 10.85 (bs, 1H), 6.95 (d, $J_{\text{HP}} = 555$ Hz, 1H), 5.69 (dt, $J = 21$ Hz, $J = 6$ Hz, 1H), 5.35 (dt, $J = 21$ Hz, $J = 7$ Hz, 1H), 2.57 (dd, $J_{\text{HP}} = 19$ Hz, $J = 7$ Hz, 2H), 2.07 (q, $J = 7$ Hz, 2H), 0.99 (t, $J = 7$ Hz, 3H); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 139.4 (d, $J_{\text{PCCC}} = 14$ Hz), 115.5 (d, $J_{\text{PCC}} = 9$ Hz), 34.0 (d, $J_{\text{PC}} = 92$ Hz), 25.8 (d, $J_{\text{PCCCC}} = 3$ Hz), 13.4 (d, $J_{\text{PCCCC}} = 3$ Hz); ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 37.53 (dm, $J_{\text{PH}} = 555$ Hz); HRMS (EI^+) calcd. for $\text{C}_5\text{H}_{11}\text{O}_2\text{P}$, ($[\text{M}]^+$) 134.0497, found 134.0495.

[3-(4-Fluoro-phenyl)-propen-2-yl] phosphinic acid (Table 1, entry 6b). m.p. = 99 – 101 °C; ^1H NMR (CDCl_3 , 300 MHz) δ 12.58 (bs, 1H), 7.30 (dd, $J = 8$ Hz, $J = 5$ Hz, 2H), 7.01 (d, $J_{\text{HP}} = 559$ Hz, 1H), 6.98 (t, $J = 9$ Hz, 2H), 6.48 (dd, $J = 16$ Hz, $J = 5$ Hz, 1H), 5.90 – 6.12 (m, 1H), 2.74 (dd, $J_{\text{HP}} = 19$ Hz, $J = 7$ Hz, 2H); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 162.6 (d, $J_{\text{FC}} = 247$ Hz), 135.0 (d, $J_{\text{PCCC}} = 14$ Hz), 132.9, 128.1 (d, $J_{\text{FCCC}} = 8$ Hz, 2C), 116.6 (d, $J_{\text{PCC}} = 9$ Hz), 115.7 (d, $J_{\text{FCC}} = 22$ Hz, 2C), 34.6 (d, $J_{\text{PC}} = 90$ Hz); ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 36.91 (dm, $J_{\text{PH}} = 559$ Hz); HRMS (EI^+) calcd. for $\text{C}_9\text{H}_{10}\text{FO}_2\text{P}$, ($[\text{M}]^+$) 200.0408, found 200.0402.

(2-Cyclohexyl-ethen-2-yl) phosphinic acid (Table 1, entry 7a). ^1H NMR (CDCl_3 , 300 MHz) δ 10.96 (bs, 1H), 6.93 (d, $J_{\text{HP}} = 552$ Hz, 1H), 5.07 (q, $J = 7$ Hz, 1H), 2.59 (dd, $J_{\text{HP}} = 19$ Hz, $J = 8$ Hz, 2H), 2.08 – 2.21 (m, 4H), 1.47 – 1.62 (m, 6H); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 146.9 (d, $J_{\text{PCCC}} = 14$ Hz), 107.0 (d, $J_{\text{PCC}} = 9$ Hz), 37.4 (d, $J_{\text{PCCCC}} = 3$ Hz), 29.4 (d, $J_{\text{PC}} = 92$ Hz), 29.3 (d, $J_{\text{PCCCC}} = 3$ Hz), 28.7 (d, $J_{\text{PCCCC}} = 3$ Hz), 28.0 (d, $J_{\text{PCCCC}} = 2$ Hz), 26.8; ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 35.93 (ddd, $J_{\text{PH}} = 552$ Hz, $J = 35$ Hz, $J = 2$ Hz); HRMS (EI^+) calcd. for $\text{C}_9\text{H}_{15}\text{O}_2\text{P}$, ($[\text{M}]^+$) 174.0810, found 174.0809.

2-[(N-Ethylloxycarbonyl-4-piperidinyl)-ethen-2-yl] phosphinic acid (Table 1, entry 7b). ^1H NMR (CDCl_3 , 300 MHz) δ 11.43 (bs, 1H), 6.97 (d, $J_{\text{HP}} = 550$ Hz, 1H), 5.18 – 5.30 (m, 1H), 4.14 (q, $J = 7$ Hz, 2 H), 3.38 – 3.55 (m, 4H), 2.63 (dd, $J_{\text{HP}} = 19$ Hz, $J = 8$ Hz, 2H), 2.13 – 2.31 (m, 4H), 1.27 (t, $J = 7$ Hz, 3H); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 155.7, 141.9 (d, $J_{\text{PCCC}} = 14$ Hz), 110.2 (d, $J_{\text{PCC}} = 9$ Hz), 61.7, 45.6, 44.7, 36.1, 29.4 (d, $J_{\text{PC}} = 91$ Hz), 28.8, 14.9; ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 35.27 (dm, $J_{\text{PH}} = 550$ Hz); HRMS (EI^+) calcd. for $\text{C}_{10}\text{H}_{18}\text{NO}_4\text{P}$, ($[\text{M}]^+$) 247.0973, found 247.0981.

Geranyl phosphinic acid (*E/Z* 50/50) (Table 1, entry 8b). ^1H NMR (CDCl_3 , 300 MHz) δ 10.10 (bs, 1H), 6.93 (d, $J_{\text{HP}} = 552$ Hz, 1H), 5.02 – 5.21 (m, 2H), 2.58 (dd, $J_{\text{HP}} = 19$ Hz, $J = 8$ Hz, 2H), 2.06 (d, $J = 4$ Hz, 4H), 1.76 and 1.65 [(d, $J = 5$ Hz) and (d, $J = 4$ Hz), 3H], 1.68 (s, 3H), 1.60 (s, 3H); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 142.2 (d, $J_{\text{PCCC}} = 14$ Hz), 142.1 (d, $J_{\text{PCCC}} = 14$ Hz), 132.4, 132.0, 123.9, 123.8, 111.3 (d, $J_{\text{PCC}} = 9$ Hz), 110.6 (d, $J_{\text{PCC}} = 9$ Hz), 39.9 (d, $J_{\text{PCCCC}} = 3$ Hz), 32.3 (d, $J_{\text{PCCCC}} = 3$ Hz), 30.2 (d, $J_{\text{PC}} = 92$ Hz), 30.1 (d, $J_{\text{PC}} = 92$ Hz), 26.7 (d, $J_{\text{PCCCC}} = 4$ Hz), 26.5 (d, $J_{\text{PCCCC}} = 3$ Hz), 25.9, (2C), 23.7 (d, $J_{\text{PCCCC}} = 3$ Hz), 17.9, 17.8, 16.7 (d, $J_{\text{PCCCC}} = 3$ Hz); ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 36.42 (dm, $J_{\text{PH}} = 552$ Hz), 36.17 (dm, $J_{\text{PH}} = 552$ Hz); HRMS (EI^+) calcd. for $\text{C}_{10}\text{H}_{19}\text{O}_2\text{P}$, ($[\text{M}]^+$) 202.1123, found 202.1127.

Farnesyl phosphinic acid (*E/Z* 50/50, *E/Z*) (Table 1, entry 8c).

^1H NMR (CDCl_3 , 300 MHz) δ 11.29 (bs, 1H), 6.94 (d, $J_{\text{HP}} = 550$ Hz, 1H), 5.03 – 5.21 (m, 3H), 2.59 (dd, $J_{\text{HP}} = 19$ Hz, $J = 7$ Hz, 2H), 1.92 – 2.18 (m, 9H), 1.56 – 1.83 (m, 12H); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 142.3 (d, $J_{\text{PCCC}} = 14$ Hz, 2C), 142.2 (d, $J_{\text{PCCC}} = 14$ Hz, 2C), 136.1, 136.0, 135.8, 135.6, 131.8, 131.7, 131.6, 131.5, 124.7, 124.6, 124.5, 124.4, 123.9 (2C), 123.7 (2C), 111.4 (d, $J_{\text{PCC}} = 9$ Hz), 111.3 (d, $J_{\text{PCC}} = 9$ Hz), 110.7 (d, $J_{\text{PCC}} = 7$ Hz), 110.6 (d, $J_{\text{PCC}} = 8$ Hz), 40.2 (d, $J_{\text{PCCCC}} = 3$ Hz, 2C), 39.9 (2C), 32.5 (d, $J_{\text{PCCCC}} = 3$ Hz), 32.3 (d, $J_{\text{PCCCC}} = 3$ Hz), 32.2, 32.1, 30.3 (d, $J_{\text{PC}} = 92$ Hz, 2C), 30.2 (d, $J_{\text{PC}} = 92$ Hz, 2C), 27.0, 26.9, 26.8 (2C), 26.7 (d, $J_{\text{PCCCC}} = 4$ Hz), 26.5 (d, $J_{\text{PCCCC}} = 3$ Hz), 26.4 (d, $J_{\text{PCCCC}} = 3$ Hz), 26.3 (d, $J_{\text{PCCCC}} = 3$ Hz), 26.0 (2C), 25.9 (2C), 23.8 (d, $J_{\text{PCCCC}} = 3$ Hz, 2C), 23.6 (2C), 17.9, 17.8, 16.8 (d, $J_{\text{PCCCC}} = 3$ Hz), 16.7 (d, $J_{\text{PCCCC}} = 3$ Hz), 16.2 (4C); ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 35.79 (dm, $J_{\text{PH}} = 548$ Hz); HRMS (EI^+) calcd. for $\text{C}_{15}\text{H}_{27}\text{O}_2\text{P}$, ($[\text{M}]^+$) 270.1754, found 270.1749.

Butyl allyl phosphinate (Table 1, entry 9).^{1,2} ^1H NMR (CDCl_3 , 300 MHz) δ 7.00 (dt, $J_{\text{HP}} = 543$ Hz, $J = 2$ Hz, 1H), 5.68 – 5.82 (m, 1H), 5.20 – 5.31 (m, 2H), 4.03 and 4.13 (dtd, $J = 10$ Hz, $J = 9$ Hz, $J_{\text{HP}} = 7$ Hz, 2H), 2.66 (ddd, $J_{\text{HP}} = 19$ Hz, $J = 8$ Hz, $J = 2$ Hz, 2H), 1.64 – 1.74 (m, 2H), 1.42 (sext., $J = 8$ Hz, 2H), 0.95 (t, $J = 8$ Hz, 3H); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 125.9 (d, $J_{\text{PCC}} = 9$ Hz), 121.5 (d, $J_{\text{PCCC}} = 14$ Hz), 66.5 (d, $J_{\text{POC}} = 8$ Hz), 35.0 (d, $J_{\text{PC}} = 90$ Hz), 32.6 (d, $J_{\text{POCC}} = 6$ Hz), 18.9, 13.8; ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 37.79 (dm, $J_{\text{PH}} = 543$ Hz); HRMS (EI^+) calcd. for $\text{C}_7\text{H}_{15}\text{O}_2\text{P}$, ($[\text{M}]^+$) 163.0888, found 163.0883.

Butyl (cyclohexen-2-yl) phosphinate (Table 1, entry 10a). ^1H NMR (CDCl_3 , 300 MHz) δ 6.87 (d, $J_{\text{HP}} = 529$ Hz, 1H), 5.93 – 6.07 (m, 1H), 5.57 – 5.79 (m, 1H), 3.95 – 4.21 (m, 2H), 2.59 (d, $J_{\text{HP}} = 25$ Hz, 1H), 1.11 – 2.12 (m, 10H), 0.95 (t, $J = 7$ Hz, 3H); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 132.6 (d, $J_{\text{PCCC}} = 13$ Hz), 132.5 (d, $J_{\text{PCCC}} = 12$ Hz), 119.8 (d, $J_{\text{PCC}} = 7$ Hz), 119.6 (d, $J_{\text{PCC}} = 6$ Hz), 66.5 (d, $J_{\text{POC}} = 7$ Hz), 66.4 (d, $J_{\text{POC}} = 8$ Hz), 36.5 (d, $J_{\text{PC}} = 94$ Hz), 36.4 (d, $J_{\text{PC}} = 94$ Hz), 32.6 (d, $J_{\text{POCC}} = 6$ Hz, 2C), 24.8 (d, $J_{\text{PCC}} = 4$ Hz), 24.7 (d, $J_{\text{PCC}} = 4$ Hz), 21.4, 21.3, 20.7 (d, $J_{\text{PCCC}} = 9$ Hz), 20.6 (d, $J_{\text{PCCC}} = 9$ Hz), 19.0 (2C), 13.8 (2C); ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 42.71 (dm, $J_{\text{PH}} = 529$ Hz), 41.94 (dm, $J_{\text{PH}} = 529$ Hz); HRMS (EI^+) calcd. for $\text{C}_{10}\text{H}_{19}\text{O}_2\text{P}$, ($[\text{M}]^+$) 202.1123, found 202.1118.

3,5,5-Trimethyl-cyclohexen-2-yl phosphinic acid (Table 1, entry 10b). ^1H NMR (CDCl_3 , 300 MHz) δ 12.24 (bs, 1H), 6.76 (dd, $J_{\text{HP}} = 544$ Hz, $J = 3$ Hz, 1H), 5.40 (d, $J = 10$ Hz, 1H), 2.44 – 2.70 (m, 1H), 1.79 – 1.92 (m, 1H), 1.69 (s, 3H), 1.52 – 1.66 (m, 1H), 1.23 – 1.41 (m, 2H), 1.01 (s, 3H), 0.85 (s, 3H); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 137.4 (d, $J_{\text{PCCC}} = 14$ Hz), 111.1 (d, $J_{\text{PCC}} = 5$ Hz), 42.7 (d, $J_{\text{PCC}} = 3$ Hz), 34.5 (d, $J_{\text{PC}} = 98$ Hz), 32.1, 30.4, 28.0 (d, $J_{\text{PCCC}} = 13$ Hz), 23.8, 23.2 (d, $J_{\text{PCCC}} = 2$ Hz); ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 42.82 (dm, $J_{\text{PH}} = 544$ Hz); HRMS (EI^+) calcd. for $\text{C}_9\text{H}_{17}\text{O}_2\text{P}$, ($[\text{M}]^+$) 188.0966, found 188.0968.

((E)-1-Methyl-buten-2-yl) phosphinic acid (Equation 2, E isomer). ^1H NMR (CDCl_3 , 300 MHz) δ 11.9 (bs, 1H), 6.81 (d, $J_{\text{HP}} = 547$ Hz, 1H), 5.64 (ddd, $J = 22$ Hz, $J = 11$ Hz, $J = 6$ Hz, 1H), 5.38 (dddd, $J = 22$ Hz, $J = 8$ Hz, $J = 6$ Hz, $J = 2$ Hz, 1H), 2.52 (dq, $J_{\text{HP}} = 25$ Hz, $J = 8$ Hz, 1H), 1.72 (dd, $J = 7$ Hz, $J = 6$ Hz, 3H), 1.26 (dd, $J_{\text{HP}} = 19$ Hz, $J = 7$ Hz, 3H); ^{13}C NMR (CDCl_3 , 75.45 MHz) δ 129.9 (d, $J_{\text{PCCC}} = 13$ Hz), 125.4 (d, $J_{\text{PCC}} = 7$ Hz), 37.7 (d, $J_{\text{PC}} = 92$ Hz), 18.4 (d, $J_{\text{PCCCC}} = 3$ Hz), 11.54 (d, $J_{\text{PCC}} = 3$ Hz); ^{31}P NMR (CDCl_3 , 121.47 MHz) δ 42.56 (dm, $J_{\text{PH}} = 547$ Hz); HRMS (EI^+) calcd. for $\text{C}_5\text{H}_{11}\text{O}_2\text{P}$, ($[\text{M}]^+$) 134.0497, found 134.0499.

References

- 1 Kabachnik, M. I.; Chang, J.-Y.; Tsvetkov, E. N. *J. Gen. Chem. USSR* **1962**, 32, 3351.
- 2 Abrunhosa-Thomas, I.; Ribiere, P.; Adcock, A.; Montchamp, J.-L. *Synthesis* **2006**, 325.
- 3 Deprère, S.; Montchamp, J.-L. *Org. Lett.* **2004**, 6, 3805.
- 4 Malatesta, L. *Gazz. Chim. Ital.* **1948**, 78, 747.
- 5 Bravo-Altamirano, K.; Huang, Z.; Montchamp, J.-L. *Tetrahedron* **2005**, 61, 6315.

Table 1, entry 1
31-P/1-H DECOUPLED

```

694
exp1 s2pul1
SAMPLE
date Oct 17 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 alfa 20.000
np 85476
fb 14800 il n
bs 64 in n
ss 4 db y
d1 1.000 hs nm
nt 8 PROCESSING
ct 8 lb 2.00
TRANSMITTER p31 fn not used
tn 121.474 sp DISPLAY
sfrq 106062 wp 10010.0
tof 55 rfi 2437.3
pw 7.117 rfp 0
DECOUPLER H1 lp -156.5
dn 0 -288.8
dof 0 PLOT
dm yyi wc 250
dmm w sc 0
dpwr 35 vs 44
dmf 6700 th 5
ai no ph

```

Table 1, entry 1
31-P/1-H COUPLED

```

694
exp1 s2pu1
SAMPLE
date Oct 17 2005 temp not used
solvent CDCl3 gain not used
file 0.008
ACQUISITION exp 20
sw 26738.0 ppg80 18.300
at 1.598 aifa 20.000
fb 85476 il
bs 14800 il n
ss 64 in n
di 4 dp y
nt 1.000 hs nn
ct 8 PROCESSING 2.00
TRANSMITTER p31 lb not used
tn 121.474 sp DISPLAY 0
tof 10608.2 wp 10010.0
tpwr 55 rfl 2437.3
pw 7.117 rfp 94.9
DECOUPLER H1 lp 53.1
dn dof 0 PLOT 250
dm ynn wc 0
dmm w sc 78
dpr 35 vs 12
dmf 6700 al no ph

```

694

exp1 s2pu1

SAMPLE
 date Oct 17 2005
 solvent CDC13
 file
 sw ACQUISITION exp
 at 4803.1 hst 0.008
 np 1.994 pw90 17.200
 fb 18158 alfa 20.000
 bs not used fl
 ss 16 in n
 nt 4 dp n
 ct 1.000 hs y
 8 fn
 PROCESSING mn
 8
 TRANSMITTER
 tn H1 sp
 sfrq 300.053 wp -80.0
 tof 300.9 rfl 3080.5
 tpwr 55 rfp 604.8
 pw 8.600 rp 0
 DECOUPLER C13 lp
 dn -108.7
 dof -81.6
 dm 0 WC
 dmm nnn 250
 dpwr C SC
 dmf 45 VS
 13100 at cdc ph 2

SPECIAL
 not used
 gain not used
 spin 20
 nst 0.008
 pw90 17.200
 alfa 20.000
 FLAGS
 n
 n
 y
 mn
 DISPLAY
 -80.0
 3080.5
 604.8
 0
 -108.7
 -81.6
 PLOT
 250
 0
 675
 2

Table 1, entry 1

Table 1, entry 1

INDEX	FREQUENCY	PPM	HEIGHT
1	3378.334	11.259	13.2
2	2383.068	7.942	27.4
3	2207.761	7.358	44.4
4	2200.432	7.334	95.4
5	2195.155	7.316	51.2
6	2187.826	7.292	74.2
7	2180.204	7.266	43.5
8	2174.634	7.248	47.5
9	2167.891	7.225	28.9
10	1963.855	6.545	16.0
11	1956.578	6.527	15.9
12	1948.024	6.492	20.6
13	1942.747	6.475	20.2
14	1846.299	6.153	6.3
15	1838.970	6.129	16.4
16	1824.605	6.081	40.2
17	1815.811	6.052	13.5
18	1808.775	6.028	4.9
19	836.861	2.789	32.2
20	829.633	2.765	31.7
21	817.613	2.725	32.4
22	810.284	2.700	30.8
23	779.503	2.598	9.3

30.4

30.4

30.4

30.4

INDEX	FREQUENCY	PPM	HEIGHT
1	10319.186	136.772	25.4
2	10315.156	136.719	23.6
3	10284.062	136.307	36.7
4	10269.667	136.116	34.0
5	9721.215	128.847	126.0
6	9663.058	128.076	59.8
7	9549.337	126.569	100.3
8	9547.322	126.542	87.1
9	8829.583	117.029	32.2
10	8819.507	116.895	32.1
11	5864.484	77.729	90.0
12	5832.527	77.305	91.7
13	5800.570	76.882	89.5
14	2676.548	35.475	29.2
15	2586.147	34.277	29.2

Table 1, entry 1

Table 1, entry 6a
31-P/1-H DECOUPLED

```

724
exp1 s2put
SAMPLE
date Oct 27 2005 temp not used
solvent CDCl3 gain not used
file 0.008
ACQUISITION exp hst 18.300
SW 28738.0 pw50 20.000
at 1.598 alfa FLAGS
np 85476 il n
fb 14800 in n
bs 64 in y
ss 4 dp y
dl 1.000 hs nn
nt 8
ct 8 lb 2.00
TRANSMITTER p31 fn not used
tn 121.474 sp DISPLAY
sfrq 10608.2 wp 10010.0 0
tof 55 rfl 2437.3
tpwr 7.117 rfp 0
pw DECOUPLER H1 lp 131.1
dn dof H1 lp -288.8
dm yyw wc PLOT 250
dmm w sc 0
dpwr 35 vs 35
dmf 6700 th no ph 5

```

INDEX	FREQUENCY	PPM	HEIGHT
1	4837.515	39.827	103.5
2	4281.834	35.252	107.8

Table 1, entry 6a
31-P/1-H COUPLED

Table 1, entry 6a

```

724
exp1 s2pu1
SAMPLE
date Oct 27 2005
solvent CDCl3
file exp
sw ACQUISITION
at 4803.1
fb 1.994
fs 19158
nt not used
di 16
nt 4
ct 1.000
hs 8
fn not used
PROCESSING
tn H1
sfrq 300.053
tof 300.9
tpwr 55
pw 8.600
DECOUPLER C13
dn 0
dof 0
dm nnn
dmm SC
dpwr C
dmf 45
13100 ai cdc ph
SPECIAL
temp not used
gain not used
spn 20
hst 0.008
pws0 17.200
alfa 20.000
FLAGS
n
n
y
nm
DISPLAY
-60.0
3060.5
596.6
0
-123.0
-39.9
PLOT
wc 250
nnn SC
vs 160
th 45
ai cdc ph

```

Table 1, entry 6a

1H NMR

INDEX	FREQUENCY	PPM	HEIGHT
1	3254.036	10.845	6.6
2	2361.375	7.870	17.0
3	2182.256	7.273	10.2
4	1807.309	6.023	17.3
5	1724.053	5.746	3.7
6	1717.896	5.725	9.1
7	1711.740	5.705	10.1
8	1702.652	5.675	12.0
9	1696.496	5.654	11.4
10	1690.340	5.634	3.9
11	1622.034	5.406	4.1
12	1615.585	5.384	10.0
13	1608.256	5.360	12.2
14	1600.634	5.335	10.3
15	1592.425	5.307	19.6
16	1591.253	5.303	31.1
17	1586.562	5.288	2.5
18	1585.683	5.285	2.6
19	904.387	3.014	2.5
20	783.607	2.612	20.4
21	776.278	2.587	20.7
22	764.552	2.548	20.0
23	757.223	2.524	19.8
24	639.081	2.130	3.3
25	631.752	2.105	14.1
26	625.009	2.083	26.7
27	618.267	2.061	28.1
28	611.524	2.038	15.2
29	604.782	2.016	4.3
30	523.870	1.746	3.5
31	376.706	1.255	7.4
32	304.883	1.016	77.6
33	297.554	0.992	162.0
34	289.932	0.966	71.1
35	279.378	0.931	3.3
36	277.619	0.925	2.9
37	272.635	0.909	2.7

724

exp1 s2bu1

SAMPLE		SPECIAL	
date	Oct 27 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	sp1n	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	1000	PROCESSING	
ct	1000	lb	1.00
TRANSMITTER C13		fn	not used
tn	75.456	sp	-150.9
sfrq	737.2	wp	15844.1
tof	58	rfl	1151.6
tpwr	9.250	rpf	0
DECOUPLER		rp	78.6
dn	H1	lp	-285.9
dof	0	PLOT	
dm	YVY	wc	250
dmm	w	sc	0
dpwr	35	vs	290
dmf	6700	th	8
		ai	no
		ph

Table 1, entry 6a

Table 1, entries 2a and 2b
31-P/1-H DECOUPLED

```

723
exp1 s2pu1
SAMPLE
date Oct 27 2005
solvent CDC13
file ACQUISITION
sw 26738.0
at 1.598
np 85476
fb 14800
bs 64
ss 4
d1 1.000
nt 8
ct 8
TRANSMITTER P31
tn 121.474
sfrq 10608.2
tof 55
tpwr 7.117
pw DECOUPLER H1 lp
dn 0
dof YYY WC
dm 35
dmm 35
dpwr 6700
dmf ai no ph
SPECIAL
temp not used
gain not used
spn 20
hst 0.008
pw90 18.300
alpha 20.000
FLAGS
n
n
y
nn
PROCESSING
2.00
lb not used
fn DISPLAY
0
10010.0
2437.3
-165.0
-288.8
PLOT
250
0
51
4

```

SAMPLE			SPECIAL		
date	solvent	exp	temp	gain	not used
file	CDCl3	exp	spin	hst	20
ACQUISITION			26738.0	pw90	0.008
sw	at	1.598	alfa	18.300	20.000
mp	fb	85476	il	FLAGS	
nb	14800	64	in		n
bs	64	bs	in		n
ss	64	ss	dp		y
d1	1.000	hs	hs		nn
nt	16	fb	PROCESSING		2.00
ct	16	fb	not used		
TRANSMITTER			DISPLAY		
tn	P31	sp	0		
sfreq	121.474	wd	10010.0		
tof	10608.2	rfl	2437.3		
tpwr	55	rfl	143.4		
pw	7.117	rfl	-288.8		
DECOUPLER			PLOT		
dn	H1	lp			
dof	0	wc	250		
dm	ynn	sc	0		
dmm	35	vs	124		
dppw	w	th			
dmp	6700	ai	no	ph	7

Table 1, entries 2a and 2b
31-P/1-H COUPLED

723

exp1 s2pu1

SAMPLE		SPECIAL	
date	Oct 27 2005	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	alfa	20.000
pp	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	594.5
tpwr	55	rfp	0
pw	8.600	rp	-111.6
DECOUPLER		lp	-79.5
dn	C13	PLOT	
dof	0	wc	250
dm	nn	sc	0
dmm	C	vs	373
dpwr	45	th	2
dmf	13100	al	cdc
		ph

Table 1, entries 2a and 2b

Table 1, entries 2a and 2b

INDEX	FREQUENCY	PPM	HEIGHT
1	3271.625	10.904	14.7
2	2357.270	7.856	31.0
3	2184.308	7.280	4.8
4	1804.084	6.013	31.0
5	1700.600	5.668	14.8
6	1693.858	5.645	18.7
7	1685.942	5.619	23.4
8	1679.493	5.597	21.3
9	1614.988	5.382	20.2
10	1607.963	5.359	23.1
11	1600.341	5.334	19.0
12	1591.546	5.304	20.3
13	804.128	2.680	4.0
14	796.799	2.656	3.3
15	782.141	2.607	39.2
16	775.692	2.585	40.2
17	763.379	2.544	38.8
18	756.343	2.521	37.6
19	613.869	2.046	49.6
20	608.299	2.027	65.0
21	524.457	1.748	2.9
22	503.936	1.680	2.3
23	480.483	1.601	2.5
24	428.888	1.429	33.1
25	421.559	1.405	64.5
26	414.523	1.382	66.0
27	407.194	1.357	36.3
28	400.158	1.334	10.2
29	376.999	1.256	8.7
30	274.394	0.914	96.1
31	267.359	0.891	162.0
32	260.030	0.867	74.9

723

exp1 s2pu1

SAMPLE		SPECIAL	
date	Oct 27 2005	temp	not used
solvent	CDCl3	gain	not used
file		set	20
ACQUISITION		exp	0.008
sw	18867.8	pw90	18.500
at	1.815	alra	20.000
np	68492	il	FLAGS
fb	10400	in	n
bs	64	in	n
ss	4	dp	y
di	1.000	hs	nn
nt	1000	PROCESSING	
ct	384	lb	1.00
TRANSMITTER		fn	not used
tn	C13	fn	not used
sfrq	75.456	sp	DISPLAY
tof	737.2	wp	-150.9
tpwr	58	rfl	15844.1
pw	9.250	rfl	1149.9
DECOUPLER		rp	0
dn	H1	rp	46.4
dof	0	lp	-238.6
dm	yyy	wc	250
dmm	35	sc	0
dpwr	6700	vs	140
dmf		al	5
		no	ph

Table 1, entries 2a and 2b

737

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jan 19 2006	temp	not used
solvent	CDC13	gain	not used
file		spin	20
ACQUISITION		hst	0.008
sw	26738.0	pw90	18.300
at	1.598	alpha	20.000
np	85476	FLAGS	
fb	14800	fl	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
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	rf1	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	145.1
dn	H1	lp	-288.6
dof	0	PLOT	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	49
dmf	6700	th	11
		ai	no
		ph

Table 1, entry 2c
31-P/1-H DECOUPLED

INDEX	FREQUENCY	PPM	HEIGHT
1	4876.274	40.146	122.5
2	4653.512	38.312	12.7
3	4328.713	35.622	119.7

Table 1, entry 2c
31-P/1-H COUPLED

Table 1, entry 2c

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3047.947	10.158	14.1	40	514.196	1.714	108.9
2	2357.270	7.856	20.1	41	508.333	1.694	58.3
3	2214.503	7.380	3.2	42	499.538	1.665	8.9
4	2200.725	7.335	3.1	43	422.145	1.407	3.6
5	2192.516	7.307	3.1				
6	2184.601	7.281	26.5				
7	2174.634	7.248	3.4				
8	1806.136	6.019	20.1				
9	1708.515	5.694	8.1				
10	1702.066	5.673	13.0				
11	1693.271	5.643	15.6				
12	1687.115	5.623	19.6				
13	1680.959	5.602	14.3				
14	1674.802	5.582	5.8				
15	1629.363	5.430	5.2				
16	1622.327	5.407	13.5				
17	1615.585	5.384	16.6				
18	1607.963	5.359	14.2				
19	1600.341	5.334	10.6				
20	1594.184	5.313	4.7				
21	1590.960	5.302	57.1				
22	1247.087	4.156	4.1				
23	1240.051	4.133	12.8				
24	1233.016	4.109	12.6				
25	1225.687	4.085	5.6				
26	1125.427	3.751	5.8				
27	1118.685	3.728	4.5				
28	776.278	2.587	29.0				
29	769.242	2.564	28.8				
30	757.516	2.525	28.5				
31	750.187	2.500	28.5				
32	613.576	2.045	81.0				
33	560.515	1.868	2.9				
34	557.290	1.857	6.6				
35	553.772	1.846	3.3				
36	550.548	1.835	3.3				
37	537.942	1.793	6.5				
38	536.183	1.787	7.8				
39	520.353	1.734	58.9

737

exp1 s2pul

SAMPLE		SPECIAL	
date	Oct 31 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	1000	PROCESSING	
ct	1000	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	1150.7
pw	9.250	rfp	0
dn	H1	rp	81.2
dof	0	lp	-227.3
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	284
dmf	6700	th	8
		ai	no
		ph

Table 1, entry 2c

Table 1, entries 3a and 8a
31-P/1-H DECOUPLED

```

721
exp1 s2pul

SAMPLE
date Oct 27 2005 temp not used
solvent CDCl3 gain not used
file exp 20
ACQUISITION
sw 26738.0 hst 0.008
at 1.598 pw90 18.300
np 85476 alfa 20.000
fb 14800 il n
bs 64 in n
ss 4 dp y
d1 1.000 hs nn
nt 8
ct 8 lb 2.00
TRANSMITTER p31 fn not used
tn 121.474 sp DISPLAY
sfrq 10608.2 wp 10010.0
tof 7.117 rfp 2437.3
pw DECOUPLER H1 lp 149.7
dn dof 0 -288.8
dm yy WC 250
dmm w SC 0
dpwr 35 VS 111
dmf 6700 th 8
ai no ph

```

721

exp1 s2pul

SAMPLE		SPECIAL	
date	Oct 27 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	alfa	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
di	1.000	hs	nn
nt	8	PROCESSING	
Ct	8	lb	2.00
TRANSMITTER		fn	not used
tn	p31	DISPLAY	
sfrq	121.474	sp	0
tof	10605.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER		rp	47.8
dn	H1	lp	32.6
dof	0	PLOT	
dm	ynn	wc	250
dmm	w	sc	0
dpwr	35	vs	172
dmf	6700	th	11
	ai	no	ph

Table 1, entries 3a and 8a
31-P/1-H COUPLED

Table 1, entries 3a and 8a

721
exp1 s2pul

SAMPLE		SPECIAL	
date	temp	gain	not used
Oct 27 2005	20	0.008	not used
solvent	CDCl3	17.200	not used
file	exp	20.000	not used
ACQUISITION			
sw	4803.1	hst	0.008
at	1.994	pw90	17.200
np	19158	alpha	20.000
fb	not used	il	FLAGS
bs	16	in	n
ss	4	dp	n
dl	1.000	hs	y
nt	8	fn	nn
ct	8	fn	not used
PROCESSING			
not used			
DISPLAY			
tn	H1	sp	-60.0
sfrq	300.053	wp	3060.5
tof	300.9	rf1	595.1
tpwr	55	rfp	0
pw	8.600	rp	-114.0
dn	DECOUPLER	lp	-64.0
dof	C13	plot	
dm	0	wc	250
dmm	nnn	sc	0
dpwr	c	vs	211
dmf	45	th	3
	13100	ai	cdc
		ph

24.53.4 10.00

Table 1, entries 3a and 8a

INDEX	FREQUENCY	PPM	HEIGHT
1	3272.211	10.906	9.5
2	2356.684	7.854	29.5
3	2214.796	7.381	3.0
4	2184.015	7.279	13.7
5	1806.723	6.021	30.0
6	1550.797	5.168	10.5
7	1549.331	5.164	9.9
8	1544.055	5.146	24.2
9	1542.882	5.142	25.6
10	1536.139	5.120	25.2
11	1528.690	5.098	8.4
12	1528.224	5.093	8.9
13	904.387	3.014	5.6
14	802.955	2.676	3.8
15	785.659	2.618	35.7
16	778.037	2.593	36.5
17	766.604	2.555	36.1
18	758.689	2.529	34.4
19	538.821	1.796	14.6
20	532.958	1.776	138.2
21	527.095	1.757	133.4
22	517.421	1.724	10.2
23	499.538	1.665	162.0
24	496.021	1.653	159.0
25	385.794	1.286	3.8
26	376.413	1.254	10.8

721

exp1 s2pu1

SAMPLE		SPECIAL	
date	Oct 27 2005	temp	not used
solvent	CDCl3	gain	not used
file	ACQUISITION exp	spn	20
sw	18867.9	hst	0.008
at	1.815	pw90	18.500
np	68432	alpha	20.000
fb	10400	il	FLAGS
bs	64	in	n
ss	4	dp	n
d1	1.000	hs	y
nt	1000	nn	nn
ct	771	PROCESSING	1.00
TRANSMITTER		fn	not used
tn	C13	DISPLAY	
sfrq	75.456	sp	-150.9
tof	737.2	wp	15844.1
tpwr	58	rf1	1151.3
pw	9.250	rfp	60.4
dn	H1	rp	-306.2
dof	0	lp	
din	yyy	WC	250
dmm	w	SC	0
dpwr	35	VS	252
dmf	6700	th	7
		ai	no
		ph

Table 1, entries 3a and 8a

650a

exp1 s2pu1

SAMPLE

date	Sep 22 2005	temp	not used
solvent	CDC13	gain	not used
file	ACQUISITION	spn	0.008
sw	26738.0	hst	18.300
at	1.598	pw50	20.000
np	85476	alfa	FLAGS
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
di	1.000	hs	nn
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	rfl	149.3
DECOUPLER	H1	lp	-0
dn	0	PLOT	250
dof	yyy	wc	0
dm	w	sc	0
dmm	35	vs	19
dpwr	6700	th	3
dmf	al	no	ph

Table 1, entry 3b
31-P/1-H DECOUPLED

P1 s2pu1

SAMPLE		SPECIAL	
date	Sep 22 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	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
dl	1.000	hs	nn
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	-100.5
dn	H1	lp	-398.4
dof	0	PLOT	
dm	ynn	wc	250
dmm	w	sc	0
dpwr	35	ss	72
dmf	6700	th	11
		ai	no
			ph

Table 1, entry 3b
31-P/1-H COUPLED

H1 s2pu1

SAMPLE SPECIAL
 date Sep 22 2005 temp not used
 solvent CDC13 gain not used
 file exp sp in 20
 ACQUISITION hst 0.008
 sw 4803.1 pw90 17.200
 at 1.994 alfa 20.000
 np not used il n
 fb 16 in n
 bs 4 dp y
 ss 1.000 hs nm
 di 8 fn not used
 ct PROCESSING
 TRANSMITTER H1 sp DISPLAY
 tn 300.053 wp -60.0
 tof 300.9 rfl 3060.5
 tpwr 55 rfp 597.5
 pw 8.600 rp -103.9
 DECOUPLER lp -91.6
 dn C13 PLOT
 dof 0 wc 250
 dm nnn sc 0
 dmm C vs 187
 dpwr 45 th 3
 dmf 13100 ai cdc ph

Table 1, entry 3b

	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
3	3419.083	11.395	10.9	40	378.465	1.261	5.9
4	2360.202	7.866	13.3	41	272.635	0.909	4.6
5	2357.857	7.858	27.7	42	266.186	0.887	4.2
6	2355.805	7.851	14.7				
7	2181.963	7.272	23.4				
8	1808.188	6.026	13.8				
9	1805.843	6.018	28.1				
10	1803.791	6.012	14.1				
11	1591.253	5.303	3.9				
12	1552.556	5.174	4.9				
13	1545.520	5.151	14.7				
14	1537.605	5.124	15.5				
15	1529.690	5.098	10.5				
16	1528.224	5.093	10.9				
17	1526.758	5.088	10.6				
18	1524.413	5.081	13.1				
19	1522.947	5.076	17.3				
20	1521.482	5.071	19.2				
21	1520.309	5.067	15.8				
22	1518.843	5.062	10.9				
23	1516.205	5.053	8.8				
24	1515.032	5.049	8.9				
25	893.247	2.977	6.2				
26	869.502	2.898	4.9				
27	794.454	2.648	19.5				
28	786.539	2.621	19.3				
29	774.812	2.582	19.5				
30	766.897	2.556	18.8				
31	637.615	2.125	5.8				
32	623.544	2.078	159.0				
33	615.042	2.050	25.7				
34	611.524	2.038	5.6				
35	609.765	2.032	5.6				
36	532.665	1.775	3.0				
37	527.975	1.760	3.2				
38	504.522	1.681	162.0				
39	499.245	1.664	86.8				
	495.141	1.650	83.3				
	479.897	1.599	160.0				

Table 1, entry 3b

670b

exp1 s2pul

SAMPLE		SPECIAL	
date	Oct 5 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spn	20
ACQUISITION		hst	0.008
sw	18867.9	pw30	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	1000	PROCESSING	
ct	192	lb	1.00
TRANSMITTER C13		fn	not used
tn	75.456	sp	-150.9
sfrq	737.2	wp	15844.1
tof	58	rfi	1134.7
tpwr	9.250	rff	0
DECOUPLER H1		rp	73.4
dn	0	lp	-248.2
dof	0	PLOT	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	99
dmf	6700	th	g
		ai	no
		ph

Table 1, entry 3b

704a

exp1 s2pu

SAMPLE		SPECIAL	
date	Oct 21 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	alpha	20.000
np	85476	il	FLAGS
fb	14800	in	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	PROCESSING	
ct	16	lb	2.00
TRANSMITTER		fn	not used
tn	P31	fn	not used
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfl	0
DECOUPLER		rp	127.6
dn	H1	lp	-288.8
dof	0	PLOT	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	56
dmf	6700	th	6
		ai	no
		ph	

Table 1, entry 3c
31-P/1-H DECOUPLED

704a

exp1 s2pul

SAMPLE		SPECIAL	
date	Oct 21 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	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	16	PROCESSING	
ct	16	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	rff	132.8
DECOUPLER		lp	-288.8
dn	H1	PLOT	
dof	0	wc	250
dm	ynn	w	0
dmm	35	vs	121
dpwr	6700	th	12
dmf		ai	no
			ph

Table 1, entry 3c
31-P/1-H COUPLED

803

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jan 23 2006	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
di	1.000	hs	nn
nt	8	fn	not used
ct		PROCESSING	
TRANSMITTER		DISPLAY	
tn	H1	sp	-50.0
sfrq	300.053	wp	3060.5
tof	300.9	rfl	600.1
tpwr	55	rfp	0
pw	8.600	rp	-98.2
DECOUPLER		lp	-113.7
dn	C13	PL01	
dof	0	wc	250
dm	nnn	sc	0
dmm	C	vs	212
dpwr	45	th	3
dmf	13100	al	cdc
		ph	

Table 1, entry 3c

Table 1, entry 3c

```

a
exp1 s2pul
SAMPLE
date Feb 18 2005
solvent CDCl3
file
ACQUISITION
sw 18867.9
at 1.815
np 68492
fb 10400
bs 64
ss 4
di 1.000
nt 512
ct 384
TRANSMITTER C13
tn 75.456
sfrq 737.2
tof 58
tpwr 9.250
pw
DECOUPLER H1
dn 0
dof 0
dm YVY
dmm W
dpr 35
dmf 6700
SPECIAL
temp not used
gain not used
sp 20
hst 0.008
pw90 18.500
alfa 20.000
FLAGS
i1 n
in n
dp y
hs nn
lb 1.00
fn not used
sp -1134.4
wp 18867.6
rfl 1151.6
rfp 0
rp 107.9
lp -258.8
PLOT
wc 250
sc 0
vs 177
th 8
ai no
ph

```

INDEX	FREQUENCY	PPM	HEIGHT
1	4599.249	37.866	153.3

Table 1, entry 4
31-P/1-H DECOUPLED

INDEX	FREQUENCY	PPM	HEIGHT
1	4878.722	40.166	89.8
2	4320.185	35.568	90.4

Table 1, entry 4
31-P/1-H COUPLED

Table 1, entry 4

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3345.794	11.151	9.3	40	-6.156	-0.021	12.1
2	2412.970	8.042	29.7	41	-7.622	-0.025	6.2
3	2203.950	7.345	22.7	42	-11.140	-0.037	4.8
4	2203.070	7.342	35.0	43	-12.019	-0.040	5.1
5	2201.311	7.336	16.2	44	-20.228	-0.067	15.5
6	2196.621	7.321	60.6	45	-59.511	-0.198	8.6
7	2195.741	7.318	61.0				
8	2190.464	7.300	30.1				
9	2188.412	7.293	97.6				
10	2182.549	7.274	9.6				
11	2176.979	7.255	105.3				
12	2173.754	7.245	136.9				
13	2171.409	7.237	70.4				
14	2166.132	7.219	73.9				
15	2163.494	7.210	52.1				
16	2161.149	7.203	15.0				
17	2156.165	7.186	17.8				
18	2149.129	7.163	6.0				
19	1918.708	6.395	32.5				
20	1912.845	6.375	33.1				
21	1855.387	6.184	29.8				
22	828.753	2.762	63.6				
23	809.405	2.698	65.9				
24	651.393	2.171	7.4				
25	597.746	1.992	151.9				
26	596.573	1.988	162.0				
27	594.228	1.980	158.2				
28	592.762	1.976	142.5				
29	58.924	0.196	7.9				
30	20.814	0.069	6.5				
31	9.674	0.032	6.3				
32	8.208	0.027	5.9				
33	7.622	0.025	5.8				
34	5.277	0.018	10.1				
35	3.518	0.012	71.5				
36	0.000	0.000	2084.2				
37	-2.052	-0.007	52.8				
38	-3.225	-0.011	86.8				
39	-5.570	-0.019	11.7

732

exp1 s2pu1

SAMPLE		SPECIAL	
date	Nov 4 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	alpha	20.000
np	68492	FLAGS	
fb	10400	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	1000	PROCESSING	
ct	717	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	1153.3
pw	9.250	rfp	37.7
DECOUPLER		rp	-252.9
dn	H1	lp	
dof	0	PLOT	
dm	yy	wc	250
dmm	w	sc	0
dpwr	35	vs	257
dmf	6700	th	7
		ai	no ph

Table 1, entry 4

736a

exp1 s2pul

SAMPLE		SPECIAL	
date	Oct 31 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
dl	1.000	hs	nn
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	rf1	2437.3
pw	7.117	rfp	140.7
DECOUPLER		rp	-0
dn	H1	lp	
dof	0	PLOT	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	61
dmf	6700	th	5
		ai	no
		ph

Table 1, entry 5
31-P/1-H DECOUPLED

736a

exp1 s2pul

date	Oct 31 2005	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
		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
d1	1.000	hs	nn
nt	16		PROCESSING
ct	16	lb	2.00
		fn	not used
tn	P31		DISPLAY
sfrq	121.474	sp	0
tor	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rffp	0
		rp	102.0
dn	H1	lp	146.1
dof	0		PLOT
dm	ynn	wc	250
dmin	w	sc	0
dpwr	35	vs	249
dmf	6700	th	12
		ai	no
		ph

Table 1, entry 5
31-P/1-H COUPLED

736a

exp1 s2pu1

SAMPLE		SPECIAL	
date	Oct 31 2005	temp	not used
solvent	CDCl3	gain	not used
file	ACQUISITION	spn	20
sw	4803.1	hst	0.008
at	1994	pw90	17.200
np	1958	alfa	20.000
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	3080.5
tof	300.9	rfl	593.3
tpwr	55	rfp	0
pw	8.600	rp	-111.6
DECOUPLER		lp	-76.5
dn	C13	plot	
dof	0	wc	250
dm	nnn	sc	0
dmm	C	vs	135
dpwr	45	tn	2
dmf	13100	al	cdc
		ph

Table 1, entry 5

Table 1, entry 5

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3483.577	11.610	8.7	40	378.465	1.261	31.0
2	2364.018	7.879	15.6	41	377.879	1.259	27.7
3	2184.308	7.280	13.1	42	371.429	1.238	13.9
4	1811.413	6.037	15.6	43	370.550	1.235	13.2
5	1837.865	5.459	15.8	44	365.859	1.219	44.1
6	1591.546	5.304	25.4	45	357.064	1.190	37.3
7	1590.666	5.301	25.3	46	283.188	0.944	2.3
8	1247.967	4.159	2.3	47	276.153	0.920	2.6
9	1240.931	4.136	6.7	48	261.789	0.872	219.7
10	1240.051	4.133	6.5	49	242.440	0.808	3.7
11	1233.602	4.111	7.0	50	226.610	0.755	2.2
12	1233.016	4.109	6.6				
13	1226.566	4.088	2.6				
14	798.558	2.661	2.1				
15	782.141	2.507	26.7				
16	762.793	2.502	26.9				
17	746.083	2.487	2.9				
18	727.907	2.426	8.6				
19	722.337	2.407	18.9				
20	719.406	2.398	12.5				
21	716.767	2.389	14.6				
22	713.836	2.379	20.3				
23	708.266	2.360	13.5				
24	681.589	2.272	22.9				
25	679.830	2.266	22.8				
26	654.325	2.181	14.1				
27	648.755	2.162	27.8				
28	643.478	2.145	16.5				
29	625.889	2.086	18.2				
30	617.680	2.059	9.3				
31	616.215	2.054	9.0				
32	614.163	2.047	32.2				
33	613.576	2.045	30.4				
34	564.326	1.881	2.2				
35	557.583	1.858	2.7				
36	537.356	1.791	2.3				
37	404.556	1.348	2.3				
38	397.813	1.326	2.6				
39	383.155	1.277	216.3

Table 1, entry 5

INDEX	FREQUENCY	PPM	HEIGHT
1	10335.670	136.991	18.7
2	10325.306	136.853	17.0
3	9277.344	122.964	34.4
4	9263.237	122.777	33.0
5	5846.132	77.486	44.9
6	5814.174	77.062	45.2
7	5782.217	76.639	44.9
8	4556.908	60.398	6.6
9	3526.220	46.737	59.2
10	3523.341	46.699	60.3
11	3034.484	40.220	128.6
12	2949.553	39.094	34.7
13	2879.881	38.170	28.4
14	2877.289	38.136	30.8
15	2858.864	37.892	33.6
16	2389.008	31.664	56.5
17	2385.841	31.622	69.2
18	2384.114	31.599	70.7
19	2380.371	31.550	63.7
20	1973.854	26.162	109.7
21	1601.309	21.224	109.2
22	1588.066	21.049	8.3

INDEX	FREQUENCY	PPM	HEIGHT
1	4236.139	34.876	126.0

Table 1, entry 6b
31-P/1-H DECOUPLED

INDEX	FREQUENCY	PPM	HEIGHT
1	4517.244	37.190	125.5
2	3856.667	32.575	124.3

Table 1, entry 6b
31-P/1-H COUPLED

Table 1, entry 6b

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3415.272	11.382	7.1	40	788.591	2.628	3.1
2	2385.413	7.950	13.1	41	657.843	2.192	3.0
3	2198.380	7.327	32.2	42	656.963	2.190	3.6
4	2192.810	7.308	39.5	43	656.377	2.188	4.0
5	2189.878	7.298	44.5	44	655.498	2.185	4.5
6	2184.601	7.281	37.5	45	654.911	2.183	5.3
7	2179.324	7.263	16.3	46	654.032	2.180	6.3
8	2136.230	7.120	4.1	47	653.446	2.178	8.5
9	2127.438	7.090	5.1	48	652.859	2.176	10.5
10	2122.159	7.073	4.9	49	650.221	2.167	162.0
11	2115.416	7.050	4.5	50	648.169	2.160	5.2
12	2113.657	7.044	4.0	51	647.582	2.158	3.3
13	2106.622	7.021	8.2	52	627.648	2.092	3.8
14	2101.638	7.004	43.9	53	614.163	2.047	38.5
15	2093.136	6.976	77.0	54	529.147	1.764	4.8
16	2086.394	6.953	21.7	55	429.474	1.431	4.3
17	2084.342	6.947	37.0	56	385.794	1.286	4.0
18	2078.186	6.926	5.8	57	384.035	1.280	3.5
19	1955.646	6.518	11.6	58	376.706	1.255	13.9
20	1950.076	6.499	12.0				
21	1939.816	6.465	14.7				
22	1933.953	6.445	12.7				
23	1826.364	6.087	12.7				
24	1819.328	6.063	6.6				
25	1811.413	6.037	12.1				
26	1804.084	6.013	13.8				
27	1795.876	5.985	12.2				
28	1788.254	5.960	9.5				
29	1780.925	5.935	3.8				
30	1587.735	5.292	6.3				
31	1396.304	4.654	4.2				
32	886.505	2.955	8.2				
33	862.759	2.875	8.7				
34	838.427	2.794	20.6				
35	831.098	2.770	20.4				
36	819.079	2.730	20.6				
37	811.750	2.705	19.2				
38	803.248	2.677	3.6				
39	795.920	2.653	4.9

```

expl s2pul
SAMPLE
date Jan 25 2006 temp not used
solvent CDCl3 gain not used
file exp 20
sw ACQUISITION hst 0.008
at 4803.1 pw90 17.200
np 1.994 alfa 20.000
fb 19158
bs not used ll n
ss 16 in n
di 4 dp y
nt 1.000 hs nn
ct 8 fn not used
TRANSMITTER H1 sp
tn 300.053 wp -60.0
sfrq 300.9 rfl 3080.5
tof 55 rfp 601.3
tpwr 8.600 rp -113.7
pw DECOUPLER C13 lp -78.3
dn dof 0 wc 250
dm nnn sc 0
dmm c vs 428
dpwr 45 th 2
dmf 13100 ai cdc ph

```

SPECIAL

Table 1, entry 6b

805a

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jan 31 2006	temp	not used
solvent	CDCl3	gain	not used
file		spin	20
ACQUISITION		hst	0.008
sw	10067.9	pw90	18.500
at	1.815	alfa	20.000
fb	68492	FLAGS	
np	10400	ll	n
bs	64	ln	n
ss	4	dp	y
dl	1.000	hs	nm
nt	1000	PROCESSING	
ct	804	lb	1.00
TRANSMITTER C13		fn	not used
tn		DISPLAY	
sfrq	75.456	sp	-150.9
tof	737.2	wp	15844.1
tpwr	58	rfl	1134.7
pw	9.250	rff	0
DECOUPLER H1		rp	72.5
dn		lp	-238.4
dof	0	PLOT	
dm	YVY	wc	250
dmm	W	sc	0
dpwr	35	vs	210
dmf	6700	th	11
		al	no
		ph

Table 1, entry 6b

Interpolated Peak Listing

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	23266	65108K	98.01	1309.33	35.931

S55

Table 1, entry 7a
31-P/1-H DECOUPLED

PEAK	POINT	HEIGHT	REL. HT	HZ	PPM
1	22781	9890K	52.03	1605.73	44.064
2	22785	9785K	51.48	1603.32	43.998
3	22813	16967K	89.26	1585.79	43.517
4	22841	9811K	51.61	1569.02	43.057
5	22868	2383K	12.53	1552.33	42.599
6	23715	16985K	89.36	1035.21	28.408
7	23744	9407K	49.49	1017.79	27.930

Table 1, entry 7a
31-P/1-H COUPLED

807a

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jan 26 2006	temp	not used
solvent	CDCl3	gain	not used
file		spn	20
ACQUISITION		hst	0.008
sw	4803.1	prg0	17.200
at	1.994	alra	20.000
np	19158	il	FLAGS
fb	not used	in	n
bs	16	in	n
ss	4	dp	y
di	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	600.1
tpwr	55	rfl	0
pw	8.600	rp	-102.5
DECOUPLER		lp	-101.0
dn	C13	PL0T	
dof	0	wc	250
dm	nmn	sc	0
dmm	C	vs	207
dpwr	45	th	3
dmf	13100	ai	cdc ph

Table 1, entry 7a

Table 1, entry 7a

INDEX	FREQUENCY	PPM	HEIGHT
1	3287.162	10.955	4.9
2	2354.339	7.846	12.8
3	2180.497	7.267	16.0
4	1801.739	6.005	12.9
5	1531.156	5.103	3.9
6	1523.534	5.078	11.2
7	1516.791	5.055	11.6
8	1509.169	5.030	4.6
9	792.695	2.642	15.3
10	785.073	2.616	15.2
11	773.347	2.577	15.3
12	765.724	2.552	14.8
13	638.788	2.129	46.5
14	622.078	2.073	10.5
15	507.161	1.690	3.1
16	464.360	1.548	103.8
17	423.904	1.413	3.6
18	411.885	1.373	3.1
19	387.553	1.282	6.2
20	376.413	1.254	18.7
21	264.134	0.880	3.4

807

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jan 26 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
ACQUISITION		ns	0.008
sw	18867.9	pw	18.500
at	1.515	af	20.000
np	68492	il	nn
fb	10400	in	n
bs	64	dp	y
ss	4	hs	nn
d1	1.000	ps	nn
nt	1000	pr	nn
ct	847	lb	1.00
TRANSMITTER		fn	not used
tn	C13	fn	not used
sfrq	75.456	sp	-150.9
tof	737.2	wp	15844.1
tpwr	58	rfl	1134.7
pw	9.250	rfl	0
DECOUPLER		rp	78.8
dn	H1	lp	-207.7
dof	0	pl	0
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	278
dmf	6700	th	13
	ai	no	ph

Table 1, entry 7a

Table 1, entry 7b
31-P/1-H DECOUPLED

INDEX	FREQUENCY	PPM	HEIGHT
1	4727.358	38.920	12.7
2	4294.890	35.360	130.7

Table 1, entry 7b
31-P/1-H COUPLED

INDEX	FREQUENCY	PPM	HEIGHT
1	4569.058	37.617	129.7
2	4018.681	33.086	123.0

Table 1, entry 7b

INDEX	FREQUENCY	PPM	HEIGHT
1	1575.715	5.251	20.8
2	1569.559	5.231	20.0
3	1254.416	4.181	28.6
4	1247.380	4.157	75.1
5	1240.345	4.134	77.8
6	1233.309	4.110	32.1
7	1041.585	3.471	108.4
8	802.076	2.673	25.9
9	784.161	2.647	27.0
10	783.021	2.610	27.8
11	775.399	2.584	25.1
12	670.155	2.233	95.7
13	619.146	2.063	66.7
14	387.846	1.293	88.3
15	380.517	1.268	162.0
16	376.706	1.255	69.4
17	373.774	1.246	80.2

Table 1, entry 7b

718

exp1 s2pul

SAMPLE		SPECIAL	
date	Oct 27 2005	temp	not used
solvent	CDCl3	gain	not used
file		spin	20
ACQUISITION			
sw	26738.0	hst	0.008
at	1.598	pw90	18.300
np	85476	alfa	20.000
fb	14800	il	FLAGS
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	8	PROCESSING	2.00
ct	8	lb	not used
TRANSMITTER			
tn	p31	fn	DISPLAY
sfrq	121.474	sp	0
tof	10608.2	wp	10010.0
tpwr	55	rfl	2437.3
pw	7.117	rfp	0
DECOUPLER			
dn	H1	lp	-137.8
dof	0	PLOT	-288.8
dm	VVY	wc	250
dmm	W	sc	0
dpwr	35	vs	45
dmf	6700	th	10
		ai	no
		ph

Table 1, entry 8b
31-P/1-H DECOUPLED

INDEX	FREQUENCY	PPM	HEIGHT
1	4784.884	39.394	132.2
2	4254.499	35.027	129.1
3	3655.979	30.100	-12.1

Table 1, entry 8b
31-P/1-H COUPLED

718

exp1 s2pul

SAMPLE		SPECIAL	
date	Oct 27 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	fn	not used
ct	TRANSMITTER	fn	not used
tn	H1	sp	DISPLAY
sfrq	300.053	wp	-60.0
tof	300.9	rfl	3060.5
tpwr	55	rff	599.2
pw	8.600	rp	0
DECOUPLER		lp	-109.9
dn	C13	pl	-80.8
dof	0	wc	PLOT
dm	nnn	sc	250
dmm	C	vs	0
dpwr	45	th	245
dmf	13100	al	2
		cdc	
		ph

Table 1, entry 8b

INDEX	FREQUENCY	PPM	HEIGHT
1	3031.530	10.103	5.0
2	2355.805	7.851	20.1
3	2179.911	7.265	16.6
4	1804.671	6.015	20.3
5	1591.839	5.305	2.7
6	1590.666	5.301	48.6
7	1553.729	5.178	5.2
8	1545.814	5.152	14.7
9	1538.778	5.128	15.3
10	1527.638	5.091	13.7
11	1526.172	5.086	14.8
12	1524.706	5.081	14.4
13	1522.947	5.076	13.9
14	1521.482	5.071	13.9
15	1520.309	5.067	12.1
16	1515.032	5.049	5.2
17	788.884	2.629	16.3
18	781.262	2.604	16.0
19	769.535	2.565	16.8
20	761.620	2.538	15.8
21	637.029	2.123	3.7
22	620.612	2.068	68.2
23	616.508	2.055	72.5
24	606.247	2.020	3.6
25	532.079	1.773	40.3
26	527.095	1.757	38.8
27	526.216	1.754	39.0
28	503.936	1.680	162.0
29	497.780	1.659	44.2
30	493.968	1.646	42.4
31	479.897	1.599	131.9
32	376.120	1.254	6.2

Table 1, entry 8b

exp1 s2pu1

date	Oct 17 2005	SAMPLE	temp	not used
solvent	CDCl3	gain	not used	
file	exp	spin	20	
ACQUISITION				
sw	18867.9	hst	0.008	
at	1.815	pw90	18.500	
np	68492	alfa	20.000	
fb	10400	il	FLAGS	
bs	64	in	n	
ss	4	dp	n	
dl	1.000	hs	y	
nt	512	PROCESSING	nm	
ct	512	lb	1.00	
TRANSMITTER C13				
tn	75.456	sp	not used	
sfrq	75.456	wp	-150.9	
tof	737.2	rfl	15844.1	
tpwr	58	rfl	1134.7	
pw	9.250	rfl	0	
DECOUPLER				
dn	H1	lp	49.6	
dof	0	lp	-225.8	
dm	VVY	WC	250	
dmm	W	SC	0	
dpwr	35	VS	176	
dmf	6700	th	31	
		ai	no	ph

Table 1, entry 8b

Table 1, entry 8c
31-P/1-H DECOUPLED

824
exp1 s2pu1

SAMPLE		SPECIAL	
date	Feb 13 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	alpha	20.000
np	85476	FLAGS	
fb	14800	il	n
bs	64	in	n
ss	4	dp	y
d1	1.000	hs	nn
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	rfl	0
DECOUPLER		rp	133.3
dn	H1	lp	-288.8
dof	0	PLOT	
dm	YYY	wc	250
dmm	w	sc	0
dpwr	35	vs	38
dmf	6700	th	5
	ai	no	ph

INDEX	FREQUENCY	PPM	HEIGHT
1	4722.054	38.877	167.5
2	4707.366	38.756	166.6
3	4172.901	34.355	165.4
4	4153.317	34.194	163.7

FREQUENCY	PPM	HEIGHT
38.877	167.5	
38.756	166.6	
34.355	165.4	
34.194	163.7

Table 1, entry 8c
31-P/1-H COUPLED

824

exp1 s2pu1

SAMPLE		SPECIAL	
date	Feb 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
np	19158	il	FLAGS
fb	not used	in	n
bs	16	in	n
ss	4	dp	y
d1	1.000	hs	PROCESSING
nt	8	fn	not used
ct	TRANSMITTER	fn	DISPLAY
tn	H1	sp	-60.0
strq	300.053	wp	3060.5
tor	300.9	rfl	598.3
tpwr	55	rfp	0
pw	8.600	rp	-106.4
DECOUPLER	lp	lp	-86.8
dn	C13	PL0T	
dof	0	wc	250
dm	nnh	sc	0
dmm	c	vs	256
dpwr	45	th	2
dmf	13100	ai	cdc
		ph

Table 1, entry 8c

824

expl s2pul

SAMPLE		SPECIAL	
date	Feb 13 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	alpha	20.000
np	68492	FLAGS	
fb	10400	il	n
bs	64	in	n
ss	4	dp	y
di	1.000	hs	nn
nt	1000	PROCESSING	
ct	582	lb	1.00
TRANSMITTER C13		fn	not used
tn	75.456	sp	-150.9
sfrq	737.2	wa	15844.1
tof	58	rf1	1134.7
tpwr	9.250	rfl	0
DECOUPLER H1		rp	67.7
dn	0	lp	-242.3
dof	0	PLOT	
dm	yy	wc	250
dmm	w	sc	0
dpwr	35	vs	229
dmf	6700	th	26
		ai	no
		ph

Table 1, entry 8c

Table 1, entry 8c

748a

exp1 s2pul

SAMPLE		SPECIAL	
date	Nov 11 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	alfa	20.000
np	85476	il	n
fb	14800	in	n
bs	64	dp	y
ss	4	hs	nn
d1	1.000	PROCESSING	
nt	8	lb	2.00
ct	8	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	-144.5
dn	DECOUPLER	lp	-161.8
dof	H1	PL0T	
dm	0	wc	250
dmm	YVY	sc	0
dpwr	W	vs	15
dmf	35	th	3
	6700	ai	no
			ph

Table 1, entry 9
31-P/1-H DECOUPLED

INDEX	FREQUENCY	PPM	HEIGHT
1	4866.074	40.062	78.4
2	4321.817	35.581	77.7

Table 1, entry 9
31-P/1-H COUPLED

Table 1, entry 9

INDI X	FREQUENCY	PPM	HEIGHT
1	2373.980	7.912	16.1
2	1829.882	6.099	15.9
3	1732.261	5.773	6.0
4	1724.346	5.747	7.5
5	1716.137	5.719	6.6
6	1588.028	5.293	21.9
7	1583.044	5.276	19.1
8	1570.439	5.234	8.4
9	1564.869	5.215	7.9
10	1248.260	4.160	9.4
11	1240.345	4.134	15.2
12	1232.429	4.107	12.5
13	1223.635	4.078	6.7
14	1213.961	4.046	14.7
15	1206.925	4.022	19.8
16	890.023	2.966	23.1
17	866.863	2.889	21.4
18	814.388	2.714	18.0
19	807.059	2.690	17.6
20	795.333	2.651	17.9
21	788.004	2.626	17.1
22	610.058	2.033	26.6
23	571.655	1.905	6.6
24	514.489	1.715	26.0
25	507.747	1.692	39.1
26	500.711	1.669	34.9
27	494.262	1.647	18.9
28	436.217	1.454	19.8
29	428.888	1.429	35.1
30	421.852	1.406	38.1
31	414.523	1.382	23.9
32	407.487	1.358	10.8
33	378.172	1.260	36.8
34	291.397	0.971	51.5
35	284.362	0.948	90.3
36	277.033	0.923	47.9
37	264.427	0.881	17.7
38	257.098	0.857	13.8

Table 1, entry 9

INDEX	FREQUENCY	PPM	HEIGHT
1	5187.978	42.712	97.8
2	5094.548	41.943	126.0

INDEX	PERCENTAGE	PPM	HEIGHT
1	97.8	42.712	97.8
2	126.0	41.943	126.0

Table 1, entry 10a
31-P/1-H DECOUPLED

Table 1, entry 10a
31-P/1-H COUPLED

```

exp1 s2pu1
SAMPLE
date Mar 17 2006
solvent CDCl3
file ACQUISITION exp
sw 26738.0 pw90
at 1.598 hst 0.008
np 85476 alfa 18.300
fb 14800 il 20.000
bs 64 in n
ss 4 dp n
di 1.000 hs nm
nt 16 lb
ct 16 fn
TRANSMITTER P31
tn P31
sfrq 121.474 sp
tof 10608.2 wp
tpwr 55 rfl 10010.0
pw 7.117 rfp 2437.3
DECOUPLER H1 lp 144.8
dn dof 0
dm ynn wc 250
dmm w sc 0
dpwr 35 vs 230
dmf 6700 tn no ph 12

```

Table 1, entry 10a

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2324.437	7.747	37.9	40	350.615	1.169	29.6
2	2189.878	7.298	5.1	41	340.061	1.133	28.7
3	1795.290	5.983	60.1	42	332.732	1.109	28.0
4	1725.812	5.752	11.3	43	291.397	0.971	193.1
5	1718.189	5.726	17.7	44	284.069	0.947	162.0
6	1687.115	5.623	13.1	45	276.740	0.922	84.4
7	1256.761	4.188	5.1	46	263.841	0.879	32.8
8	1249.432	4.164	16.9	47	256.219	0.854	25.8
9	1240.931	4.136	29.3	48	-0.000	-0.000	23.6
10	1233.602	4.111	25.1				
11	1226.273	4.087	11.2				
12	1220.410	4.067	11.5				
13	1213.374	4.044	27.0				
14	1205.752	4.018	31.8				
15	1198.423	3.994	18.5				
16	1189.629	3.965	6.0				
17	790.056	2.633	15.2				
18	764.845	2.549	15.5				
19	615.922	2.053	68.3				
20	578.691	1.929	29.2				
21	572.534	1.908	27.6				
22	559.929	1.866	28.5				
23	548.202	1.827	28.9				
24	541.753	1.806	29.9				
25	520.939	1.736	23.0				
26	514.489	1.715	53.6				
27	507.161	1.690	73.5				
28	500.125	1.667	63.4				
29	493.382	1.644	36.5				
30	484.294	1.614	26.0				
31	444.425	1.481	12.0				
32	437.096	1.457	35.3				
33	429.767	1.432	61.3				
34	422.145	1.407	66.9				
35	414.816	1.382	38.2				
36	407.781	1.359	17.1				
37	377.292	1.257	80.0				
38	362.928	1.210	13.3				
39	357.651	1.192	12.4

Table 1, entry 10a

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	10009.692	132.670	25.4	16	2792.573	37.013	24.3
2	10000.767	132.552	29.3	17	2706.778	35.876	30.6
3	9997.024	132.502	25.8	18	2699.005	35.773	22.7
4	9988.387	132.388	28.9	19	2465.804	32.682	58.0
5	9044.934	119.883	26.1	20	2460.046	32.606	56.4
6	9038.312	119.795	26.5	21	1869.560	24.779	38.2
7	9029.675	119.681	32.7	22	1865.529	24.726	54.3
8	9023.341	119.597	29.2	23	1861.499	24.673	37.2
9	5863.620	77.717	71.9	24	1616.782	21.429	53.0
10	5831.663	77.294	75.4	25	1607.569	21.307	51.1
11	5799.706	76.870	73.4	26	1563.233	20.719	38.0
12	5022.947	66.575	25.0	27	1556.899	20.635	35.4
13	5015.462	66.476	51.1	28	1554.308	20.601	36.2
14	5007.688	66.373	29.6	29	1547.974	20.517	26.2
15	2800.922	37.124	30.7	30	1433.101	18.995	88.4
16	2792.573	37.013	24.3	31	1041.843	13.809	85.0

Table 1, 10b
31-P/1-H DECOUPLED

```

880
exp1 s2pu1
SAMPLE
date Mar 14 2006
solvent CDC13
file exp
ACQUISITION
sw 26738.0
at 1.598
np 85476
fb 14800
bs 64
ss 4
d1 1.000
nt 8
ct 8
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
not used
DISPLAY
tn p31
sfrq 121.474
tof 10608.2
tpwr 55
pw 7.117
DECOUPLER
H1
dn 0
dof 0
dm yyy
dmm w
dpwr 35
dmf 6700
ai no
ph
wc 250
sc 0
vs 36
th 4
PLOT
-1342.5
77.0
rfp
rfi
wp
sp
0
10010.0
2437.3
0

```

Table 1, entry 10b
31-P/1-H COUPLED

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	5447.051	44.845	145.8	1	5447.051	44.845	145.8
2	4908.505	40.412	157.1	2	4908.505	40.412	157.1
3	4368.736	35.968	19.4	3	4368.736	35.968	19.4

Table 1, entry 10b

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	3558.039	11.858	8.2	40	553.479	1.845	10.9
2	2432.318	8.106	5.4	41	551.134	1.837	11.4
3	2407.400	8.023	9.5	42	510.972	1.703	44.2
4	2370.756	7.901	2.5	43	508.333	1.694	51.1
5	2302.743	7.675	20.1	44	492.503	1.641	16.9
6	2299.812	7.665	21.2	45	487.226	1.624	5.5
7	2185.774	7.285	6.4	46	484.001	1.613	7.2
8	2181.377	7.270	2.1	47	481.070	1.603	7.9
9	2175.513	7.250	2.2	48	475.793	1.586	14.9
10	2174.048	7.246	2.3	49	471.395	1.571	10.4
11	1818.156	6.059	2.4	50	468.171	1.560	8.9
12	1760.111	5.866	20.1	51	465.239	1.551	8.7
13	1757.179	5.856	20.9	52	418.920	1.396	7.4
14	1623.500	5.411	10.6	53	406.315	1.354	10.1
15	1613.240	5.377	10.8	54	403.090	1.343	8.6
16	1591.253	5.303	6.5	55	396.054	1.320	5.9
17	1407.737	4.692	3.0	56	393.709	1.312	7.3
18	1039.239	3.464	2.7	57	389.898	1.299	15.3
19	890.902	2.969	73.4	58	383.155	1.277	4.6
20	867.157	2.890	56.6	59	377.879	1.259	6.9
21	866.570	2.888	62.4	60	376.413	1.254	6.2
22	806.180	2.687	2.8	61	325.404	1.084	2.5
23	800.317	2.667	2.3	62	317.195	1.057	2.4
24	796.799	2.656	2.9	63	314.264	1.047	2.5
25	790.936	2.636	3.4	64	311.039	1.037	30.6
26	788.591	2.628	3.5	65	305.176	1.017	162.0
27	785.366	2.617	3.6	66	299.606	0.999	9.1
28	783.021	2.610	3.7	67	298.433	0.995	8.9
29	780.382	2.601	3.5	68	292.863	0.976	5.0
30	768.656	2.562	3.1	69	286.707	0.956	5.6
31	766.018	2.553	3.3	70	284.655	0.949	8.3
32	758.102	2.527	6.0	71	278.205	0.927	3.4
33	739.634	2.465	3.0	72	271.463	0.905	73.0
34	660.481	2.201	5.7	73	267.945	0.893	9.4
35	651.687	2.172	2.8	74	265.600	0.885	4.6
36	583.381	1.944	4.5	75	257.978	0.860	155.0
37	582.795	1.942	4.5	76	242.733	0.809	2.4
38	581.915	1.939	4.8	77	-0.000	-0.000	19.7
39	568.137	1.893	5.6

880

exp1 s2pul

SAMPLE SPECIAL
 date Mar 17 2006 temp not used
 solvent CDCl3 gain not used
 file exp 20
 ACQUISITION nst 0.008
 sw 18867.9 pw90 18.500
 at 1.815 alfa 20.000
 np 68492
 bs 10400 il n
 fb 64 in n
 ss 4 dp y
 d1 1.000 hs nn
 nt 1000
 ct 463 lb 1.00
 TRANSMITTER C13 fn not used
 tn 75.456 sp
 tof 737.2 wp 15844.1
 tpwr 58 rfl 1134.7
 pw 9.250 rfg 0
 DECOUPLER tp 109.9
 dn tp -312.5
 dof 0
 dm yy wc 250
 dmm w sc 0
 dpwr 35 vs 199
 dmf 6700 th 14
 al no ph

Table 1, entry 10b

890

exp1 s2pul

SAMPLE
 date Mar 17 2006
 solvent CDC13
 file exp
 ACQUISITION
 sw 26738.0
 at 1.598
 np 85476
 fb 14800
 bs 64
 ss 4
 dl 1.000
 nt 8
 ct 8
 lb 2.00
 fn not used
 tn P31
 sfrq 121.474
 tof 10608.2
 tpwr 55
 pw 7.117
 rf 10010.0
 rfp 2437.3
 rp 97.5
 lp 184.0
 wc 250
 w SC 0
 vs 43
 th 12
 ai no
 ph

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
 DISPLAY 0.
 10010.0
 2437.3
 97.5
 184.0
 250
 0
 43
 12
 no
 ph

equation 2
 31-P/1-H DECOUPLED

890

exp1 s2pul

SAMPLE
 date Mar 17 2006
 solvent CDCl3
 file exp
 ACQUISITION
 sw 26738.0
 at 1.598
 np 85478
 fb 14800
 bs 64
 ss 1.000
 dl 16
 nt 16
 ct 16
 TRANSMITTER P31
 tn 121.474
 sfrq 10608.2
 tof 55
 tpwr 7.117
 pw 149.3
 DECOUPLER H1
 dn H1
 dof 0
 dm ynn
 dnm 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 y
 dp nn
 hs 2.00
 lb not used
 fn DISPLAY
 sp 0
 wp 10010.0
 rf1 2437.3
 rfp 0
 lp -288.8
 wc 250
 sc 0
 vs 229
 th 10
 ai no
 ph

equation 2
 31-P/1-H COUPLED

INDEX	FREQUENCY	PPM	HEIGHT
1	5385.444	44.338	104.1
2	4830.171	39.767	116.0
3	4254.499	35.027	20.0

equation 2
31-P/1-H COUPLED

890

exp1 s2pu1

SAMPLE		SPECIAL	
date	Mar 17 2006	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	20
sw	4803.1	hst	0.008
at	1.994	pw90	17.200
np	19158	alra	20.000
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	597.5
tpwr	55	rfa	0
pw	8.600	rp	-108.5
dn	DECOUPLER	lp	-64.9
dof	C13	wc	250
dm	nnn	sc	0
dmm	C	vs	367
dpwr	45	th	2
dmf	13100	ai	cdc
		ph

equation 2

```

890
exp1 s2pu1
SAMPLE
date Mar 17 2006
solvent CDC13
file exp
ACQUISITION
sw 18867.9
at 1.815
np 68492
fb 10400
bs 64
ss 4
di 1.000
nt 2000
ct 771
TRANSMITTER C13
tn 75.456
sfrq 737.2
tof 58
tpwr 9.250
pw DECOUPLER H1
dn 0
dof 0
dm YYY
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
n
n
n
y
nn
PROCESSING
1.00
fn not used
DISPLAY
-150.9
15844.1
1134.7
71.5
-256.3
PLOT
250
0
249
11
ai no ph

```

equation 2