General Synthesis of P-Stereogenic Compounds: The Menthyl Phosphinate Approach.

Olivier Berger and Jean-Luc Montchamp*

Department of Chemistry, Box 298860, Texas Christian University, Fort Worth, Texas 76129

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General Chemistry:

^{1}H NMR spectra were recorded on a 300 or 400-MHz spectrometer. Chemical shift for ^{1}H NMR spectra (in parts per million) relative to internal tetramethylsilane (Me_4Si, δ = 0.00 ppm) with $CDCl_3$. ^{13}C NMR spectra were recorded at 75.5 or 101 MHz. Chemical shifts for ^{13}C NMR spectra are reported (in parts per million) relative to $CDCl_3$ (δ = 77.0 ppm). ^{31}P NMR spectra were recorded at 121.5 or 162 MHz, and chemical shifts reported (in parts per million) relative to external 85% phosphoric acid (δ = 0.0 ppm). TLC plates were visualized by UV or immersion in permanganate potassium (3 g KMnO4, 20 g K2CO3, 5 mL 5% NaOHaq and 300 mL of water) followed by heating.

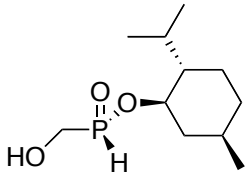
Reagent and solvents:

All starting materials were purchased from commercial sources and used as received. The solvents were distilled under N_2 and dried according to standard procedures (THF from Na/ benzophenone ketyl; DMF from $MgSO_4$; CH_3CN, toluene and dichloromethane from CaH_2).

^{31}P NMR Yield Measurements:

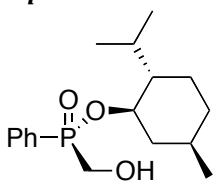
The NMR yields are determined by integration of all the resonances in the ^{31}P spectra, an approach which is valid if no phosphorus-containing gas (i.e. PH_3) evolves, or if the precipitate in a heterogeneous mixture does not contain phosphorus. The yields determined by NMR are generally accurate within ~10% of the value indicated, and are reproducible.

(R_p)-Menthyl (hydroxymethyl)-H-phosphinate 2:[1]



Paraformaldehyde (9.91 g, 330 mmol, 1.1 equiv) and hypophosphorous acid (39.6 g, 300 mmol, 1 equiv, 50% w.t. in water) were introduced in a round bottom flask and the reaction mixture was stirred for 24h at 75°C. The reaction mixture was cooled down to rt and the oil obtained was diluted in toluene (300 mL). L-menthol (46.9 g, 300 mmol, 1 equiv) was added and the reaction mixture was stirred for 24h at reflux under N_2 in a flask equipped with a Dean-Stark trap. The solvent was then removed under vacuum and the residue obtained was dissolved in a mixture of diethyl ether/hexane (50 mL: 200 mL) and the flask was placed in the fridge for 4h (2°C). The solid obtained was filtered and solubilized in diethyl ether (200 mL) and placed in the fridge (2°C) for 3h to afford the product as white needles (6.54 g, 10%, >99% de). Mp = 101-102°C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 34.9 (dm, *J* = 542 Hz); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.16 (dm, *J* = 542 Hz, 1H), 4.04-4.23 (m, 2H), 3.82-4.00 (m, 2H), 2.14-2.24 (m, 1H), 1.98-2.11 (m, 1H), 2.04 (dquint., *J* = 2.4 and 7.0 Hz, 1H), 1.62-1.73 (m, 2H), 1.34-1.52 (m, 2H), 1.24 (q, *J* = 12.0 Hz, 1H), 0.93 (d, *J* = 6.7 Hz, 6H), 0.76-1.10 (m, 2H), 0.80 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 79.3 (d, J_{POC} = 8.3 Hz), 59.7 (d, J_{PC} = 111 Hz), 48.5 (d, J_{POCC} = 5.5 Hz), 43.3, 33.8, 31.5, 25.6, 22.9, 21.8, 20.8, 15.7; HRMS (ESI+) m/z calcd for $C_{11}H_{23}O_3P$ ($[M+Na]^+$) 257.1385, found 257.1423; $[\alpha]_D^{22}$ = -61.4° (chloroform).

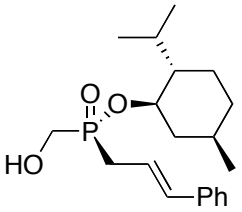
(S_p)-Menthyl (hydroxymethyl)phenylphosphinate 3:[1]



To a solution of phenylphosphinic acid (42.6 g, 300 mmol, 1 equiv) in toluene (300 mL) was added L-menthol (46.9 g, 300 mmol, 1 equiv). The reaction mixture was stirred at reflux for 24 h under N_2 in a

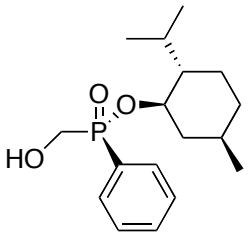
flask equipped with a Dean-stark trap. After cooling down the reaction to rt, paraformaldehyde (9.01 g, 300 mmol, 1 equiv) was added and the reaction mixture was stirred at reflux for 24 h under N_2. The solvent was then removed under vacuum and the crude obtained was recrystallized at rt in diethyl ether (200 mL) to afford the product as colorless crystals (24.2 g, 26%, 97% de). Mp = 138-139°C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 37.2 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.77-7.87 (m, 2H), 7.52-7.60 (m, 1H), 7.42-7.51 (m, 2H), 4.29-4.43 (m, 2H), 3.93-4.10 (m, 2H), 2.26 (dquint., *J* = 2.6 and 7.0 Hz, 1H), 1.80-1.91 (m, 1H), 1.57-1.73 (m, 2H), 1.26-1.47 (m, 2H), 0.96 (d, *J* = 7.1 Hz, 3H), 0.74-1.13 (m, 3H), 0.89 (d, *J* = 7.0 Hz, 3H), 0.78 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 132.3 (d, J_{PCCCC} = 2.8 Hz), 131.7 (d, J_{PCCC} = 9.9 Hz, 2C), 130.6 (d, J_{PC} = 123 Hz), 128.3 (d, J_{PCC} = 12.1 Hz, 2C), 77.1 (d, J_{POC} = 8.3 Hz), 60.2 (d, J_{PC} = 117 Hz), 48.7 (d, J_{POCC} = 6.1 Hz), 43.2, 34.0, 31.4, 25.5, 22.8, 21.9, 21.1, 15.7; HRMS (EI+) m/z calcd for $C_{16}H_{28}O_3P$ ($[M+H]^+$) 311.1776, found 311.1766; $[\alpha]_D^{22}$ = -46.7° (chloroform).

(R_p)-Menthyl cinnamyl(hydroxymethyl)phosphinate 4:[1]



To a solution of cinnamylphosphinic acid (9.11 g, 50 mmol, 1 equiv) in toluene (100 mL) was added L-menthol (7.81 g, 50 mmol, 1 equiv). The reaction mixture was stirred at reflux for 24 h under N_2 in a flask equipped with a Dean-Stark trap. After cooling down the reaction to rt, paraformaldehyde (1.5 g, 50 mmol, 1 equiv) was added and the reaction mixture was stirred at reflux for 24 h under N_2. The solvent was then removed under vacuum and the crude obtained was recrystallized at rt in a mixture ethyl acetate/diethyl ether (30 mL : 150 mL) to afford the product as a white solid (5.6 g, 32%, > 99% de). Mp = 145-146°C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 48.8 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.19-7.39 (m, 5H), 6.55 (dd, *J* = 4.7 and 15.8 Hz, 1H), 6.12-6.27 (m, 1H), 4.20-4.34 (m, 1H), 3.87 (s, 2H), 3.64 (s, 1H), 2.85 (dd, *J* = 7.6 and 17.6 Hz, 2H), 2.06-2.22 (m, 2H), 1.60-1.71 (m, 2H), 1.28-1.54 (m, 2H), 1.15 (q, *J* = 11.7 Hz, 1H), 0.74-1.07 (m, 2H), 0.91 (d, *J* = 6.4 Hz, 3H), 0.86 (d, *J* = 6.8 Hz, 3H), 0.77 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 136.8 (d, J_{PCCCC} = 3.3 Hz), 135.0 (d, J_{PCC} = 12.2 Hz), 128.5 (2C), 127.5, 126.2 (d, J_{PCCCCC} = 1.7 Hz, 2C), 118.4 (d, J_{PCCC} = 10.5 Hz), 76.7 (d, J_{POC} = 8.3 Hz), 59.5 (d, J_{PC} = 106 Hz), 48.6 (d, J_{POCC} = 5.6 Hz, 2C), 43.5, 34.0, 31.6 (d, J_{PC} = 87.3 Hz), 31.5, 25.5, 22.7, 22.1, 21.0, 15.5; HRMS (EI+) m/z calcd for $C_{20}H_{31}O_3P$ ($[M]^+$) 350.2011, found 350.2012; $[\alpha]_D^{24}$ = -51.6° (chloroform).

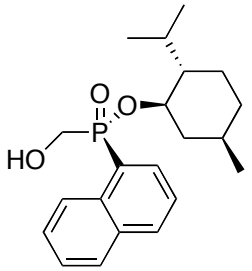
(R_p)-Menthyl (hydroxymethyl)phenylphosphinate 3:[1]



In a round bottom flask was introduced *(R_p)-2* (117 mg, 0.5 mmol, 1 equiv), $Pd(OAc)_2$ (2.3 mg, 0.01 mmol, 2.0 mol %), xantphos (6.4 mg, 0.011 mmol, 2.2 mol %), a mixture of DMF and 1,2-dimethoxyethane (2.25 mL : 0.25 mL), DIPEA (0.11 mL, 0.65 mmol, 1.3 equiv) and bromobenzene (0.05 mL, 0.5 mmol, 1 equiv). The reaction mixture was stirred under a flow of N_2 for 10 minutes and then heated at 115°C for 24 hours before cooling down to rt. The solvent was then removed under vacuum and the resulting residue was dissolved in ethyl acetate and washed with a saturated aqueous solution of $NaHCO_3$ and brine. The organic layer was dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (hexane/ethyl acetate 5:5 to 3:7) to afford the product as a white solid (106 mg, 68%, de = 95%). Mp

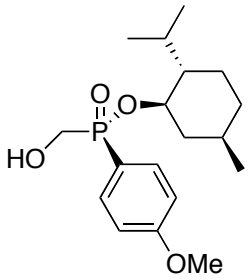
= 103-105°C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 37.4 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.80-7.91 (m, 2H), 7.45-7.62 (m, 3H), 4.09-4.21 (m, 1H), 4.02-4.08 (m, 2H), 2.77-2.87 (m, 1H), 2.29-2.39 (m, 1H), 1.90-2.05 (m, 1H), 1.58-1.69 (m, 3H), 1.22-1.50 (m, 2H), 0.93 (d, *J* = 6.2 Hz, 3H), 0.85 (d, *J* = 7.0 Hz, 3H), 0.76-1.02 (m, 2H), 0.47 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 132.3 (d, J_{PCCCC} = 2.7 Hz), 131.8 (d, J_{PCCC} = 9.9 Hz, 2C), 129.4 (d, J_{PC} = 124 Hz), 128.4 (d, J_{PCC} = 12.1 Hz, 2C), 77.4 (d, J_{POC} = 8.3 Hz), 60.4 (d, J_{PC} = 115 Hz), 48.6 (d, J_{POCC} = 6.0 Hz), 43.6, 34.0, 31.5, 25.4, 22.6, 22.0, 21.0, 15.2; HRMS (EI+) m/z calcd for $C_{17}H_{27}O_3P$ ($[M+H]^+$) 311.1776, found 311.1773; $[\alpha]_D^{22}$ = -37.9° (chloroform).

(R_p)-Menthyl (hydroxymethyl)-1-naphtylphosphinate 5:[1]



In a round bottom flask was introduced *(R_p)-2* (117 mg, 0.5 mmol, 1 equiv), $Pd(OAc)_2$ (2.3 mg, 0.01 mmol, 2.0 mol %), xantphos (6.4 mg, 0.011 mmol, 2.2 mol %), a mixture of DMF and 1,2-dimethoxyethane (2.25 mL : 0.25 mL), DIPEA (0.11 mL, 0.65 mmol, 1.3 equiv) and 1-bromonaphthalene (0.06 mL, 0.5 mmol, 1 equiv). The reaction mixture was stirred under a flow of N_2 for 10 minutes and then heated at 115°C for 24 hours before cooling down to rt. The solvent was then removed under vacuum and the resulting residue was dissolved in ethyl acetate and washed with a saturated aqueous solution of $NaHCO_3$ and brine. The organic layer was dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (hexane/ethyl acetate 5:5 to 0:10) to afford the product as a white solid (152 mg, 84%, 94% de). Mp = 102-103°C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 38.6 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.54-8.60 (m, 1H), 8.20-8.30 (m, 1H), 8.03-8.10 (m, 1H), 7.88-7.96 (m, 1H), 7.52-7.64 (m, 3H), 4.29-4.43 (m, 1H), 4.08-4.27 (m, 2H), 2.35-2.44 (m, 1H), 1.88-2.00 (m, 1H), 1.59-1.74 (m, 3H), 1.35-1.54 (m, 3H), 0.96 (d, *J* = 6.2 Hz, 3H), 0.84-1.04 (m, 2H), 0.74 (d, *J* = 7.0 Hz, 3H), 0.44 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 134.3 (d, J_{PCCC} = 7.7 Hz), 133.6 (d, J_{PCCC} = 9.4 Hz), 133.5 (d, J_{PCCCC} = 2.7 Hz), 133.0 (d, J_{PCC} = 11.6 Hz), 129.0, 127.3, 126.2, 126.2 (d, J_{PCCC} = 3.3 Hz), 126.1 (d, J_{PC} = 121 Hz), 124.7 (d, J_{PCC} = 13.8 Hz), 78.0 (d, J_{POC} = 8.3 Hz), 61.8 (d, J_{PC} = 111 Hz), 48.7 (d, J_{POCC} = 5.0 Hz), 43.6, 34.0, 31.7, 25.4, 22.7, 22.1, 20.9, 15.2; HRMS (EI+) m/z calcd for $C_{21}H_{29}O_3P$ ($[M]^+$) 360.1854, found 360.1860; $[\alpha]_D^{22}$ = -52.3° (chloroform).

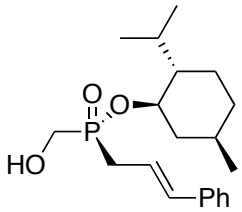
(R_p)-Menthyl (hydroxymethyl)p-anisylphosphinate 6:[1]



In a round bottom flask was introduced *(R_p)-2* (117 mg, 0.5 mmol, 1 equiv), $Pd(OAc)_2$ (2.3 mg, 0.01 mmol, 2.0 mol %), xantphos (6.4 mg, 0.011 mmol, 2.2 mol %), a mixture of DMF and 1,2-dimethoxyethane (2.25 mL : 0.25 mL), DIPEA (0.11 mL, 0.65 mmol, 1.3 equiv) and 4-bromoanisole (0.06 mL, 0.5 mmol, 1 equiv). The reaction mixture was stirred under a flow of N_2 for 10 minutes and then heated at 115°C for 24 hours before cooling down to rt. The solvent was then removed under vacuum and the resulting residue was dissolved in ethyl acetate and washed with a saturated aqueous solution of $NaHCO_3$ and brine. The organic layer was dried over $MgSO_4$, filtered and

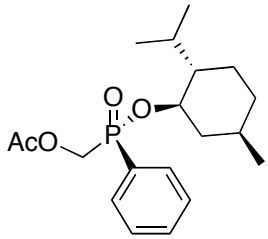
concentrated under vacuum. The crude obtained was purified by column chromatography (hexane/ethyl acetate 5:5 to 0:10) to afford the product as a white solid (90 mg, 53%, 81% de). Mp = 110-112°C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 37.8 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.74-7.84 (m, 2H), 6.96-7.03 (m, 2H), 4.05-4.18 (m, 1H), 3.96-4.05 (m, 2H), 3.87 (s, 3H), 2.60-2.71 (m, 1H), 2.29-2.39 (m, 1H), 2.01 (dquint., *J* = 2.6 and 7.3 Hz, 1H), 1.58-1.69 (m, 3H), 1.20-1.48 (m, 2H), 0.93 (d, *J* = 6.5 Hz, 3H), 0.87 (d, *J* = 6.7 Hz, 3H), 0.76-1.02 (m, 2H), 0.51 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 162.8 (d, J_{PCCCC} = 2.8 Hz), 133.7 (d, J_{PCCC} = 11.1 Hz, 2C), 120.5 (d, J_{PC} = 131 Hz), 114.0 (d, J_{PCC} = 13.2 Hz, 2C), 77.2 (d, J_{POC} = 7.7 Hz), 60.5 (d, J_{PC} = 117 Hz), 55.3, 48.7 (d, J_{POCC} = 6.0 Hz), 43.6, 34.0, 31.5, 25.4, 22.7, 22.0, 21.0, 15.3; HRMS (EI+) m/z calcd for $C_{18}H_{29}O_4P$ ([M]$^+$) 340.1803, found 340.1801; $[\alpha]_D^{24}$ = -68.3° (chloroform).

(R_p)-Menthyl cinnamyl(hydroxymethyl)phosphinate 4:[1]



To a solution of *(R_p)-2* (468 mg, 2 mmol, 1 equiv, > 99% de) in *tert*-amyl alcohol (10 mL) was added Pd_2dba_3 (18.3 mg, 0.02 mmol, 1 mol%), xantphos (23.2 mg, 0.04 mmol, 2 mol%) and cinnamyl alcohol (0.26 mL, 2 mmol, 1 equiv). The reaction mixture was stirred at reflux for 20 h under N_2 in a flask equipped with a Dean-Stark trap. After cooling down the reaction to rt, the solvent was removed under vacuum and the residue obtained was purified by column chromatography (dichloromethane/acetone 100:0 to 90:10) to afford the product as a white solid (681 mg, 97%, > 99% de). Mp = 145-146°C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 48.8 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.19-7.39 (m, 5H), 6.55 (dd, *J* = 4.7 and 15.8 Hz, 1H), 6.12-6.27 (m, 1H), 4.20-4.34 (m, 1H), 3.87 (s, 2H), 3.64 (s, 1H), 2.85 (dd, *J* = 7.6 and 17.6 Hz, 2H), 2.06-2.22 (m, 2H), 1.60-1.71 (m, 2H), 1.28-1.54 (m, 2H), 1.15 (q, *J* = 11.7 Hz, 1H), 0.74-1.07 (m, 2H), 0.91 (d, *J* = 6.4 Hz, 3H), 0.86 (d, *J* = 6.8 Hz, 3H), 0.77 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 136.8 (d, J_{PCCCC} = 3.3 Hz), 135.0 (d, J_{PCC} = 12.2 Hz), 128.5 (2C), 127.5, 126.2 (d, J_{PCCCCC} = 1.7 Hz, 2C), 118.4 (d, J_{PCCC} = 10.5 Hz), 76.7 (d, J_{POC} = 8.3 Hz), 59.5 (d, J_{PC} = 106 Hz), 48.6 (d, J_{POCC} = 5.6 Hz, 2C), 43.5, 34.0, 31.6 (d, J_{PC} = 87.3 Hz), 31.5, 25.5, 22.7, 22.1, 21.0, 15.5; HRMS (EI+) m/z calcd for $C_{20}H_{31}O_3P$ ([M]$^+$) 350.2011, found 350.2012; $[\alpha]_D^{24}$ = -51.6° (chloroform).

(R_p)-Menthyl (acetoxymethyl)phenylphosphinate 3a:[2]

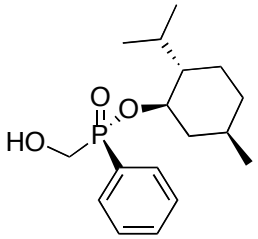


To a solution of *(R_p)-2* (703 mg, 3 mmol, 1 equiv, >99% de) in dichloromethane (15 mL) at 0°C under N_2 was added pyridine (0.30 mL, 3.75 mmol, 1.25 equiv) and acetic anhydride (0.34 mL, 3.6 mmol, 1.2 equiv). The ice-bath was removed and the reaction mixture was stirred for 16h at rt. The solvent was removed under vacuum and the residue obtained was solubilized in ethyl acetate. The organic layer was washed with $NaHCO_3$ and brine, dried over $MgSO_4$, filtered and concentrated under vacuum to afford the product as a white solid (829 mg, 100%, 94% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 26.8 (dm, *J* = 567 Hz); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.14 (dt, *J* = 1.8 and 567 Hz, 1H), 4.09-4.21 (m, 2H), 3.94-4.05 (m, 1H), 2.01-2.08 (m, 1H), 1.95 (s, 3H), 1.83-1.92 (m, 1H), 1.47-1.55 (m, 2H), 1.20-1.36 (m, 2H), 1.10 (q, *J* = 11.1 Hz, 1H), 0.79-0.92 (m, 1H), 0.60-0.79 (m, 1H), 0.76 (d, *J* = 7.1 Hz, 3H), 0.75 (d, *J* = 6.4 Hz, 3H), 0.63 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 170.0 (d, J_{PCOC} = 6.5 Hz),

79.6 (d, J_{POC} = 7.8 Hz), 60.0 (d, J_{PC} = 113 Hz), 48.4 (d, J_{POCC} = 6.1 Hz), 43.2, 33.6, 31.4, 25.5, 22.8, 21.7, 20.7, 20.1, 15.6.

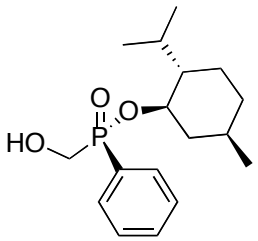
To a suspension of $Mn(OAc)_2$ (8.7 mg, 0.05 mmol, 5 mol%), MnO_2 (261 mg, 3 mmol, 3 equiv.), sodium acetate (246 mg, 3 mmol, 3 equiv.) and benzene (1.8 mL, 20 mmol, 20 equiv.) in acetic acid (2.5 mL) at 70^0C under N_2 was added a solution of *(R_p)-7* (276 mg, 1 mmol, 1 equiv, 94% de) in acetic acid (2.5 mL) over 2 hours *via* a syringe pump. The reaction mixture was then stirred for an additional 2h at 70^0C under N_2. Ethyl acetate (~ 30 mL) and an aqueous solution of $Na_2S_2O_4$ 0.2M saturated with $NaHCO_3$ (~40 mL) were added. The biphasic suspension was stirred vigorously for 5 minutes, filtered through celite and the two layers were separated. The organic layer was washed with an aqueous solution of $Na_2S_2O_4$ 0.2M saturated with $NaHCO_3$ (~40 mL), a saturated aqueous solution of $NaHCO_3$ (~ 40 mL) and brine (~ 40 mL), dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 98:2 to 94:6) to afford the product as a yellow oil (183 mg, 52%, de = 94%). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 31.8 (s, 97%); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.72-7.81 (m, 2H), 7.47-7.54 (m, 1H), 7.37-7.45 (m, 2H), 4.41 (dm, *J* = 43.8 Hz, 2H), 4.07-4.17 (m, 1H), 2.17-2.26 (m, 1H), 1.95 (s, 3H), 1.87-1.98 (m, 1H), 1.51-1.60 (m, 2H), 1.27-1.41 (m, 2H), 1.21 (q, *J* = 11.4 Hz, 1H), 0.69-0.92 (m, 2H), 0.84 (d, *J* = 6.4 Hz, 3H), 0.77 (d, *J* = 7.0 Hz, 3H), 0.44 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 169.8 (d, J_{PCOC} = 8.3 Hz), 132.7 (d, J_{PCCCC} = 2.5 Hz), 131.7 (d, J_{PCCC} = 9.9 Hz, 2C), 129.2 (d, J_{PC} = 131 Hz), 128.4 (d, J_{PCC} = 13.0 Hz, 2C), 77.8 (d, J_{POC} = 7.5 Hz), 60.6 (d, J_{PC} = 120 Hz), 48.6 (d, J_{POCC} = 6.3 Hz), 43.5, 33.9, 31.5, 25.4, 22.7, 21.9, 21.0, 20.4, 15.3; HRMS (CI+, methane) m/z calcd for $C_{19}H_{30}O_4P$ ($[M+H]^+$) 353.1882, found 353.1873; $[\alpha]_D^{27}$ = -78.2^0 (chloroform).

(R_p)-Menthyl (hydroxylmethyl)phenylphosphinate 3b:[1]



To a solution of *(R_p)-3a* (150 mg, 0.43 mmol, 1 equiv) in methanol (2 mL) was added potassium carbonate (6 mg, 0.043 mmol, 0.1 equiv.) and the mixture was stirred for 20 h at rt. The solvent was removed under vacuum and then the residue was solubilized in EtOAc (20 mL). Water (20 mL) and $NaHSO_4$ were added until the pH was around 1. The aqueous layer was saturated with NaCl and the 2 layers were separated. The organic layer was washed with saturated $NaHCO_3$ (20 mL) and brine (20 mL), dried over $MgSO_4$, filtered and concentrated under vacuum to afford the product as a white solid (123 mg, 92%, 94% de). Mp = 103-105^{0}C; ^{31}P NMR (121.47 MHz, $CDCl_3$): δ = 37.4 (s); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.80-7.91 (m, 2H), 7.45-7.62 (m, 3H), 4.09-4.21 (m, 1H), 4.02-4.08 (m, 2H), 2.77-2.87 (m, 1H), 2.29-2.39 (m, 1H), 1.90-2.05 (m, 1H), 1.58-1.69 (m, 3H), 1.22-1.50 (m, 2H), 0.93 (d, *J* = 6.2 Hz, 3H), 0.85 (d, *J* = 7.0 Hz, 3H), 0.76-1.02 (m, 2H), 0.47 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 132.4, 131.8 (d, J_{PCCC} = 9.7 Hz, 2C), 129.4 (d, J_{PC} = 124 Hz), 128.4 (d, J_{PCC} = 12.4 Hz, 2C), 77.5 (d, J_{POC} = 7.3 Hz), 60.4 (d, J_{PC} = 115 Hz), 48.7 (d, J_{POCC} = 5.7 Hz), 43.6, 34.0, 31.6, 25.4, 22.7, 22.0, 21.0, 15.3; $[\alpha]_D^{27}$ = -69.0^0 (chloroform).

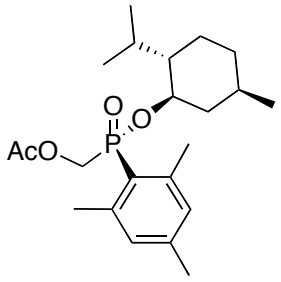
(R_p)-Menthyl (hydroxylmethyl)phenylphosphinate 3b:[1]



To a suspension of $Mn(OAc)_2$ (313 mg, 1.81 mmol, 5 mol%), MnO_2 (9.45 g, 108.6 mmol, 3 equiv.), sodium acetate (8.91 g, 108.6 mmol, 3 equiv.) and benzene (32.4 mL, 362 mmol, 10 equiv.) in acetic acid (90 mL) at 70^0C under N_2 was added a solution of *(S_p)/(R_p)-7* (10 g, 36.2 mmol, 1 equiv, ratio 54:46) in acetic acid (90 mL) over 2 hours *via* a syringe pump. The reaction mixture was then stirred for an additional 2 hours at 70^0C under N_2. Ethyl acetate (~ 250 mL) and an aqueous solution of $Na_2S_2O_4$ 0.2M saturated with $NaHCO_3$ (~ 250 mL) were added. The suspension was stirred vigorously for 5 minutes, filtered through celite and the two layers were separated. The organic layer was washed with an aqueous solution of $Na_2S_2O_4$ 0.2M saturated with $NaHCO_3$ (~ 250 mL), a saturated aqueous solution of $NaHCO_3$ (~ 250 mL) and brine (~ 250 mL), dried over $MgSO_4$, filtered and concentrated under vacuum to afford the product as a yellow oil (9.91 g, 78%). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 32.0 (s, 54%), 31.9 (s, 46%).

To a solution of *(S_p)/(R_p)-3a* (8.42 g, 24 mmol, 1 equiv, ratio 54:46) in methanol (50 mL) was added potassium carbonate (330 mg, 2.4 mmol, 0.1 equiv.) and the mixture was stirred for 20 h at rt. The solvent was removed under vacuum and then the residue was solubilized in EtOAc (100 mL). Water (100 mL) and $NaHSO_4$ were added until the pH was around 1. The aqueous layer was saturated with NaCl and the 2 layers were separated. The organic layer was washed with saturated $NaHCO_3$ (100 mL) and brine (100 mL), dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was precipitated in hexane to afford the product as a white solid (1.82 g, 24%, 95% de). Mp = 103-105^{0}C; ^{31}P NMR (121.47 MHz, $CDCl_3$): δ = 37.4 (s); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.80-7.91 (m, 2H), 7.45-7.62 (m, 3H), 4.09-4.21 (m, 1H), 4.02-4.08 (m, 2H), 2.77-2.87 (m, 1H), 2.29-2.39 (m, 1H), 1.90-2.05 (m, 1H), 1.58-1.69 (m, 3H), 1.22-1.50 (m, 2H), 0.93 (d, *J* = 6.2 Hz, 3H), 0.85 (d, *J* = 7.0 Hz, 3H), 0.76-1.02 (m, 2H), 0.47 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 132.4, 131.8 (d, J_{PCCC} = 9.7 Hz, 2C), 129.4 (d, J_{PC} = 124 Hz), 128.4 (d, J_{PCC} = 12.4 Hz, 2C), 77.5 (d, J_{POC} = 7.3 Hz), 60.4 (d, J_{PC} = 115 Hz), 48.7 (d, J_{POCC} = 5.7 Hz), 43.6, 34.0, 31.6, 25.4, 22.7, 22.0, 21.0, 15.3; $[\alpha]_D^{27}$ = -69.0^0 (chloroform).

(R_p)-menthyl (acetoxymethyl) mesitylphosphinate 8a:[2]

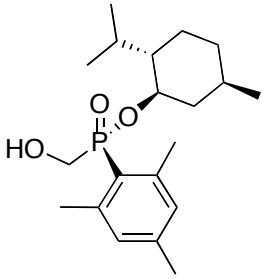


To a solution of *(R_p)-2* (703 mg, 3 mmol, 1 equiv, >99% de) in dichloromethane (15 mL) at 0°C under N_2 was added pyridine (0.30 mL, 3.75 mmol, 1.25 equiv) and acetic anhydride (0.34 mL, 3.6 mmol, 1.2 equiv). The ice-bath was removed and the reaction mixture was stirred for 16h at rt. The solvent was removed under vacuum and the residue obtained was solubilized in ethyl acetate. The organic layer was washed with $NaHCO_3$ and brine, dried over $MgSO_4$, filtered and concentrated under vacuum to afford the product as a white solid (829 mg, 100%, 94% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 26.8 (dm, *J* = 567 Hz); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.14 (dt, *J* = 1.8 and 567 Hz, 1H), 4.09-4.21 (m, 2H), 3.94-4.05 (m, 1H), 2.01-2.08 (m, 1H), 1.95 (s, 3H), 1.83-1.92 (m, 1H), 1.47-1.55 (m, 2H), 1.20-1.36 (m, 2H), 1.10 (q, *J* = 11.1 Hz, 1H), 0.79-0.92 (m, 1H), 0.60-0.79 (m, 1H), 0.76 (d, *J* = 7.1 Hz, 3H), 0.75 (d, *J* = 6.4 Hz, 3H), 0.63 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 170.0 (d, J_{PCOC} = 6.5 Hz), 79.6 (d, J_{POC} = 7.8 Hz), 60.0 (d, J_{PC} = 113 Hz), 48.4 (d, J_{POCC} = 6.1 Hz), 43.2, 33.6, 31.4, 25.5, 22.8, 21.7, 20.7, 20.1, 15.6.

To a suspension of $Mn(OAc)_2$ (26 mg, 0.15 mmol, 5 mol%), MnO_2 (783 mg, 9 mmol, 3 equiv.), sodium acetate (738 mg, 9 mmol, 3 equiv.) and mesitylene (2.1 mL, 15 mmol, 5 equiv.) in acetic acid (7.5 mL) at 70^0C under N_2 was added a solution of *(R_p)-7* (828 mg, 3 mmol, 1 equiv, 94% de) in acetic acid (7.5 mL) over 2 h *via* a syringe pump. The reaction mixture was then stirred for an additional 2h at 70^0C under N_2. Ethyl acetate (~ 50 mL) and an aqueous solution of $Na_2S_2O_4$ 0.2M saturated with $NaHCO_3$ (~ 50 mL) were added. The suspension was stirred vigorously for 10 minutes, filtered through celite and the two layers were separated. The organic layer was washed with an aqueous solution of

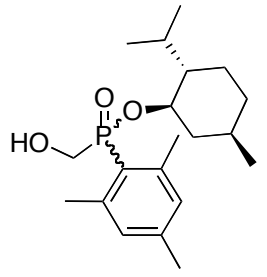
$Na_2S_2O_4$ 0.2M saturated with $NaHCO_3$ (~ 50 mL), a saturated aqueous solution of $NaHCO_3$ (~ 50 mL) and brine (~ 50 mL), dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 100:0 to 95:5) to afford the product as a colorless oil (876 mg, 79%, 94% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 36.0 (s, 3%), 35.7 (s, 97%); ^1H NMR (400 MHz, $CDCl_3$): δ = 6.80 (d, *J* = 4.0 Hz, 2H), 4.31-4.47 (m, 1H), 4.35-4.40 (m, 2H), 2.54 (s, 6H), 2.18 (s, 3H), 2.06-2.13 (m, 1H), 1.91 (s, 3H), 1.82 (dquint., *J* = 1.8 and 6.8 Hz, 1H), 1.53-1.64 (m, 2H), 1.36-1.47 (m, 1H), 1.36-1.47 (m, 1H), 1.31 (t, *J* = 11.3 Hz, 1H), 1.20 (q, *J* = 11.6 Hz, 1H), 0.94 (dq, *J* = 2.6 and 12.6 Hz, 1H), 0.74-0.89 (m, 1H), 0.86 (d, *J* = 6.5 Hz, 3H), 0.73 (d, *J* = 7.0 Hz, 3H), 0.59 (d, *J* = 6.8 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 169.8 (d, J_{PCOC} = 7.7 Hz), 143.3 (d, J_{PCC} = 11.6 Hz, 2C), 141.9 (d, J_{PCCCC} = 2.8 Hz), 130.7 (d, J_{PCCC} = 13.5 Hz, 2C), 123.3 (d, J_{PC} = 131 Hz), 76.8 (d, J_{POC} = 7.6 Hz), 62.4 (d, J_{PC} = 110 Hz), 48.6 (d, J_{POCC} = 4.7 Hz), 43.6, 34.0, 31.6, 25.7, 23.3 (d, J_{PCCC} = 2.3 Hz, 2C), 22.8, 22.0, 20.9 (2C), 20.3, 15.4; HRMS (EI+) m/z calcd for $C_{22}H_{35}O_4P$ ($[M]^+$) 394.2273, found 394.2274.

(R_p)-Menthyl (hydroxymethyl)mesitylphosphinate 8b:



To a solution of *(R_p)-8a* (876 mg, 2.22 mmol, 1 equiv, 94% de) in methanol (10 mL) was added potassium carbonate (31 mg, 0.22 mmol, 0.1 equiv.) and the mixture was stirred for 20 h at rt. The solvent was removed under vacuum and then the residue was solubilized in ethyl actate. Water and $NaHSO_4$ were added until the pH was around 1. The aqueous layer was saturated with NaCl and the 2 layers were separated. The organic layer was washed with saturated $NaHCO_3$ and brine, dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 100:0 to 90:10) to afford the product as a colorless oil (741 mg, 95%, 94% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 42.3 (s); ^1H NMR (400 MHz, $CDCl_3$): δ = 6.82 (s, 1H), 6.82 (s, 1H), 5.87 (s, 1H), 4.34-4.47 (m, 1H), 3.90-4.07 (m, 2H), 2.57 (s, 6H), 2.25-2.33 (m, 1H), 2.21 (s, 3H), 1.82-1.94 (m, 1H), 1.57-1.68 (m, 2H), 1.39-1.53 (m, 1H), 1.19-1.37 (m, 2H), 0.71-1.04 (m, 2H), 0.92 (d, *J* = 6.2 Hz, 3H), 0.77 (d, *J* = 6.9 Hz, 3H), 0.62 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 143.3 (d, J_{PCC} = 11.2 Hz, 2C), 141.6 (d, J_{PCCCC} = 2.5 Hz), 130.6 (d, J_{PCCC} = 12.7 Hz, 2C), 123.5 (d, J_{PC} = 122 Hz), 76.7 (d, J_{POC} = 7.8 Hz), 62.4 (d, J_{PC} = 107 Hz), 48.8 (d, J_{POCC} = 4.4 Hz), 43.6, 34.2, 31.7, 25.7, 23.4 (d, J_{PCCC} = 1.8 Hz, 2C), 22.9, 22.2, 21.0, 21.0, 15.4; HRMS (EI+) m/z calcd for $C_{20}H_{33}O_3P$ ($[M]^+$) 352.2167, found 352.2164; $[\alpha]_D^{25}$ = -21.1^0 (chloroform).

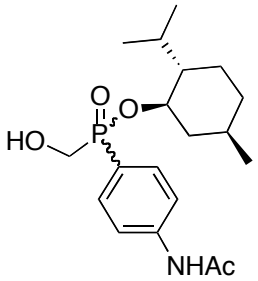
(R_p)/(S_p) Menthyl (hydroxylmethyl)mesitylphosphinate 8b:



To a suspension of $Mn(OAc)_2$ (467 mg, 2.7 mmol, 5 mol%), MnO_2 (13.92 g, 160 mmol, 3 equiv.), sodium acetate (13.12 g, 160 mmol, 3 equiv.) and mesitylene (37 mL, 266 mmol, 5 equiv.) in acetic acid (90 mL) at 70^{0}C under N_2 was added a solution of *(S_p)/(R_p)-7* (14.7 g, 53.2 mmol, 1 equiv, ratio

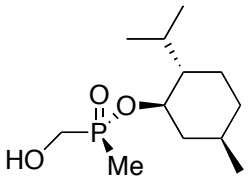
54:46) in acetic acid (90 mL) over 2 hours *via* a syringe pump. The reaction mixture was then stirred for an additional 2h at 70^{0}C under N_2. Ethyl acetate (~ 250 mL) and an aqueous solution of $Na_2S_2O_4$ 0.2M saturated with $NaHCO_3$ (~ 250 mL) were added. The suspension was stirred vigorously for 5 minutes, filtered through celite and the two layers were separated. The organic layer was washed with an aqueous solution of $Na_2S_2O_4$ 0.2M saturated with $NaHCO_3$ (~ 250 mL), a saturated aqueous solution of $NaHCO_3$ (~ 250 mL) and brine (~ 250 mL), dried over $MgSO_4$, filtered and concentrated under vacuum.
The crude obtained was solubilized in methanol (100 mL) and potassium carbonate (733 mg, 5.3 mmol, 0.1 equiv.) was added and the mixture was stirred for 20 h at rt. The solvent was removed under vacuum and then the residue was solubilized in EtOAc (150 mL). Water (150 mL) and $NaHSO_4$ were added until the pH was around 1. The aqueous layer was saturated with NaCl and the 2 layers were separated. The organic layer was washed with saturated $NaHCO_3$ (150 mL) and brine (150 mL), dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was precipitated in hexane to afford the product as a white solid (11.3 g, 60% on 3 steps). ^{31}P NMR (121.5 MHz, $CDCl_3$): δ = 41.5 (50%, s), 41.0 (50%, s); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 6.92 (s, 2H), 4.32-4.49 (m, 1H), 3.77-4.12 (m, 3H), 2.64 (s, 3H), 2.61 (s, 3H), 2.21-2.34 (m, 4H), 1.84-1.95 (m, 1H), 1.61-1.73 (m, 2H), 1.22-1.54 (m, 3H), 0.76-1.13 (m, 9.5H), 0.64 (d, *J* = 6.7 Hz, 1.5H).

(R_p)/(S_p) Menthyl-[4-(acetamido)phenyl](hydroxylmethyl)phosphinate 9b:



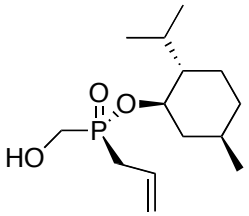
To a suspension of $Mn(OAc)_2$ (385 mg, 2.23 mmol, 5 mol%), MnO_2 (11.62 g, 133.5 mmol, 3 equiv.), sodium acetate (10.95 g, 133.5 mmol, 3 equiv.) and acetanilide (30.1 g, 222.6 mmol, 5 equiv.) in acetic acid (90 mL) at 70^{0}C under N_2 was added a solution of *(S_p)/(R_p)-7* (12.3 g, 44.5 mmol, 1 equiv) in acetic acid (90 mL) over 2 hours *via* a syringe pump. The reaction mixture was then stirred for an additional 2h at 70^{0}C under N_2. Ethyl acetate (~ 200 mL) and an aqueous solution of $Na_2S_2O_4$ 0.2M saturated with $NaHCO_3$ (~ 200 mL) were added. The suspension was stirred vigorously for 5 minutes, filtered through celite and the two layers were separated. The organic layer was washed with an aqueous solution of $Na_2S_2O_4$ 0.2M saturated with $NaHCO_3$ (~ 200 mL), a saturated aqueous solution of $NaHCO_3$ (~ 200 mL) and brine (~ 200 mL), dried over $MgSO_4$, filtered and concentrated under vacuum.
The crude obtained was solubilized in methanol (100 mL) and potassium carbonate (622 mg, 4.5 mmol, 0.1 equiv.) was added and the mixture was stirred for 20 h at rt. The solvent was removed under vacuum and then the residue was solubilized in EtOAc (150 mL). Water (150 mL) and $NaHSO_4$ were added until the pH was around 1. The aqueous layer was saturated with NaCl and the 2 layers were separated. The organic layer was washed with saturated $NaHCO_3$ (150 mL) and brine (150 mL), dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was precipitated in hexane to afford the product as a white solid (6.0 g, 37% on 3 steps). ^{31}P NMR (121.5 MHz, $CDCl_3$): δ = 37.8 (48%, s), 37.2 (52%, s); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 9.44 (s, 1H), 7.31-7.48 (m, 2H), 7.13-7.26 (m, 2H), 4.16-4.33 (m, 1.5H), 3.95-4.12 (m, 1.5H), 2.11-2.24 (m, 1H), 2.15 (s, 3H), 1.93-2.05 (m, 1H), 1.75-1.83 (m, 1H), 1.58-1.72 (m, 2H), 1.22-1.44 (m, 2H), 0.95-1.08 (m, 1H), 0.98 (d, *J* = 7.0 Hz, 1.5H), 0.93 (d, *J* = 6.4 Hz, 1.5H), 0.86 (d, *J* = 6.8 Hz, 1.5H), 0.85 (d, *J* = 6.9 Hz, 1.5H), 0.80 (d, *J* = 6.5 Hz, 1.5H), 0.55 (d, *J* = 6.9 Hz, 1.5H).

(R_p)-Menthyl (hydroxymethyl)methylphosphinate 10:[1]



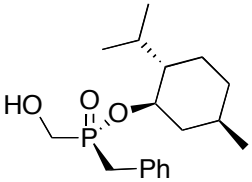
To a solution of *(R_p)-2* (234 mg, 1 mmol, 1 equiv, 98% de) in dichloromethane (10 mL) at 0°C and under N_2 was added bis(trimethylsilyl)acetamide (0.49 mL, 2 mmol, 2 equiv) followed by iodomethane (0.062 mL, 1 mmol, 1 equiv). The ice-bath was removed and the reaction mixture was then stirred for 20 h at rt. Methanol was added (0.08 mL, 2 mmol, 2 equiv) and the reaction mixture was concentrated under vacuum. The residue obtained was dissolved in ethyl acetate and the organic layer was washed with a saturated aqueous solution of $NaHCO_3$ and brine, dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 10:0 to 7:3) to afford the product as a white solid (188 mg, 76%, > 99% de). Mp = 82-83°C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 51.9 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 4.15-4.28 (m, 1H), 3.73-3.90 (m, 2H), 3.07-3.16 (m, 1H), 2.08-2.18 (m, 1H), 2.06 (dquint., *J* = 2.3 and 7.0 Hz, 1H), 1.62-1.73 (m, 2H), 1.52 (d, *J* = 13.7 Hz, 3H), 1.40-1.58 (m, 1H), 1.24-1.38 (m, 1H), 1.15 (q, *J* = 11.1 Hz, 1H), 0.93 (d, *J* = 6.7 Hz, 3H), 0.91 (d, *J* = 6.7 Hz, 3H), 0.78-1.08 (m, 2H), 0.82 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 76.2 (d, J_{POC} = 7.8 Hz), 60.6 (d, J_{PC} = 111 Hz), 48.4 (d, J_{POCC} = 5.5 Hz), 43.4, 33.9, 31.4, 25.6, 22.7, 21.9, 20.9, 15.6, 11.8 (d, J_{PC} = 91.2 Hz); HRMS (EI+) m/z calcd for $C_{12}H_{26}O_3P$ ($[M+H]^+$) 249.1620, found 249.1621; $[\alpha]_D^{22}$ = -60.6° (chloroform).

(R_p)-Menthyl allyl(hydroxymethyl)phosphinate 11:[1]



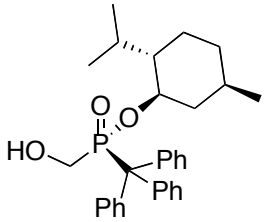
To a solution of *(R_p)-2* (117 mg, 0.5 mmol, 1 equiv, 98% de) in dichloromethane (5 mL) at 0°C and under N_2 was added bis(trimethylsilyl)acetamide (0.25 mL, 1 mmol, 2 equiv) followed by allyl bromide (0.09 mL, 1 mmol, 2 equiv). The ice-bath was removed and the reaction mixture was stirred for 36 h at rt. Methanol was added (0.04 mL, 1 mmol, 2 equiv) and the reaction mixture was then concentrated under vacuum. The residue obtained was dissolved in ethyl acetate and the organic layer was washed with a saturated aqueous solution of $NaHCO_3$ and brine, dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 100:0 to 96:4) to afford the product as white solid (88 mg, 64%, 95% de). Mp = 69-71°C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 48.4 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.74-5.93 (m, 1H), 5.19-5.32 (m, 2H), 4.18-4.32 (m, 1H), 3.81-3.89 (m, 2H), 3.53-3.64 (m, 1H), 2.64-2.77 (m, 2H), 2.06-2.18 (m, 2H), 1.61-1.72 (m, 2H), 1.40-1.54 (m, 1H), 1.24-1.39 (m, 1H), 1.15 (q, *J* = 11.5 Hz, 1H), 0.92 (d, *J* = 7.0 Hz, 6H), 0.78-1.08 (m, 2H), 0.81 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 127.2 (d, J_{PCCC} = 9.4 Hz), 120.3 (d, J_{PCC} = 12.7 Hz), 76.7 (d, J_{POC} = 8.3 Hz), 59.1 (d, J_{PC} = 107 Hz), 48.5 (d, J_{POCC} = 5.5 Hz), 43.4, 34.0, 32.4 (d, J_{PC} = 86.8 Hz), 31.5, 25.5, 22.7, 22.0, 21.0, 15.6; HRMS (EI+) m/z calcd for $C_{14}H_{27}O_3P$ ($[M]^+$) 274.1698, found 274.1694; $[\alpha]_D^{24}$ = -71.3° (chloroform).

(R_p)-Menthyl benzyl(hydroxymethyl)phosphinate 12:



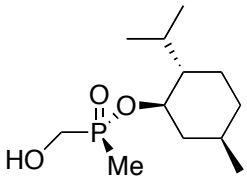
To a solution of *(R_p)-2* (1.17 g, 5 mmol, 1 equiv, 98% de) in dichloromethane (50 mL) was added at 0°C and under N_2 bis(trimethylsilyl)acetamide (2.45 mL, 10 mmol, 2 equiv) followed by benzylbromide (1.2 mL, 10 mmol, 2 equiv). The ice bath was removed and the reaction mixture was stirred for 12 h at rt. Methanol was then added (0.40 mL, 10 mmol, 2 equiv) and the mixture was concentrated under vacuum. The residue obtained was dissolved in ethyl acetate and the organic layer was washed with a saturated aqueous solution of $NaHCO_3$ and brine, dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 98:2 to 90:10) to afford the product as a white solid (1.353 g, 84%, > 99% de). Mp = 133-134°C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 47.1 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.22-7.36 (m, 5H), 4.35 (dt, *J* = 6.0 Hz, 1H), 4.16-4.26 (m, 1H), 3.71-3.86 (m, 2H), 3.17-3.32 (m, 2H), 2.02-2.09 (m, 1H), 1.85 (dquint., *J* = 2.5 and 7.0 Hz, 1H), 1.59-1.68 (m, 2H), 1.36-1.50 (m, 1H), 1.23-1.33 (m, 1H), 1.12 (q, *J* = 11.2 Hz, 1H), 0.97 (dq, *J* = 3.0 and 12.7 Hz, 1H), 0.76-0.93 (m, 1H), 0.90 (d, *J* = 6.6 Hz, 3H), 0.83 (d, *J* = 7.0 Hz, 3H), 0.67 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 131.3 (d, J_{PCC} = 7.9 Hz), 130.0 (d, J_{PCCC} = 5.6 Hz, 2C), 128.5 (d, J_{PCCCC} = 2.6 Hz, 2C), 126.7 (d, J_{PCCCCC} = 3.1 Hz), 76.6 (d, J_{POC} = 7.9 Hz), 59.2 (d, J_{PC} = 106 Hz), 48.6 (d, J_{POCC} = 5.3 Hz), 43.4, 34.1 (d, J_{PC} = 86.2 Hz), 34.0, 31.5, 25.3, 22.7, 22.1, 21.1, 15.4; HRMS (EI+) m/z calcd for $C_{18}H_{29}O_3P$ ([M]$^+$) 324.1854, found 324.1852; $[\alpha]_D^{24}$ = -27.9° (chloroform).

(R_p)-Menthyl (hydroxymethyl)triphenylmethylphosphinate 13:



To a solution of *(R_p)-2* (468 mg, 2 mmol, 1 equiv, 96% de) in dichloromethane (10 mL) was added at 0°C under N_2 bis(trimethylsilyl)acetamide (1.0 mL, 4 mmol, 2 equiv) followed by bromotriphenylmethane (1.29 g, 4 mmol, 2 equiv). The ice bath was removed and the reaction mixture was stirred for 12 h at rt. Methanol was then added (0.16 mL, 4 mmol, 2 equiv) and the mixture was concentrated under vacuum. The residue obtained was dissolved in ethyl acetate and the organic layer was washed with a saturated aqueous solution of $NaHCO_3$ and brine, dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 100:0 to 95:5) to afford the product as a white solid (465 mg, 49%, 95% de). Mp = 157-158°C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 47.7 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.43-7.55 (m, 6H), 7.22-7.35 (m, 9H), 4.27-4.47 (m, 2H), 3.75-3.88 (m, 1H), 3.44-3.55 (m, 1H), 2.18-2.27 (m, 1H), 1.48-1.67 (m, 3H), 1.34-1.45 (m, 1H), 1.03-1.19 (m, 2H), 0.76-1.01 (m, 2H), 0.90 (d, *J* = 6.3 Hz, 3H), 0.68 (d, *J* = 6.9 Hz, 3H), 0.64 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 141.3 (s, 3C), 130.9 (d, J_{PCCC} = 4.5 Hz, 6C), 128.0 (s, 6C), 127.0 (s, 3C), 78.4 (d, J_{POC} = 9.1 Hz), 63.3 (d, J_{PC} = 83.5 Hz), 60.6 (d, J_{PC} = 92.9 Hz), 49.1 (d, J_{POCC} = 5.3 Hz), 42.5, 34.0, 31.6, 24.5, 22.7, 22.2, 21.3, 15.8; HRMS (EI+) m/z calcd for $C_{30}H_{37}O_3P$ ([M]$^+$) 476.2480, found 476.2470; $[\alpha]_D^{25}$ = -9.9° (chloroform).

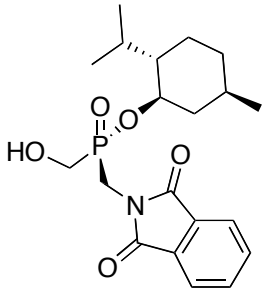
(R_p)-Menthyl (hydroxymethyl)methylphosphinate 10:[1]



To a solution of *(R_p)-2* (234 mg, 1 mmol, 1 equiv, 98% de) in tetrahydrofuran (5 mL) at -78°C and under N_2 was added a solution of LiHMDS 1.0M in tetrahydrofuran (2.0 mL, 2 mmol, 2 equiv). After 15 minutes of stirring, iodomethane (0.062 mL, 1 mmol, 1 equiv) was added at -78°C and then the reaction mixture was allowed to warm-up to room temperature over 1 hour. The mixture was then stirred for an additional 1 hour at rt. A saturated aqueous solution of NH_4Cl was added and the two

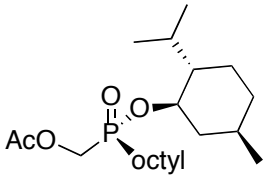
layers were separated. The aqueous layer was extracted twice with dichloromethane. The combined organic layer was washed with brine, dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 10:0 to 7:3) to afford the product as a white solid (202 mg, 81%, > 99% de). Mp = 82-83°C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 51.9 (s); ^1H NMR (400 MHz, $CDCl_3$): δ = 4.15-4.28 (m, 1H), 3.73-3.90 (m, 2H), 3.07-3.16 (m, 1H), 2.08-2.18 (m, 1H), 2.06 (dquint., *J* = 2.3 and 7.0 Hz, 1H), 1.62-1.73 (m, 2H), 1.52 (d, *J* = 13.7 Hz, 3H), 1.40-1.58 (m, 1H), 1.24-1.38 (m, 1H), 1.15 (q, *J* = 11.1 Hz, 1H), 0.93 (d, *J* = 6.7 Hz, 3H), 0.91 (d, *J* = 6.7 Hz, 3H), 0.78-1.08 (m, 2H), 0.82 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 76.2 (d, J_{POC} = 7.8 Hz), 60.6 (d, J_{PC} = 111 Hz), 48.4 (d, J_{POCC} = 5.5 Hz), 43.4, 33.9, 31.4, 25.6, 22.7, 21.9, 20.9, 15.6, 11.8 (d, J_{PC} = 91.2 Hz); HRMS (EI+) m/z calcd for $C_{12}H_{26}O_3P$ ($[M+H]^+$) 249.1620, found 249.1621; $[\alpha]_D^{22}$ = -60.6° (chloroform).

(R_p)-Menthyl hydroxymethyl(N-methylphthalimide)phenylphosphinate 14:



To a solution of *(S_p)/(R_p)-2* (4.69 g, 20 mmol, 1.0 equiv, ratio 55:45) and N-(bromomethyl)phtalimide (4.8 g, 20 mmol, 1.0 equiv) in toluene was added at rt under N_2 hexamethyldisilazane (10.4 mL, 50 mmol, 2.5 equiv) and trimethylsilyl chloride (6.35 mL, 50 mmol, 2.5 equiv). The reaction mixture was stirred for 16 hours at reflux under N_2. Methanol (2.02 mL, 50 mmol, 2.5 equiv) was added and the mixture was concentrated under vacuum. The residue was dissolved in ethyl acetate and the organic layer was washed with $NaHCO_3$ and brine, dried over $MgSO_4$, filtered and concentrated under vacuum. The residue obtained was purified by crystallization in a mixture of dichloromethane/diethyl ether (20 mL:200 mL) to afford the product as a white solid (2.06 g, 26%, >99% de). Mp = 161-162°C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 41.1 (s); ^1H NMR (400 MHz, $CDCl_3$): δ = 7.88-7.93 (m, 2H), 7.75-7.81 (m, 2H), 4.30-4.40 (m, 1H), 4.11-4.25 (m, 2H), 3.96 (d, *J* = 3.5 Hz, 2H), 3.43 (1H), 2.25-2.33 (m, 1H), 2.00 (dquint., *J* = 2.6 and 7.0 Hz, 1H), 1.62-1.71 (m, 2H), 1.42-1.56 (m, 1H), 1.31-1.39 (m, 1H), 1.28 (q, *J* = 11.1 Hz, 1H), 0.81-1.07 (m, 2H), 0.94 (d, *J* = 6.5 Hz, 3H), 0.85 (d, *J* = 7.0 Hz, 3H), 0.78 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 167.8 (2C), 134.5 (2C), 131.8 (2C), 123.8 (2C), 78.5 (d, J_{POC} = 8.1 Hz), 59.2 (d, J_{PC} = 105 Hz), 48.5 (d, J_{POCC} = 6.0 Hz), 43.6, 34.8 (d, J_{PC} = 98.0 Hz), 33.9, 31.6, 25.6, 22.8, 21.9, 20.9, 15.5; HRMS (EI+) m/z calcd for $C_{21}H_{30}O_5P$ ($[M+H]^+$) 394.1783, found 394.1777; $[\alpha]_D^{25}$ = -24.5° (chloroform).

(R_p)-Menthyl (acetoxymethyl)octylphosphinate 15:[3]

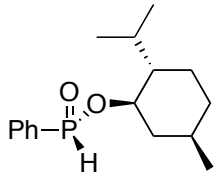


To a solution of *(R_p)-2* (703 mg, 3 mmol, 1 equiv, >99% de) in dichloromethane (15 mL) at 0°C under N_2 was added pyridine (0.30 mL, 3.75 mmol, 1.25 equiv) and acetic anhydride (0.34 mL, 3.6 mmol, 1.2 equiv). The ice-bath was removed and the reaction mixture was stirred for 16h at rt. The solvent was removed under vacuum and the residue obtained was solubilized in ethyl acetate. The organic layer was washed with $NaHCO_3$ and brine, dried over $MgSO_4$, filtered and concentrated under vacuum to afford the product as a white solid (829 mg, 100%, 95% de).[7] ^{31}P NMR (162 MHz, $CDCl_3$): δ = 26.8 (dm, *J* = 567 Hz); ^1H NMR (400 MHz, $CDCl_3$): δ = 7.14 (dt, *J* = 1.8 and 567 Hz, 1H), 4.09-4.21 (m, 2H), 3.94-4.05 (m, 1H), 2.01-2.08 (m, 1H), 1.95 (s, 3H), 1.83-1.92 (m, 1H), 1.47-1.55 (m, 2H), 1.20-

1.36 (m, 2H), 1.10 (q, J = 11.1 Hz, 1H), 0.79-0.92 (m, 1H), 0.60-0.79 (m, 1H), 0.76 (d, J = 7.1 Hz, 3H), 0.75 (d, J = 6.4 Hz, 3H), 0.63 (d, J = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 170.0 (d, J_{PCOC} = 6.5 Hz), 79.6 (d, J_{POC} = 7.8 Hz), 60.0 (d, J_{PC} = 113 Hz), 48.4 (d, J_{POCC} = 6.1 Hz), 43.2, 33.6, 31.4, 25.5, 22.8, 21.7, 20.7, 20.1, 15.6.

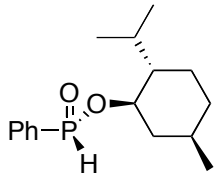
To a solution of *(R_p)-7* (553 mg, 2 mmol, 1 equiv) in DMSO (5 mL) was added 1-octene (0.31 mL, 2 mmol, 1 equiv) and $Mn(OAc)_2$ (17 mg, 0.1 mmol, 5 mol%). The reaction mixture was stirred for 16 h at 100^{0}C under air. Ethyl acetate and an aqueous solution of $Na_2S_2O_4$ at 0.5M were added and the two layers were stirred for 10 minutes and then separated. The organic layer was washed with brine, dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (hexane/ethyl acetate 8:2 to 7:3) to afford the product as a white solid (424 mg, 55%, 94% de). Mp = 57-59^{0}C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 46.3 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.97-4.12 (m, 2H), 1.88 (s, 3H), 1.49-1.59 (1H), 1.32-1.47 (m, 3H), 0.98-1.28 (m, 9H), 0.91 (q, J = 11.5 Hz, 1H), 0.71-0.84 (m, 1H), 0.54-0.72 (m, 1H), 0.68 (d, J = 7.0 Hz, 3H), 0.68 (d, J = 6.4 Hz, 3H), 0.64 (t, J = 6.8 Hz, 3H), 0.59 (d, J = 7.0 Hz, 3H); ^{13}C NMR (75.46 MHz, $CDCl_3$): δ = 169.5 (d, J_{PCOC} = 7.9 Hz), 76.2 (d, J_{POC} = 7.7 Hz), 59.6 (d, J_{PC} = 106 Hz), 48.4 (d, J_{POCC} = 6.0 Hz), 43.1, 33.8, 31.6, 31.3, 30.5 (d, J_{PCC} = 15.5 Hz), 28.8, 28.7, 27.2 (d, J_{PC} = 95.9 Hz), 25.4, 22.6, 22.4, 21.8, 20.9 (d, J_{PCCC} = 4.2 Hz), 20.8, 20.3, 15.4, 13.8; HRMS (EI+) m/z calcd for $C_{21}H_{41}O_4P$ ($[M+H]^+$) 389.2821, found 389.2812; $[\alpha]_D$ = -34.6^0.

(R_p)-Menthyl phenyl-H-phosphinate 1:[1]



To a solution of *N*-chlorosuccinimide (4.0 g, 30 mmol, 3 equiv) in dichloromethane (150 mL) at -78^{o}C and under N_2 was added dropwise a solution of dimethyl sulfide (2.2 mL, 30 mmol, 3 equiv) in dichloromethane (10 mL). After 30 minutes at -78^{o}C, a solution of *(S_p)-3* (3.1 g, 10 mmol, 1 equiv, >99% de) in dichloromethane (30 mL) was added over 20 minutes. After 1h at -78^{o}C, triethylamine (7 mL, 50 mmol, 5 equiv) was added over 15 minutes and the reaction was stirred for 30 minutes at -78^{o}C. After warming up the reaction to rt, water was added and the two layers were separated. The aqueous layer was extracted with dichloromethane (X2). The combined organic layer was dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (hexane/ethyl acetate 9:1 to 8:2) to afford the product as a colorless oil (2.58 g, 92%, >99% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 24.7 (dm, J = 553 Hz); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.73-7.84 (m, 2H), 7.66 (d, J = 553 Hz, 1H), 7.46-7.64 (m, 3H), 4.22-4.36 (m, 1H), 2.14-2.27 (m, 2H), 1.62-1.75 (m, 2H), 1.38-1.54 (m, 2H), 1.24 (q, J = 11.2 Hz, 1H), 0.78-1.13 (m, 2H), 0.96 (d, J = 7.0 Hz, 3H), 0.90 (d, J = 6.4 Hz, 3H), 0.86 (d, J = 7.0 Hz, 3H); $[\alpha]_D^{23}$ = -35.5^o (chloroform, literature with 90% de: -21.0^o in benzene).

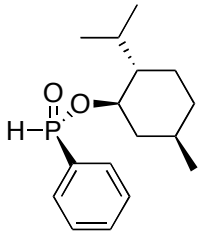
(R_p)-Menthyl phenyl-H-phosphinate 1:[1]



To a solution of *N*-chlorosuccinimide (12.95 g, 97 mmol, 3 equiv) in dichloromethane (400 mL) at -78^{o}C and under N_2 was added dropwise a solution of 1-(methylthio)dodecane (21 g, 97 mmol, 3 equiv) in dichloromethane (30 mL). After 30 minutes at -78^{o}C, a solution of *(S_p)-3* (10 g, 32 mmol, 1 equiv, >99% de) in dichloromethane (70 mL) was added over 20 minutes. After 1h at -78^{o}C, triethylamine (22.5 mL, 161 mmol, 5 equiv) was added over 15 minutes and the reaction was stirred for 30 minutes at -78^{o}C. After warming up the reaction to rt, water was added and the two layers were separated. The aqueous layer was extracted with dichloromethane (X2). The combined organic

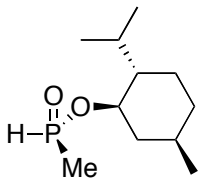
layer was dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (hexane/ethyl acetate 9:1 to 7:3) to afford the product as a colorless oil (5.8 g, 65%, >99% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 24.7 (dm, *J* = 553 Hz); ^1H NMR (400 MHz, $CDCl_3$): δ = 7.73-7.84 (m, 2H), 7.66 (d, *J* = 553 Hz, 1H), 7.46-7.64 (m, 3H), 4.22-4.36 (m, 1H), 2.14-2.27 (m, 2H), 1.62-1.75 (m, 2H), 1.38-1.54 (m, 2H), 1.24 (q, *J* = 11.2 Hz, 1H), 0.78-1.13 (m, 2H), 0.96 (d, *J* = 7.0 Hz, 3H), 0.90 (d, *J* = 6.4 Hz, 3H), 0.86 (d, *J* = 7.0 Hz, 3H); $[\alpha]_D^{23}$ = -35.5° (chloroform, literature with 90% de: -21.0° in benzene).

***(S_p)-Menthyl phenyl-H-phosphinate1:*[1]**



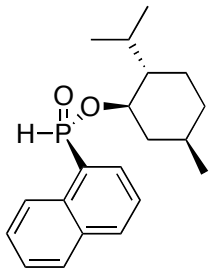
To a solution of *N*-chlorosuccinimide (110 mg, 0.82 mmol, 1.5 equiv) in dichloromethane (5 mL) at -78°C and under N_2 was added dropwise a solution of dimethyl sulfide (0.06 mL, 0.82 mmol, 1.5 equiv) in dichloromethane (1 mL). After 10 minutes at -78°C, a solution of *(R_p)-Menthyl (hydroxymethyl)phenylphosphinate* (170 mg, 0.55 mmol, 1 equiv, >99% de) in dichloromethane (2 mL) was added over 20 minutes. After 1h at -78°C, triethylamine (0.38 mL, 2.74 mmol, 5 equiv) was added over 15 minutes and the reaction was allowed to warm up to rt. After 1h at rt, water was added and the two layers were separated. The aqueous layer was extracted with dichloromethane (X2). The combined organic layer was dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (hexane/ethyl acetate 6:4) to afford the product as a colorless oil (125 mg, 81%, > 99% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 22.4 (d, *J* = 557 Hz); ^1H NMR (400 MHz, $CDCl_3$): δ = 7.67-7.82 (m, 2H), 7.68 (d, *J* = 557 Hz, 1H), 7.42-7.62 (m, 3H), 4.18-4.32 (m, 1H), 2.25-2.35 (m, 1H), 2.02-2.16 (m, 1H), 1.62-1.75 (m, 2H), 1.22-1.58 (m, 3H), 0.80-1.14 (m, 2H), 0.95 (d, *J* = 6.4 Hz, 3H), 0.88 (d, *J* = 7.0 Hz, 3H), 0.67 (d, *J* = 7.0 Hz, 3H); $[\alpha]_D^{23}$ = -77.4° (chloroform, literature with 70% de: -89.6° in benzene).

***(S_p)-Menthyl methyl-H-phosphinate 16:*[1]**



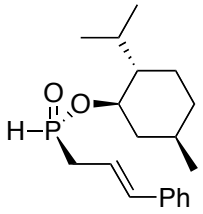
To a solution of *N*-chlorosuccinimide (470 mg, 3.5 mmol, 3 equiv) in dichloromethane (35 mL) at -78°C and under N_2 was added dropwise a solution of dimethyl sulfide (0.26 mL, 3.5 mmol, 3 equiv) in dichloromethane (3 mL). After 10 minutes at -78°C, a solution of *(R_p)-10* (290 mg, 1.17 mmol, 1 equiv, >99% de) in dichloromethane (5 mL) was added over 20 minutes. After 1h at -78°C, triethylamine (0.81 mL, 5.84 mmol, 5 equiv) was added over 15 minutes and the reaction was stirred for 30 minutes at -78°C. After warming up the reaction to rt, water was added and the two layers were separated. The aqueous layer was extracted with dichloromethane (X2). The combined organic layer was dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (hexane/ethyl acetate 8:2 to 4:6) to afford the product as a colorless oil (134 mg, 61%, 96% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 28.5 (dm, J = 537 Hz); ^1H NMR (400 MHz, $CDCl_3$): δ = 7.33 (d, *J* = 537 Hz, 1H), 4.15-4.29 (m, 1H), 2.06-2.20 (m, 2H), 1.62-1.73 (m, 2H), 1.52 (d, *J* = 15.2 Hz, 3H), 1.24-1.58 (m, 2H), 1.14 (q, *J* = 11.4 Hz, 1H), 0.93 (d, *J* = 6.2 Hz, 6H), 0.78-1.10 (m, 2H), 0.83 (d, *J* = 7.1 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 76.8 (d, J_{POC} = 7.2 Hz), 48.4 (d, J_{POCC} = 6.1 Hz), 41.8, 34.0, 31.4, 29.6, 25.7, 23.1, 22.0, 20.8, 15.9, 15.3 (d, J_{PC} = 95.6 Hz); $[\alpha]_D^{23}$ = -92.2° (chloroform, literature: -96.6° in benzene).

(S_p)-Menthyl-1-naphtyl-H-phosphinate 17:[1]



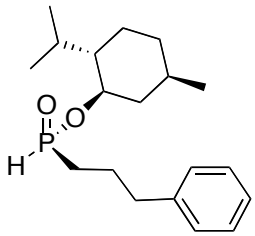
To a solution of *N*-chlorosuccinimide (100 mg, 0.75 mmol, 3 equiv) in dichloromethane (15 mL) at -78°C and under N_2 was added dropwise a solution of dimethyl sulfide (0.055 mL, 0.75 mmol, 3 equiv) in dichloromethane (2 mL). After 10 minutes at -78°C, a solution of *(R_p)-5* (90 mg, 0.25 mmol, 1 equiv, 94% de) in dichloromethane (2 mL) was added over 20 minutes. After 1h at -78°C, triethylamine (0.38 mL, 2.74 mmol, 5 equiv) was added over 15 minutes and the reaction was stirred for 30 minutes at -78°C. After warming up the reaction to rt, water was added and the two layers were separated. The aqueous layer was extracted with dichloromethane (X2). The combined organic layer was dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (hexane/ethyl acetate 9:1 to 7:3) to afford the product as a colorless oil (72 mg, 87%, 94% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 23.3 (dm, J = 557 Hz); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.45-8.51 (m, 1H), 7.99-8.10 (m, 2H), 8.05 (d, *J* = 557 Hz, 1H), 7.90-7.96 (m, 1H), 7.54-7.67 (m, 3H), 4.31-4.44 (m, 1H), 2.34-2.44 (m, 1H), 2.05 (dquint., *J* = 2.6 and 7.0 Hz, 1H), 1.61-1.74 (m, 2H), 1.24-1.56 (m, 3H), 0.97 (d, *J* = 6.4 Hz, 3H), 0.75-1.10 (m, 2H), 0.80 (d, *J* = 7.0 Hz, 3H), 0.61 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 133.6 (d, J_{PCCCC} = 2.8 Hz), 133.4 (d, J_{PCC} = 10.5 Hz), 132.5 (d, J_{PCCC} = 10.0 Hz), 131.7 (d, J_{PCCC} = 14.4 Hz), 128.9 (d, J_{PCCCC} = 1.7 Hz), 127.5, 126.7 (d, J_{PC} = 132 Hz), 126.7, 125.2 (d, J_{PCCC} = 7.2 Hz), 124.6 (d, J_{PCC} = 16.6 Hz), 77.8 (d, J_{POC} = 7.1 Hz), 48.5 (d, J_{POCC} = 6.7 Hz), 43.5, 42.2 (d, J_{POCC} = 1.1 Hz), 34.0, 31.6, 25.3, 22.8, 22.0, 20.8, 15.4; $[\alpha]_D^{23}$ = -74.0° (chloroform).

(S_p)-Menthyl cinnamyl-H-phosphinate 18:[1]



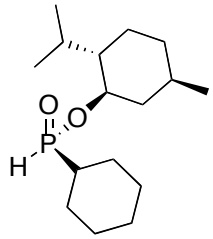
To a solution of *N*-chlorosuccinimide (200 mg, 1.5 mmol, 3 equiv) in dichloromethane (20 mL) at -78°C and under N_2 was added dropwise a solution of dimethyl sulfide (0.11 mL, 1.5 mmol, 3 equiv) in dichloromethane (3 mL). After 10 minutes at -78°C, a solution of *(R_p)-4* (175 mg, 0.5 mmol, 1 equiv, >99% de) in dichloromethane (3 mL) was added over 20 minutes. After 1h at -78°C, triethylamine (0.35 mL, 2.5 mmol, 5 equiv) was added over 15 minutes and the reaction was stirred for 30 minutes at -78°C. After warming up to rt, water was added and the two layers were separated. The aqueous layer was then extracted with dichloromethane (X2). The combined organic layer was dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (hexane/ethyl acetate 9:1 to 7:3) to afford the product as a colorless oil (132 mg, 82%, > 99% de). ^{31}P NMR (121.47 MHz, $CDCl_3$): δ = 30.9 (dm, *J* = 539 Hz); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.20-7.41 (m, 5H), 7.17 (d, *J* = 539 Hz, 1H), 6.56 (dd, *J* = 5.9 and 15.8 Hz, 1H), 6.05-6.20 (m, 1H), 4.37-4.63 (m, 1H), 2.80 (dd, *J* = 7.6 and 18.5 Hz, 2H), 2.06-2.24 (m, 2H), 1.62-1.73 (m, 2H), 1.34-1.55 (m, 2H), 1.15 (q, *J* = 11.4 Hz, 1H), 0.75-1.12 (m, 2H), 0.92 (d, *J* = 6.5 Hz, 3H), 0.91 (d, *J* = 7.0 Hz, 3H), 0.82 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (75.46 MHz, $CDCl_3$): δ = 136.8 (d, J_{PCCCC} = 3.3 Hz), 135.8 (d, J_{PCC} = 14.4 Hz), 128.6 (d, *J* = 1.1 Hz, 2C), 127.8, 126.2 (d, J_{PCCCCC} = 2.3 Hz, 2C), 117.0 (d, J_{PCCC} = 10.0 Hz), 77.3 (d, J_{POC} = 7.8 Hz), 48.4 (d, J_{POCC} = 6.1 Hz), 41.8, 34.3 (d, J_{PC} = 91.8 Hz), 34.0, 31.4, 25.7, 23.1, 21.9, 20.8, 15.8; HRMS (EI+) m/z calcd for $C_{19}H_{29}O_2P$ ($[M]^+$) 320.1905, found 320.1907; $[\alpha]_D^{23}$ = -89.8° (chloroform).

(S_p)-Menthyl (3-phenylpropyl)-H-phosphinate 19:



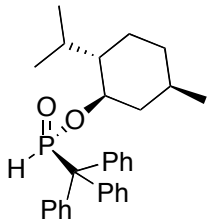
To a solution of *N*-chlorosuccinimide (721 mg, 5.4 mmol, 3 equiv) in dichloromethane (30 mL) at -78°C and under N_2 was added dropwise a solution of dimethyl sulfide (0.4 mL, 5.4 mmol, 3 equiv) in dichloromethane (5 mL). After 10 minutes at -78°C, a solution of *(R_p)-menthyl hydroxymethyl(3-phenylpropyl)-H-phosphinate* (630 mg, 1.8 mmol, 1 equiv, 98% de) in dichloromethane (5 mL) was added over 20 minutes. After 1h at -78°C, triethylamine (1.25 mL, 9 mmol, 5 equiv) was added over 15 minutes and the reaction was allowed to warm up to rt. After 1h at rt, water was added and the two layers were separated. The aqueous layer was extracted with dichloromethane (X2). The combined organic layer was dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 99:1 to 97:3) to afford the product as a colorless oil (554 mg, 96%, 96% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 33.3 (dsextuplet, *J* = 12.7 and 528 Hz); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.16-7.23 (m, 2H), 7.05-7.13 (m, 3H), 7.08 (d, *J* = 528 Hz, 1H), 4.07-4.19 (m, 1H), 2.62 (t, *J* = 7.4 Hz, 2H), 1.99-2.10 (m, 2H), 1.77-1.91 (m, 2H), 1.53-1.73 (m, 4H), 1.21-1.43 (m, 2H), 1.02 (q, *J* = 11.6 Hz, 1H), 0.94 (dquint., *J* = 3.0 and 12.4 Hz, 1H), 0.84 (d, *J* = 6.8 Hz, 6H), 0.69-0.85 (m, 1H), 0.74 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 140.7, 128.4 (2C), 128.4 (2C), 126.1, 76.7 (d, J_{POC} = 7.4 Hz), 48.3 (d, J_{POCC} = 5.9 Hz), 41.6, 36.2 (d, J_{PCC} = 16.0 Hz), 33.9, 31.3, 28.1 (d, J_{PC} = 95.0 Hz), 25.6, 22.9, 22.4 (d, J_{PCCC} = 2.5 Hz), 21.9, 20.8, 15.7; $[\alpha]_D^{23}$ = -27.3^0 (chloroform).

(S_p)-Menthyl cyclohexyl-H-phosphinate 20:



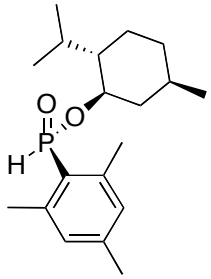
To a solution of *N*-chlorosuccinimide (400 mg, 3 mmol, 3 equiv) in dichloromethane (25 mL) at -78°C and under N_2 was added dropwise a solution of dimethyl sulfide (0.22 mL, 3 mmol, 3 equiv) in dichloromethane (3 mL). After 10 minutes at -78°C, a solution of *(S_p)-methyl (hydroxymethyl)cyclohexylphosphinate* (316 mg, 1 mmol, 1 equiv, > 99% de) in dichloromethane (5 mL) was added over 20 minutes. After 1h at -78°C, triethylamine (0.70 mL, 5 mmol, 5 equiv) was added over 15 minutes and the reaction was allowed to warm up to rt. After 1h at rt, water was added and the two layers were separated. The aqueous layer was then extracted with dichloromethane (X2). The combined organic layer was dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 100:0 to 90:10) to afford the product as a colorless oil (181 mg, 63%, > 99% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 37.1 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.84 (d, *J* = 512 Hz, 1H), 4.08-4.17 (m, 1H), 2.01-2.14 (m, 2H), 1.71-1.90 (m, 4H), 1.53-1.69 (m, 4H), 1.10-1.47 (m, 7H), 1.04 (q, *J* = 11.2 Hz, 1H), 0.99 (dq, *J* = 2.4 and 12.6 Hz, 1H), 0.78-0.95 (m, 1H), 0.89 (d, *J* = 7.0 Hz, 3H), 0.88 (d, *J* = 6.5 Hz, 3H), 0.78 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 76.4 (d, J_{POC} = 7.8 Hz), 48.4 (d, J_{POCC} = 5.8 Hz), 41.5 (d, J_{POCC} = 1.2 Hz), 37.1 (d, J_{PC} = 97.7 Hz), 34.0, 31.3, 25.8 (2C), 25.6 (d, J_{PCC} = 10.7 Hz), 25.6 (d, J_{PCC} = 9.5 Hz), 24.1 (2C), 22.9, 21.9, 20.8, 15.6.

(S_p)-Menthyl triphenylmethyl-H-phosphinate 21:



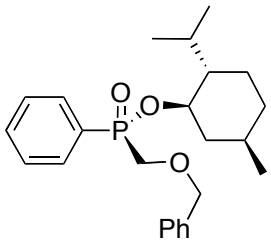
To a solution of *N*-chlorosuccinimide (100 mg, 0.75 mmol, 3 equiv) in dichloromethane (20 mL) at -78°C and under N_2 was added dropwise a solution of dimethyl sulfide (0.06 mL, 0.75 mmol, 3 equiv) in dichloromethane (2 mL). After 10 minutes at -78°C, a solution of *(R_p)-13* (119 mg, 0.25 mmol, 1 equiv, 95% de) in dichloromethane (3 mL) was added over 20 minutes. After 1h at -78°C, triethylamine (0.17 mL, 1.25 mmol, 5 equiv) was added over 15 minutes and the reaction was allowed to warm up to rt. After 1h at rt, water was added and the two layers were separated. The aqueous layer was extracted with dichloromethane (X2). The combined organic layer was dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 100:0 to 97:3) to afford the product as a white solid (93 mg, 83%, 95% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 35.4 (dd, *J* = 4.4 and 550 Hz); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.66 (d, *J* = 549 Hz, 1H), 7.27-7.37 (m, 15H), 4.21-4.32 (m, 1H), 2.03-2.11 (m, 1H), 1.57-1.69 (m, 3H), 1.35-1.48 (m, 1H), 1.15-1.24 (m, 1H), 1.00 (q, *J* = 11.4 Hz, 1H), 0.98 (dq, *J* = 3.2 and 14.7 Hz, 1H), 0.89 (d, *J* = 6.6 Hz, 3H), 0.81 (dq, *J* = 3.4 and 12.1 Hz, 1H), 0.74 (d, *J* = 7.1 Hz, 3H), 0.70 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 140.5 (d, J_{PCC} = 3.2 Hz, 3C), 130.6 (d, J_{PCCC} = 6.9 Hz, 6C), 128.2 (s, 6C), 127.2 (d, J_{PCCCCC} = 1.8 Hz, 3C), 78.7 (d, J_{POC} = 8.7 Hz), 62.0 (d, J_{PC} = 90.7 Hz), 48.6 (d, J_{POCC} = 5.8 Hz), 41.1 (d, J_{POCC} = 1.9 Hz), 33.9, 31.5, 24.8, 22.6, 22.0, 20.9, 15.5; $[\alpha]_D^{23}$ = -21.1^0 (chloroform).

(S_p)-Menthyl mesityl-H-phosphinate 22:



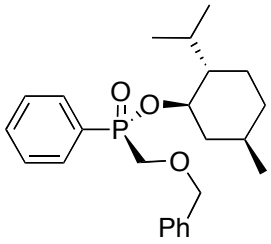
To a solution of *N*-chlorosuccinimide (400 mg, 3 mmol, 3 equiv) in dichloromethane (40 mL) at -78°C and under N_2 was added dropwise a solution of dimethyl sulfide (0.22 mL, 3 mmol, 3 equiv) in dichloromethane (3 mL). After 10 minutes at -78°C, a solution of *(R_p)-8b* (352 mg, 1 mmol, 1 equiv, 94% de) in dichloromethane (5 mL) was added over 20 minutes. After 1h at -78°C, triethylamine (0.7 mL, 5 mmol, 5 equiv) was added over 15 minutes and the reaction was allowed to warm up to rt. After 1h at rt, water was added and the two layers were separated. The aqueous layer was extracted with dichloromethane (X2). The combined organic layer was dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 100:0 to 97:3) to afford the product as a colorless oil (275 mg, 85%, 94% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 18.7 (d, *J* = 548 Hz); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.09 (d, *J* = 548 Hz, 1H), 6.89 (s, 1H), 6.88 (s, 1H), 4.27-4.37 (m, 1H), 2.58 (s, 6H), 2.27-2.34 (m, 1H), 2.31 (s, 3H), 2.17 (dquint., *J* = 2.7 and 7.0 Hz, 1H), 1.65-1.74 (m, 2H), 1.45-1.57 (m, 1H), 1.34-1.43 (m, 1H), 1.22 (q, *J* = 11.3 Hz, 1H), 1.06 (dq, *J* = 3.2 and 12.5 Hz, 1H), 0.77-0.99 (m, 1H), 0.97 (d, *J* = 6.6 Hz, 3H), 0.90 (d, *J* = 7.1 Hz, 3H), 0.80 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 142.2 (d, J_{PCCCC} = 1.9 Hz), 141.5 (d, J_{PCCC} = 11.4 Hz, 2C), 130.0 (d, J_{PCC} = 12.2 Hz, 2C), 123.7 (d, J_{PC} = 136 Hz), 76.8 (d, J_{POC} = 7.0 Hz), 48.4 (d, J_{POCC} = 6.3 Hz), 41.6, 34.0, 31.4, 25.3, 22.8, 21.9, 21.1, 20.9, 20.8 (2C), 15.4; $[\alpha]_D^{22}$ = -28.8^0 (chloroform).

(S_p)-Menthyl (benzoxymethyl)phenylphosphinate 23:



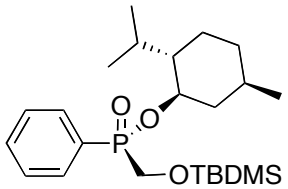
To a suspension of NaH (120 mg, 3 mmol, 1.5 equiv, 60% in mineral oil) in dichloromethane (15 mL) was added at 0°C under N_2 a solution of *(S_p)-3* (621 mg, 2 mmol, 1 equiv, >99% de) in dichloromethane (5 mL). After 30 minutes at 0°C, benzylbromide (0.29 mL, 2.4 mmol, 1.2 equiv) was added. The reaction was stirred for 4 hours at rt. A saturated solution of NH_4Cl was added and the two layers were separated. The aqueous layer was extracted with dichloromethane (3X). The combined organic layers was dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (hexane/ethyl acetate 9:1 to 7:3) to afford the product as colorless oil (801 mg, 100%, >99% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 31.3 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.80-7.91 (m, 2H), 7.52-7.62 (m, 1H), 7.42-7.52 (m, 2H), 7.24-7.34 (m, 3H), 7.13-7.21 (m, 2H), 4.56 (s, 2H), 4.29-4.43 (m, 1H), 3.75-3.94 (m, 2H), 2.18-2.33 (m, 1H), 1.90-2.01 (m, 1H), 1.56-1.72 (m, 2H), 1.22-1.48 (m, 2H), 0.74-1.17 (m, 3H), 0.92 (d, *J* = 7.0 Hz, 3H), 0.82 (d, *J* = 6.7 Hz, 3H), 0.79 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ = 137.0, 132.3 (d, J_{PCCCC} = 2.7 Hz), 131.8 (d, J_{PCCC} = 10.0 Hz, 2C), 131.2 (d, J_{PC} = 129 Hz), 128.3 (2C), 128.3 (d, J_{PCC} = 12.7 Hz, 2C), 127.8 (3C), 77.2 (d, J_{POC} = 7.2 Hz), 75.0 (d, J_{PCOC} = 12.2 Hz), 67.1 (d, J_{PC} = 119 Hz), 48.7 (d, J_{POCC} = 6.1 Hz), 43.4, 34.0, 31.5, 25.5, 22.8, 21.9, 21.1, 15.7; HRMS (EI+) m/z calcd for $C_{24}H_{34}O_3P$ ($[M+H]^+$) 401.2244, found 401.2246; $[\alpha]_D^{25}$ = -12.2° (chloroform).

(S_p)-Menthyl (benzoxymethyl)phenylphosphinate 23:



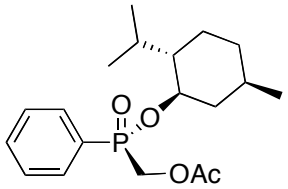
To a solution of *(S_p)-3* (3.1 g, 10 mmol, 1 equiv, >99% de) in acetonitrile (30 mL) was added benzylbromide (6 mL, 50 mmol, 5 equiv) followed by potassium fluoride on alumina (7.25 g, 50 mmol, 5 equiv, 40% w.t). The reaction was stirred for 3 days at rt under N_2. The suspension was filtered through celite. The solid was washed twice with acetonitrile and the filtrate was concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 100:0 to 95:5) to afford the product as colorless oil (3.97 g, 99%, >99% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 31.3 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.80-7.91 (m, 2H), 7.52-7.62 (m, 1H), 7.42-7.52 (m, 2H), 7.24-7.34 (m, 3H), 7.13-7.21 (m, 2H), 4.56 (s, 2H), 4.29-4.43 (m, 1H), 3.75-3.94 (m, 2H), 2.18-2.33 (m, 1H), 1.90-2.01 (m, 1H), 1.56-1.72 (m, 2H), 1.22-1.48 (m, 2H), 0.74-1.17 (m, 3H), 0.92 (d, *J* = 7.0 Hz, 3H), 0.82 (d, *J* = 6.7 Hz, 3H), 0.79 (d, *J* = 6.4 Hz, 3H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ = 137.0, 132.3 (d, J_{PCCCC} = 2.7 Hz), 131.8 (d, J_{PCCC} = 10.0 Hz, 2C), 131.2 (d, J_{PC} = 129 Hz), 128.3 (2C), 128.3 (d, J_{PCC} = 12.7 Hz, 2C), 127.8 (3C), 77.2 (d, J_{POC} = 7.2 Hz), 75.0 (d, J_{PCOC} = 12.2 Hz), 67.1 (d, J_{PC} = 119 Hz), 48.7 (d, J_{POCC} = 6.1 Hz), 43.4, 34.0, 31.5, 25.5, 22.8, 21.9, 21.1, 15.7; HRMS (EI+) m/z calcd for $C_{24}H_{34}O_3P$ ($[M+H]^+$) 401.2244, found 401.2246; $[\alpha]_D^{25}$ = -12.2° (chloroform).

(S_p)-Menthyl [(tert-butyldimethylsilyloxy)methyl]phenylphosphinate 24:



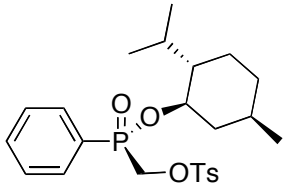
To a solution of *(S_p)-3* (3.1 g, 10 mmol, 1 equiv, >99% de) in dichloromethane (20 mL) was added at 0°C under N_2 imidazole (1.5 mL, 27 mmol, 2.7 equiv) followed by *tert*-butyldimethylsilyl chloride (2.6 mL, 15 mmol, 1.5 equiv). The ice bath was removed and the reaction was stirred for 16 h under N_2 at rt. The solvent was then removed under vacuum and the crude obtained was dissolved in ethyl acetate. The organic layer was washed with $NaHCO_3$ and brine, dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 100:0 to 95:5) to afford the product as colorless oil (4.23 g, 100%, > 99% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 35.6 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.83-7.90 (m, 2H), 7.53-7.58 (m, 1H), 7.43-7.50 (m, 2H), 4.31-4.41 (m, 1H), 4.03 (dd, *J* = 9.3 and 13.8 Hz, 1H), 3.94 (dd, *J* = 4.6 and 13.8 Hz, 1H), 2.34 (dquint., *J* = 2.5 and 7.0 Hz, 1H), 1.98-2.05 (m, 1H), 1.61-1.73 (m, 2H), 1.32-1.48 (m, 2H), 1.12 (q, *J* = 11.1 Hz, 1H), 0.85-1.09 (m, 2H), 0.97 (d, *J* = 7.1 Hz, 3H), 0.89 (d, *J* = 6.9 Hz, 3H), 0.82 (d, *J* = 6.8 Hz, 3H), 0.81 (s, 9H), -0.06 (s, 3H), -0.10 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 132.1 (d, J_{PCCCC} = 2.8 Hz), 132.1 (d, J_{PCCC} = 9.7 Hz, 2C), 130.8 (d, J_{PC} = 128 Hz), 128.0 (d, J_{PCC} = 12.5 Hz, 2C), 76.9 (d, J_{POC} = 7.5 Hz), 61.0 (d, J_{PC} = 123 Hz), 48.7 (d, J_{POCC} = 6.1 Hz), 43.5, 34.0, 31.5, 25.6 (3C), 25.4, 22.9, 21.9, 21.1, 18.2, 15.8, -5.9, -6.0; HRMS (EI+) m/z calcd for $C_{23}H_{42}O_3PSi$ ($[M+H]^+$) 425.2641, found 425.2629; $[\alpha]_D^{25}$ = -14.1° (chloroform).

(S_p)-Menthyl (acetoxymethyl)phenylphosphinate 25:[2]



To a solution of *(S_p)-3* (1.55 g, 5 mmol, 1 equiv, >99% de) in dichloromethane (10 mL) was slowly added at 0°C and under N_2 triethylamine (0.87 mL, 6.25 mmol, 1.25 equiv) followed by acetic anhydride (0.57 ml, 6 mmol, 1.2 equiv). The ice bath was removed and the reaction mixture was stirred at rt for 16 h. The solvent was removed under vacuum and the residue obtained was solubilized in ethyl acetate. The organic layer was washed with a saturated aqueous solution of $NaHCO_3$ and brine, dried over $MgSO_4$, filtered and concentrated under vacuum to afford the product as a colorless oil (1.73 g, 98%, >99% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 30.3 (s).

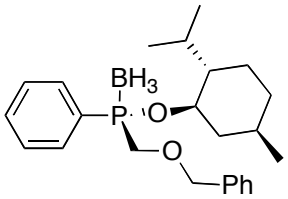
(S_p)-Menthyl [(tosyloxy)methyl]phenylphosphinate 26:[4]



To a solution of *(S_p)-3* (3.1 g, 10 mmol, 1 equiv, >99% de) in dichloromethane (20 ml) under N_2 was added *N*,*N*-diisopropylethylamine (4.4 mL, 25 mmol, 2.5 equiv). The mixture was cooled down to 0°C and a solution of tosyl chloride (2.89 g, 20 mmol, 2 equiv) in dichloromethane (15 ml) was added over 1h. The ice-bath was removed and the solution was stirred for 20h at rt. A saturated aqueous solution of $NaHCO_3$ was added and the two layers were separated. The aqueous layer was extracted with dichloromethane (2X). The combined organic layers was dried over $MgSO_4$, filtered and concentrated under vacuum. The residue obtained was purified by column chromatography (hexanes/ethyl acetate 9:1 to 7:3) to afford the product as colorless crystals (4.6 g, 99%, > 99% de). Mp = 68-70°C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 29.3 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.67-7.75 (m,

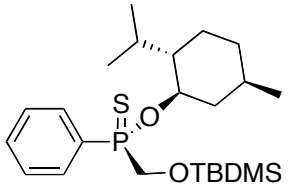
2H), 7.49-7.56 (m, 3H), 7.36-7.43 (m, 2H), 7.17-7.22 (m, 2H), 4.10-4.35 (m, 3H), 2.35 (s, 3H), 2.10 (dquint., J = 2.6 and 7.0 Hz, 1H), 1.86-1.93 (m, 1H), 1.53-1.66 (m, 2H), 1.22-1.42 (m, 2H), 1.06 (q, J = 11.1 Hz, 1H), 0.76-1.06 (m, 2H), 0.87 (d, J = 7.0 Hz, 3H), 0.75 (d, J = 6.8 Hz, 3H), 0.74 (d, J = 6.5 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 145.2, 133.0 (d, J_{PCCCC} = 2.8 Hz), 131.8 (d, J_{PCCC} = 10.0 Hz, 2C), 131.4, 129.9 (2C), 129.3 (d, J_{PC} = 137 Hz), 128.5 (d, J_{PCC} = 13.3 Hz, 2C), 128.0 (2C), 78.5 (d, J_{POC} = 7.4 Hz), 64.3 (d, J_{PC} = 115 Hz), 48.5 (d, J_{POCC} = 6.1 Hz), 43.3, 33.9, 31.5, 25.5, 22.8, 21.8, 21.6, 21.0, 15.6; HRMS (EI+) m/z calcd for $C_{24}H_{34}O_5PS$ ([M+H]$^+$) 465.1865, found 465.1857; $[\alpha]_D^{25}$ = -20.1° (chloroform).

(S_p)-Menthyl (benzoxymethyl)phenylphosphinate borane 27:



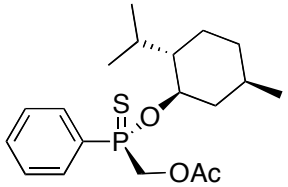
To a solution of *(S_p)-23* (400 mg, 1 mmol, 1 equiv, >99% de) in benzene (5 mL) was added triethylamine (0.7 mL, 5 mmol, 5 equiv) followed by trichlorosilane (0.5 mL, 5 mmol, 5 equiv). After 2 hours at reflux under N_2, the reaction was cooled down to rt and then borane dimethylsulfide (2.5 mL, 5 mmol, 5 equiv, 2.0M solution in tetrahydrofuran) was added and the reaction was stirred for 12 hours at rt under N_2. The solvent was removed under vacuum and the crude obtained was purified by column chromatography (hexane/ethyl acetate 100:0 to 90:10) to afford the product as colorless oil (302 mg, 76%, > 99% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 104.7 (d, J = 85.8 Hz); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.80-7.87 (m, 2H), 7.53-7.59 (m, 1H), 7.46-7.52 (m, 2H), 7.27-7.36 (m, 3H), 7.19-7.24 (m, 2H), 4.54-4.62 (m, 2H), 4.18-4.28 (m, 1H), 3.87-3.98 (m, 2H), 2.23 (dquint., J = 2.6 and 7.0 Hz, 1H), 1.84-1.91 (m, 1H), 1.61-1.72 (m, 2H), 1.34-1.49 (m, 2H), 0.74-1.30 (m, 3H), 1.04 (dq, J = 3.6 and 12.5 Hz, 1H), 0.98 (q, J = 11.1 Hz, 1H), 0.95 (d, J = 7.0 Hz, 3H), 0.77-0.90 (m, 1H), 0.84 (d, J = 6.9 Hz, 3H), 0.82 (d, J = 6.6 Hz, 3H); ^{13}C NMR (100.62 MHz, $CDCl_3$): δ = 137.1, 131.8 (d, J_{PCCCC} = 2.6 Hz), 131.5 (d, J_{PC} = 57.2 Hz), 131.2 (d, J_{PCCC} = 10.5 Hz, 2C), 128.4 (d, J_{PCC} = 10.2 Hz, 2C),128.4 (2C), 127.8, 127.8 (2C), 80.3 (d, J_{POC} = 3.8 Hz), 75.2 (d, J_{PCOC} = 7.8 Hz), 69.9 (d, J_{PC} = 56.1 Hz), 48.9 (d, J_{POCC} = 6.5 Hz), 43.5, 34.1, 31.4, 25.4, 22.9, 22.0, 21.0, 15.9; HRMS (EI+) m/z calcd for $C_{24}H_{35}BO_2P$ ([M-H]$^+$) 397.2468, found 397.2461; $[\alpha]_D^{24}$ = -0.9° (chloroform).

(R_p)-Menthyl [(tert-butyldimethylsilyloxy)methyl]phenylthiophosphinate 28a:



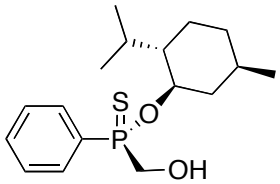
To a solution of *(S_p)-24* (2.12 g, 5 mmol, 1 equiv, >99% de) in toluene (20 mL) was added Lawesson's reagent (1.21 g, 3 mmol, 0.6 equiv). The reaction mixture was stirred for 16 hours at reflux under N_2. After cooling down the reaction to rt, the solvent was concentrated under vacuum and the residue obtained was purified by column chromatography (Hexane/ethyl acetate 98:2 to 95:5) to afford the product as a yellow oil (2.05 g, 93%, > 99% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 82.4 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.92-8.01 (m, 2H), 7.49-7.55 (m, 1H), 7.41-7.48 (m, 2H), 4.45-4.56 (m, 1H), 4.13 (dd, J = 8.1 and 13.2 Hz, 1H), 3.95 (d, J = 13.3 Hz, 1H), 2.29 (dquint., J = 2.5 and 7.0 Hz, 1H), 1.79-1.87 (m, 1H), 1.60-1.73 (m, 2H), 1.32-1.53 (m, 2H), 0.75-1.13 (m, 3H), 0.95 (d, J = 7.0 Hz, 3H), 0.89 (d, J = 6.9 Hz, 3H), 0.82 (s, 9H), 0.78 (d, J = 6.5 Hz, 3H), -0.05 (s, 3H), -0.12 (s, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 133.2 (d, J_{PC} = 101 Hz), 132.0 (d, J_{PCCC} = 10.4 Hz, 2C), 131.9 (d, J_{PCCCC} = 3.0 Hz), 127.9 (d, J_{PCC} = 12.6 Hz, 2C), 77.2 (d, J_{POC} = 7.9 Hz), 67.7 (d, J_{PC} = 99.1 Hz), 48.5 (d, J_{POCC} = 7.1 Hz), 43.4, 34.1, 31.4, 25.6 (3C), 25.3, 23.0, 22.0, 21.1, 18.2, 16.1, -5.8, -5.8; HRMS (EI+) m/z calcd for $C_{23}H_{42}O_2PSSi$ ([M+H]$^+$) 441.2412, found 441.2394; $[\alpha]_D^{25}$ = -16.8° (chloroform).

(R_p)-Menthyl (acetoxymethyl)phenylthiophosphinate 28b:



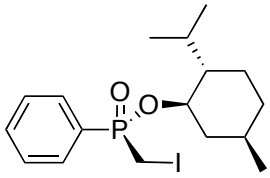
To a solution of *(S_p)-25* (1.73 g, 5 mmol, 1 equiv, >99% de) in toluene (10 mL) was added Lawesson's reagent (1.21 g, 3 mmol, 0.6 equiv). The reaction mixture was stirred for 16 hours at reflux under N_2. After cooling down the reaction to rt, the solvent was concentrated under vacuum and the residue obtained was purified by column chromatography (Hexane/ethyl acetate 99:1 to 97:3) to afford the product as a colorless oil (1.85 g, 100%, > 99% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 78.9 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.92-7.99 (m, 2H), 7.55-7.61 (m, 1H), 7.47-7.53 (m, 2H), 4.48-4.61 (m, 3H), 2.19 (dquint., *J* = 2.7 and 7.0 Hz, 1H), 2.04 (s, 3H), 1.61-1.80 (m, 3H), 1.46-1.55 (m, 1H), 1.33-1.45 (m, 1H), 1.08 (dq, *J* = 3.4 and 13.3 Hz, 1H), 0.98 (q, *J* = 11.8 Hz, 1H), 0.98 (d, *J* = 7.0 Hz, 3H), 0.91 (d, *J* = 7.0 Hz, 3H), 0.85 (dq, *J* = 3.3 and 11.9 Hz, 1H), 0.78 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 169.8 (d, J_{PCOC} = 7.6 Hz), 132.9 (d, J_{PC} = 104 Hz), 132.4 (d, J_{PCCCC} = 2.8 Hz), 131.6 (d, J_{PCCC} = 11.0 Hz, 2C), 128.3 (d, J_{PCC} = 13.1 Hz, 2C), 77.9 (d, J_{POC} = 7.5 Hz), 65.6 (d, J_{PC} = 98.2 Hz), 48.4 (d, J_{POCC} = 7.3 Hz), 43.2, 34.1, 31.4, 25.6, 23.0, 21.9, 21.0, 20.5, 16.0.

(R_p)-Menthyl (hydroxymethyl)phenylthiophosphinate 29:



To a solution of *(R_p)-28b* (1.84 g, 5 mmol, 1 equiv, >99% de) in methanol (10 mL) was added potassium carbonate (69 mg, 0.5 mmol, 0.1 equiv.) and the mixture was stirred for 16 h at rt. The solvent was removed under vacuum and then the residue was solubilized in ethyl acetate. The organic layer was washed with saturated $NaHCO_3$ (150 mL) and brine (150 mL), dried over $MgSO_4$, filtered and concentrated under vacuum to afford the product as a white solid (1.63 g, 100%, 96% de). Mp = 67-68 °C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 83.6 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.87-7.97 (m, 2H), 7.51-7.58 (m, 1H), 7.43-7.50 (m, 2H), 4.47 (dd, *J* = 4.4 and 10.6 Hz, 1H), 3.84-4.03 (m, 2H), 2.75 (s, 1H), 2.17 (dquint., *J* = 2.6 and 7.0 Hz, 1H), 1.58-1.77 (m, 3H), 1.43-1.52 (m, 1H), 1.29-1.41 (m, 1H), 1.05 (dq, *J* = 3.0 and 12.9 Hz, 1H), 0.76-1.01 (m, 2H), 0.95 (d, *J* = 7.0 Hz, 3H), 0.88 (d, *J* = 6.9 Hz, 3H), 0.75 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 133.0 (d, J_{PC} = 98.4 Hz), 132.4 (d, J_{PCCCC} = 3.0 Hz), 131.4 (d, J_{PCCC} = 10.6 Hz, 2C), 128.4 (d, J_{PCC} = 12.9 Hz, 2C), 77.9 (d, J_{POC} = 8.3 Hz), 65.2 (d, J_{PC} = 88.0 Hz), 48.5 (d, J_{POCC} = 7.1 Hz), 43.2, 34.1, 31.4, 25.6, 23.0, 22.0, 21.0, 16.0; HRMS (EI+) m/z calcd for $C_{17}H_{27}O_2PS$ ($[M]^+$) 326.1469, found 326.1466; $[\alpha]_D^{25}$ = -24.9° (chloroform).

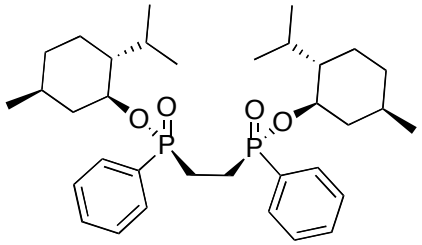
(S_p)-Menthyl (iodomethyl)phenylphosphinate 30:[4]



To a solution of *(S_p)-26* (4.78 g, 12.5 mmol, 1 equiv, >99% de) in acetone (40 ml) was added sodium iodide (7.5 g, 50 mmol, 4 equiv). The reaction mixture was stirred for 24h at reflux. The solvent was removed under vacuum and the residue obtained was dissolved in dichloromethane. The organic layer was washed with brine, dried over $MgSO_4$, filtered and concentrated under vacuum. The solid obtained was purified by column chromatography (dichloromethane/ethyl acetate 10:0 to 9:1) to

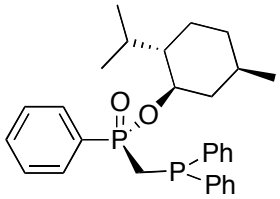
afford the product as a yellow solid (4.06 g, 94%, 96% de). Mp = 66-68^{0}C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 31.9 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.59-7.68 (m, 2H), 7.30-7.36 (m, 1H), 7.20-7.28 (m, 2H), 4.11-4.23 (m, 1H), 3.01 (dd, *J* = 8.9 and 12.7 Hz, 1H), 2.88 (dd, *J* = 5.9 and 12.7 Hz, 1H), 2.12-2.24 (m, 1H), 1.58-1.67 (m, 1H), 1.35-1.51 (m, 2H), 1.18-1.28 (m, 1H), 1.03-1.17 (m, 1H), 0.54-0.86 (m, 3H), 0.75 (d, *J* = 7.0 Hz, 3H), 0.69 (d, *J* = 6.9 Hz, 3H), 0.52 (d, *J* = 6.6 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 132.5 (d, J_{PCCCC} = 2.7 Hz), 131.8 (d, J_{PCCC} = 9.8 Hz, 2C), 130.2 (d, J_{PC} = 136 Hz), 128.3 (d, J_{PCC} = 13.1 Hz, 2C), 77.9 (d, J_{POC} = 7.2 Hz), 48.6 (d, J_{POCC} = 6.2 Hz), 42.9, 33.8, 31.3, 25.5, 22.7, 21.8, 21.0, 15.8, -6.5 (d, J_{PC} = 102 Hz); HRMS (EI+) m/z calcd for $C_{17}H_{27}IO_2P$ ([M+H]$^+$) 421.0793, found 421.0793; $[\alpha]_D$ = -29.5^0.

(R_p, R_p)-Ethane-1,2-diylbis(menthyl phenylphosphinate 31:[4]



To a solution of *(S_p)-30* (420.3 mg, 1 mmol, 1 equiv, 96% de) in THF (8 mL) at -78^{0}C under N_2 was slowly added isopropylmagnesium chloride (0.55 mL, 1.1 mmol, 1.1 equiv, 2.0M in THF). After 1 h of stirring at -78^{0}C, $CuCl_2$ (403 mg, 3 mmol, 3 equiv) was added. The dry ice-bath was removed and the reaction mixture was stirred for 2 h at rt. A saturated solution of NH_4Cl was added and the two layers were separated. The aqueous layer was extracted with dichloromethane (3X). The combined organic layers was dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (hexane/ethyl acetate 8:2 to 6:4) to afford the product as a white solid (237 mg, 81%, de = 89%). Mp = 78-79^{0}C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 39.7 (m); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.77-7.87 (m, 4H), 7.44-7.59 (m, 6H), 4.23-4.36 (m, 2H), 2.21 (dquint., *J* = 2.3 and 7.0 Hz, 2H), 1.78-1.87 (m, 2H), 1.66 (d, *J* = 14.4 Hz, 4H), 1.56-1.72 (m, 4H), 1.25-1.44 (m, 4H), 0.74-1.11 (m, 6H), 0.97 (d, *J* = 7.1 Hz, 6H), 0.90 (d, *J* = 7.0 Hz, 6H), 0.78 (d, *J* = 6.4 Hz, 6H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 133.8 (d, J_{PC} = 129 Hz, 2C), 131.8 (d, J_{PCCCC} = 2.7 Hz, 2C), 130.7 (d, J_{PCCC} = 9.9 Hz, 4C), 128.3 (d, J_{PCC} = 12.7 Hz, 4C), 76.2 (d, J_{POC} = 7.2 Hz, 2C), 48.6 (d, J_{POCC} = 6.1 Hz, 4C), 43.1 (2C), 34.0 (2C), 31.3 (2C), 25.7 (2C), 21.9 (d, J_{PC} = 138 Hz, 2C), 21.8 (2C), 17.1 (2C), 15.7 (2C); $[\alpha]_D^{25}$ = -17.1^o (chloroform).

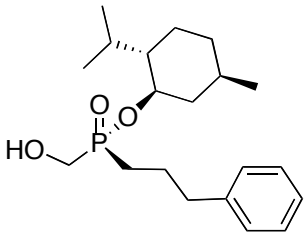
(S_p)-Menthyl [(diphenylphosphino)methyl]phenylphosphinate 32:



To a solution of *(S_p)-30* (676 mg, 2 mmol, 1 equiv, 96% de) in tetrahydrofuran (10 mL) was added at -78^{o}C and under N_2 isopropylmagnesium chloride (1.1 mL, 2.2 mmol, 1.1 equiv, 2.0M solution). After 2 hours at -78^{o}C, chlorodiphenylphosphine (0.37 mL, 2 mmol, 1 equiv) was added. The dry-ice bath was removed and the reaction was allowed to warm up to rt and was stirred for 4 hours at rt. A saturated solution of NH_4Cl was added and the two layers were separated. The aqueous layer was extracted with dichloromethane (3X). The combined organic layers was dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 100:0 to 98:2) to afford the product as a white solid (371 g, 39%, 96% de). Mp = 54-55^{o}C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 32.2 (d, *J* = 9.7 Hz, 46%), 11.9 (s, 54%); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.84-7.92 (m, 2H), 7.50-7.59 (m, 4H), 7.39-7.49 (m, 4H), 7.32-7.38 (m, 1H), 7.21-7.31 (m, 4H), 4.09-4.20 (m, 1H), 3.03 (dd, *J* = 2.1 and 11.1 Hz, 1H), 2.99 (d, *J* = 11.1 Hz, 1H), 2.07 (dquint., *J* = 2.1 and 7.0 Hz, 1H), 1.51-1.65 (m, 3H), 1.12-1.29 (m, 2H), 0.69-1.50 (m, 3H), 0.94 (q, *J* = 11.0 Hz, 1H), 0.69-0.97 (m, 2H), 0.87 (d, *J* = 7.0 Hz, 3H), 0.80 (d, *J* = 6.9 Hz, 3H), 0.70 (d, *J* = 6.5 Hz, 3H);

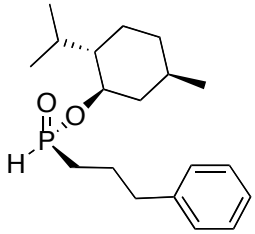
^{13}C NMR (101 MHz, $CDCl_3$): δ = 132.8 (d, J_{PCCC} = 9.9 Hz, 2C), 132.7 (d, J_{PCCC} = 9.6 Hz, 2C), 132.2 (d, J_{PC} = 134 Hz), 132.1 (d, J_{PCCCC} = 2.8 Hz), 131.5 (d, J_{PCCC} = 10.5 Hz, 2C), 131.4 (d, J_{PCCCC} = 2.3 Hz), 131.2 (d, J_{PCCCC} = 2.4 Hz), 128.7 (d, J_{PCC} = 10.7 Hz, 2C), 128.5 (d, J_{PC} = 134 Hz), 128.5 (d, J_{PC} = 135 Hz), 128.4 (d, J_{PCC} = 10.3 Hz, 2C), 128.2 (d, J_{PCC} = 13.3 Hz, 2C), 77.7 (d, J_{POC} = 7.4 Hz), 48.5 (d, J_{POCC} = 5.3 Hz), 42.9, 33.9, 31.4, 28.8 (dd, J_{PC} = 92.6 Hz), 25.5, 22.7, 21.9, 21.2, 15.6; HRMS (EI+) m/z calcd for $C_{29}H_{36}O_2P_2$ ($[M]^+$) 478.2191, found 478.2190; $[\alpha]_D^{24}$ = -0.9° (chloroform).

(R_p)-Menthyl (hydroxymethyl) (3-phenylpropyl)phosphinate 33:



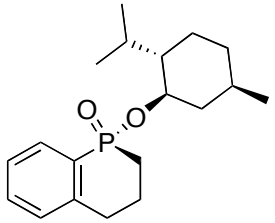
To a suspension of Pd/C (191 mg, 0.18 mmol, 10 mol%) in ethanol (2 mL) flushed with N_2 was added a solution of *(R_p)-4* (630 g, 1.8 mmol, 1 equiv, >99% de) in ethanol (8 mL). The tube was placed in a hydrogenator and stirred for 20 hours at 50 psi of H_2. The suspension was then filtered through celite and the solid was washed with ethanol three times. The filtrate was concentrated under vacuum to afford the product as a white solid (633 g, 100%, 98% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 52.5 (septuplet, *J* = 6.5 Hz); ^1H NMR (400 MHz, $CDCl_3$): δ = 7.24-7.31 (m, 2H), 7.13-7.21 (m, 3H), 4.12-4.22 (m, 1H), 3.83 (dd, *J* = 6.1 and 14.5 Hz, 1H), 3.75 (dd, *J* = 1.9 and 14.5 Hz, 1H), 2.69 (t, *J* = 7.4 Hz, 2H), 2.01-2.12 (m, 2H), 1.88-1.99 (m, 2H), 1.74-1.84 (m, 2H), 1.61-1.69 (m, 2H), 1.37-1.51 (m, 1H), 1.25-1.34 (m, 1H), 1.16 (q, *J* = 11.3 Hz, 1H), 0.98 (dquint., *J* = 2.5 and 12.8 Hz, 1H), 0.90 (d, *J* = 6.4 Hz, 3H), 0.90 (d, *J* = 7.2 Hz, 3H), 0.69-0.90 (m, 1H), 0.77 (d, *J* = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 141.0, 128.5 (2C), 128.4 (2C), 126.1, 76.3 (d, J_{POC} = 7.7 Hz), 59.8 (d, J_{PC} = 105 Hz), 48.6 (d, J_{POCC} = 5.6 Hz), 43.6, 36.8 (d, J_{PCC} = 15.7 Hz), 34.0, 31.5, 25.9 (d, J_{PC} = 90.2 Hz), 25.6, 23.3 (d, J_{PCCC} = 3.5 Hz), 22.7, 22.0, 21.0, 15.6.

(S_p)-Menthyl (3-phenylpropyl)-H-phosphinate 19:



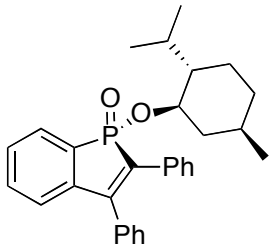
To a solution of *N*-chlorosuccinimide (721 mg, 5.4 mmol, 3 equiv) in dichloromethane (30 mL) at -78°C and under N_2 was added dropwise a solution of dimethyl sulfide (0.4 mL, 5.4 mmol, 3 equiv) in dichloromethane (5 mL). After 10 minutes at -78°C, a solution of *(R_p)-33* (630 mg, 1.8 mmol, 1 equiv, 98% de) in dichloromethane (5 mL) was added over 20 minutes. After 1h at -78°C, triethylamine (1.25 mL, 9 mmol, 5 equiv) was added over 15 minutes and the reaction was allowed to warm up to rt. After 1h at rt, water was added and the two layers were separated. The aqueous layer was extracted with dichloromethane (X2). The combined organic layer was dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 99:1 to 97:3) to afford the product as a colorless oil (554 mg, 96%, 96% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 33.3 (dsextuplet, *J* = 12.7 and 528 Hz); ^1H NMR (400 MHz, $CDCl_3$): δ = 7.16-7.23 (m, 2H), 7.05-7.13 (m, 3H), 7.08 (d, *J* = 528 Hz, 1H), 4.07-4.19 (m, 1H), 2.62 (t, *J* = 7.4 Hz, 2H), 1.99-2.10 (m, 2H), 1.77-1.91 (m, 2H), 1.53-1.73 (m, 4H), 1.21-1.43 (m, 2H), 1.02 (q, *J* = 11.6 Hz, 1H), 0.94 (dquint., *J* = 3.0 and 12.4 Hz, 1H), 0.84 (d, *J* = 6.8 Hz, 6H), 0.69-0.85 (m, 1H), 0.74 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 140.7, 128.4 (2C), 128.4 (2C), 126.1, 76.7 (d, J_{POC} = 7.4 Hz), 48.3 (d, J_{POCC} = 5.9 Hz), 41.6, 36.2 (d, J_{PCC} = 16.0 Hz), 33.9, 31.3, 28.1 (d, J_{PC} = 95.0 Hz), 25.6, 22.9, 22.4 (d, J_{PCCC} = 2.5 Hz), 21.9, 20.8, 15.7; $[\alpha]_D^{23}$ = -27.3° (chloroform).

(R_p)-1-Menthyloxy-1,2,3,4-tetrahydro-1-phosphinoline-1-oxide 34:



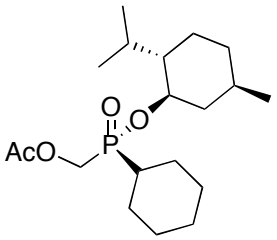
To a suspension of $Mn(OAc)_2$ (8.7 mg, 0.05 mmol, 5 mol%), MnO_2 (261 mg, 3 mmol, 3 equiv.) and sodium acetate (246 mg, 3 mmol, 3 equiv.) in acetic acid (2.5 mL) at 70^0C under N_2 was added a solution of *(S_p)-19* (322 mg, 1 mmol, 1 equiv, 96% de) in acetic acid (2.5 mL) over 2 h *via* a syringe pump. The reaction mixture was then stirred for an additional 2h at 70^0C under N_2. Ethyl acetate (~ 30 mL) and an aqueous solution of $Na_2S_2O_4$ 0.2M saturated with $NaHCO_3$ (~40 mL) were added. The suspension was stirred vigorously for 5 minutes, filtered through celite and the two layers were separated. The organic layer was washed with an aqueous solution of $Na_2S_2O_4$ 0.2M saturated with $NaHCO_3$ (~40 mL), a saturated aqueous solution of $NaHCO_3$ (~ 40 mL) and brine (~ 40 mL), dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 100:0 to 95:5) to afford the product as a colorless oil (300 mg, 94%, 96% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 36.3 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.71-7.79 (m, 1H), 7.28-7.34 (m, 1H), 7.20-7.27 (m, 1H), 7.05-7.12 (m, 1H), 4.17-4.28 (m, 1H), 2.33-2.47 (m, 2H), 1.54-1.79 (m, 6H), 1.53-1.62 (m, 2H), 1.29-1.42 (m, 1H), 1.18-1.28 (m, 1H), 1.02 (q, *J* = 10.9 Hz, 1H), 0.94 (dquint., *J* = 2.6 and 12.5 Hz, 1H), 0.83 (d, *J* = 7.0 Hz, 3H), 0.79 (d, *J* = 6.9 Hz, 3H), 0.79 (d, *J* = 6.6 Hz, 3H), 0.75 (dquint., *J* = 3.2 and 12.8 Hz, 1H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 143.7 (d, J_{PCC} = 9.9 Hz), 131.6 (d, J_{PCCCC} = 2.4 Hz), 129.8 (d, J_{PCCC} = 5.1 Hz), 129.8 (d, J_{PC} = 123 Hz), 128.9 (d, J_{PCC} = 11.5 Hz), 126.5 (d, J_{PCCC} = 11.5 Hz), 76.4 (d, J_{POC} = 7.2 Hz), 48.6 (d, J_{POCC} = 5.9 Hz), 43.6, 34.0, 31.5, 31.3 (d, J_{PCC} = 8.5 Hz), 26.5 (d, J_{PC} = 92.8 Hz), 25.6, 22.7, 22.0, 21.2 (d, J_{PCCC} = 5.1 Hz), 21.1, 15.6; HRMS (EI+) m/z calcd for $C_{19}H_{29}O_2P$ ([M]$^+$) 320.1905, found 320.1907; $[\alpha]_D^{22}$ = -8.6° (chloroform).

(S_p)-1-menthyl-2,3-diphenyl-1-phosphindole 35:[5]



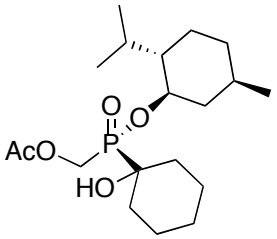
To a suspension of $Mn(OAc)_2$ (8.7 mg, 0.05 mmol, 5 mol%), MnO_2 (261 mg, 3 mmol, 3 equiv.), sodium acetate (246 mg, 3 mmol, 3 equiv.) and diphenylacetylene (178 mg, 1 mmol, 1 equiv.) in acetic acid (2.5 mL) at 70^0C under N_2 was added a solution of *(R_p)-1* (280 mg, 1 mmol, 1 equiv, >99% de) in acetic acid (2.5 mL) over 2 h *via* a syringe pump. The reaction mixture was then stirred for an additional 2h at 70^0C under N_2. Ethyl acetate (~ 30 mL) and an aqueous solution of $Na_2S_2O_4$ 0.2M saturated with $NaHCO_3$ (~40 mL) were added. The suspension was stirred vigorously for 5 minutes, filtered through celite and the two layers were separated. The organic layer was washed with an aqueous solution of $Na_2S_2O_4$ 0.2M saturated with $NaHCO_3$ (~40 mL), a saturated aqueous solution of $NaHCO_3$ (~ 40 mL) and brine (~ 40 mL), dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 100:0 to 98:2) to afford the product as a white solid (130 mg, 29%, > 99% de). Mp = 159-160°C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 44.8 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.69-7.79 (m, 1H), 7.05-7.56 (m, 13H), 4.29-4.42 (m, 1H), 2.25-2.38 (s, 1H), 1.53-1.76 (m, 3H), 1.38-1.51 (m, 1H), 1.16-1.36 (m, 2H), 0.75-1.10 (m, 2H), 0.92 (d, *J* = 6.4 Hz, 3H), 0.66 (d, *J* = 7.0 Hz, 3H), 0.43 (d, *J* = 6.8 Hz, 3H); HRMS (EI+) m/z calcd for $C_{30}H_{33}O_2P$ ([M]$^+$) 456.2218, found 456.2212; $[\alpha]_D^{25}$ = -52.9° (chloroform).

(R_p)-Menthyl (acetoxymethyl)cyclohexylphosphinate 36:



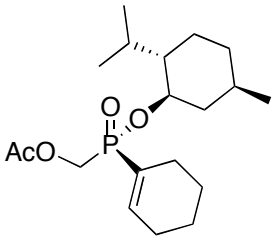
To a solution of *(R_p)-7* (2.76 g, 10 mmol, 1 equiv, 96% de) in acetonitrile (40 mL) in a sealed tube was added cyclohexene (2.03 mL, 20 mmol, 2 equiv) and AIBN (82 mg, 0.5 mmol, 5 mol%) and the reaction was stirred at reflux under N_2 for 2 hours. After cooling down the reaction to rt, AIBN (82 mg, 0.5 mmol, 5 mol%) was added and the reaction was stirred at reflux under N_2 for 2 hours. 2 additional addition of AIBN was made every 2 hours. 2 hours after the last addition, the reaction was cooled down to rt (81% NMR).

(S_p)-Menthyl (acetoxymethyl)(1-hydroxycyclohexyl)phosphinate 37:



To a solution of *(Sp)/(Rp)-7* (13.8 g, 50 mmol, 1 equiv, dr 54:46) in toluene (75 mL) was added at rt and under N_2 pyridine (0.4 mL, 5 mmol, 0.1 equiv) and cyclohexanone (10.3 mL, 100 mmol, 2 equiv). The reaction mixture was stirred for 3 days at reflux. After cooling down to rt, the solvent was removed under vacuum and the residue obtained was purified by column chromatography (dichloromethane/acetone 98:2 to 90:10) to afford the (R_p)/(S_p) mixture product as a white solid (15 g, 80%, 54:46 dr). This solid was recrystallized at -18°C in diethyl ether to afford the product as a white solid (5.1 g, 27%, 92% de). Mp = 125-126°C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 42.6 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 4.46-4.55 (m, 2H), 4.25-4.35 (m, 1H), 3.22 (s, 1H), 2.19 (dquint., *J* = 2.7 and 7.0 Hz, 1H), 2.10-2.16 (m, 1H), 2.12 (s, 3H), 1.91-1.98 (m, 1H), 1.77-1.85 (m, 1H), 1.64-1.74 (m, 7H), 1.54-1.63 (m, 3H), 1.41-1.52 (m, 1H), 1.31-1.40 (m, 1H), 1.16-1.29 (m, 1H), 1.14 (q, *J* = 11.2 Hz, 1H), 1.01 (dquint., *J* = 3.5 and 12.4 Hz, 1H), 0.92 (d, *J* = 7.0 Hz, 3H), 0.92 (d, *J* = 6.5 Hz, 3H), 0.78-0.92 (m, 1H), 0.81 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 170.2 (d, J_{PCOC} = 7.5 Hz), 77.5 (d, J_{POC} = 8.9 Hz), 72.3 (d, J_{PC} = 116 Hz), 57.2 (d, J_{PC} = 97.9 Hz), 48.8 (d, J_{POCC} = 5.3 Hz), 43.2, 34.0, 31.6, 30.8 (d, J_{PCCC} = 3.5 Hz), 30.2 (d, J_{PCCC} = 3.7 Hz), 25.4, 22.6, 22.0, 21.2, 20.7, 20.0 (d, J_{PCC} = 9.9 Hz), 19.9 (d, J_{PCC} = 10.1 Hz), 15.4; $[\alpha]_D^{24}$ = -22.4° (chloroform).

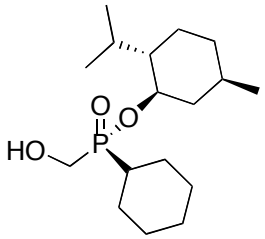
(R_p)-Menthyl (acetoxymethyl)(cyclohex-1-ene)phosphinate 38:



To a solution of *(S_p)-37* (374 mg, 1 mmol, 1 equiv, 92% de) in benzene (5 mL) was added at rt under N_2 thionyl chloride (0.087 mL, 1.2 mmol, 1.2 equiv.) followed by triethylamine (0.17 mL, 1.2 mmol, 1.2 equiv.). The mixture was then stirred for 16 h at reflux under N_2. After cooling down the reaction to rt, the solvent was removed under vacuum. The residue obtained was solubilized in ethyl acetate and the organic layer was washed with a saturated aqueous solution of $NaHCO_3$ and brine, dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column

chromatography (dichloromethane/acetone 99:1 to 95:5) to afford the product as a colorless oil (307 mg, 86%, 92% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 32.8 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.71 (d, *J* = 20.4 Hz, 1H), 4.03-4.31 (m, 3H), 1.98-2.16 (m, 5H), 1.99 (s, 3H), 1.92 (dquint., *J* = 2.5 and 7.0 Hz, 1H), 1.46-1.61 (m, 6H), 1.27-1.40 (m, 1H), 1.17-1.26 (m, 1H), 1.06 (q, *J* = 11.4 Hz, 1H), 0.88 (dq, *J* = 2.4 and 10.6 Hz, 1H), 0.79 (d, *J* = 6.4 Hz, 3H), 0.79 (d, *J* = 7.0 Hz, 3H), 0.67-0.79 (m, 1H), 0.64 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 170.0 (d, J_{PCOC} = 8.1 Hz), 144.3 (d, J_{PCC} = 7.9 Hz), 129.0 (d, J_{PC} = 127 Hz), 76.7 (d, J_{POC} = 7.3 Hz), 59.4 (d, J_{PC} = 115 Hz), 48.5 (d, J_{POCC} = 5.8 Hz), 43.5, 33.9, 31.5, 26.0 (d, J_{PCC} = 15.4 Hz), 25.5, 24.1 (d, J_{PCCC} = 9.8 Hz), 22.7, 21.9 (d, J_{PCCC} = 8.9 Hz), 21.9, 21.2, 21.0, 20.4, 15.6; HRMS (EI+) m/z calcd for $C_{19}H_{33}O_4P$ ([M]$^+$) 356.2116, found 356.2114; $[\alpha]_D^{23}$ = -28.7^0 (chloroform).

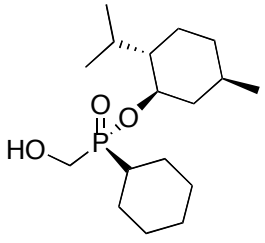
(R_p)-Menthyl (hydroxymethyl)cyclohexylphosphinate 39:



To a solution of *(R_p)-38* (356 mg, 1 mmol, 1 equiv, 92% de) in methanol (5 mL) was added potassium carbonate (14 mg, 0.1 mmol, 0.1 equiv.) and the mixture was stirred for 20 h at rt. The solvent was removed under vacuum and the crude obtained was purified by column chromatography (dichloromethane/acetone 95:5 to 85:15) to afford the product as a white solid (135 mg, 43%, 80% de). Mp = 80-81^{o}C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 38.7 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.74 (d, *J* = 20.4 Hz, 1H), 5.16 (s, 1H), 4.03-4.13 (m, 1H), 3.76-3.89 (m, 2H), 2.07-2.22 (m, 5H), 1.99 (dquint., *J* = 2.5 and 7.0 Hz, 1H), 1.52-1.71 (m, 6H), 1.32-1.46 (m, 1H), 1.19-1.30 (m, 1H), 1.10 (q, *J* = 11.2 Hz, 1H), 0.92 (dq, *J* = 2.5 and 12.8 Hz, 1H), 0.86 (d, *J* = 6.1 Hz, 3H), 0.84 (d, *J* = 6.6 Hz, 3H), 0.73-0.90 (m, 1H), 0.69 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 143.6 (d, J_{PCC} = 7.4 Hz), 129.0 (d, J_{PC} = 119 Hz), 76.4 (d, J_{POC} = 7.7 Hz), 58.8 (d, J_{PC} = 112 Hz), 48.6 (d, J_{POCC} = 5.5 Hz), 43.7, 34.0, 31.5, 26.1 (d, J_{PCC} = 14.7 Hz), 25.5, 24.2 (d, J_{PCCC} = 9.7 Hz), 22.7, 22.0, 22.0 (d, J_{PCCC} = 10.4 Hz), 21.1, 21.0, 15.7; HRMS (EI+) m/z calcd for $C_{17}H_{31}O_3P$ ([M]$^+$) 314.2011, found 314.2007; $[\alpha]_D^{24}$ = -29.9^o (chloroform).

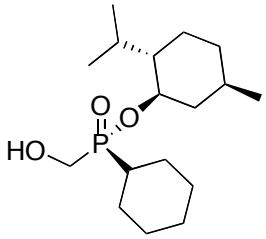
To a suspension of Pd/C (34 mg, 0.032 mmol, 10 mol%) in ethanol (1 mL) flushed with N_2 was added a solution of *(R_p)-menthyl (hydroxymethyl)(cyclohex-1-ene)phosphinate* (100 mg, 0.32 mmol, 1 equiv, 80% de) in ethanol (4 mL). The tube was placed in a hydrogenator and stirred for 20 hours at 50 psi of H_2. The suspension was then filtered through celite and the solid was washed with ethanol three times. The filtrate was concentrated under vacuum to afford the product as a white solid (102 mg, 100%, 80% de). Mp = 134-135^{o}C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 51.8 (s, 90%); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.06 (s, 1H), 4.10-4.23 (m, 1H), 3.76-3.89 (m, 2H), 2.04-2.22 (m, 2H), 1.74-2.03 (m, 5H), 1.69-1.71 (m, 3H), 1.16-1.48 (m, 7H), 1.07 (q, *J* = 11.4 Hz, 1H), 0.97 (dq, *J* = 2.5 and 12.8 Hz, 1H), 0.89 (d, *J* = 7.0 Hz, 3H), 0.88 (d, *J* = 6.4 Hz, 3H), 0.74-0.87 (m, 1H), 0.79 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 76.0 (d, J_{POC} = 8.0 Hz), 58.2 (d, J_{PC} = 99.1 Hz), 48.8 (d, J_{POCC} = 5.2 Hz), 43.5, 36.1 (d, J_{PC} = 92.3 Hz), 34.1, 31.5, 26.3 (d, J_{PCC} = 14.1 Hz), 26.3 (d, J_{PCC} = 13.9 Hz), 25.9, 25.4, 25.3 (d, J_{PCCC} = 3.3 Hz), 24.8 (d, J_{PCCC} = 2.9 Hz), 22.6, 22.1, 21.1, 15.5; HRMS (EI+) m/z calcd for $C_{17}H_{33}O_3P$ ([M]$^+$) 316.2167, found 316.2162; $[\alpha]_D^{23}$ = -32.1^o (chloroform).

(R_p)-Menthyl (hydroxymethyl)cyclohexylphosphinate 39:



To a solution of *(S_p)/(R_p)-7* (13.8 g, 50 mmol, 1 equiv, 54:46 dr) in toluene (75 mL) was added at rt and under N_2 pyridine (0.4 mL, 5 mmol, 0.1 equiv) and cyclohexanone (10.3 mL, 100 mmol, 2 equiv). The reaction mixture was stirred for 3 days at reflux. After cooling down to rt, the solvent was removed under vacuum and the residue obtained was purified by column chromatography (dichloromethane/acetone 98:2 to 90:10) to afford the (R_p)/(S_p) mixture as a white solid (15 g, 80%). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 43.9 (s, 46%), 42.8 (s, 54%).
To a solution of *(S_p)/(R_p)-37* (10.5 g, 28 mmol, 1 equiv, 54:46 dr) in benzene (80 mL) was added at rt under N_2 thionyl chloride (2.3 mL, 31 mmol, 1.1 equiv.) followed by triethylamine (4.3 mL, 31 mmol, 1.1 equiv.). The mixture was then stirred for 16 h at reflux under N_2. After cooling down the reaction, the solvent was removed under vacuum. The residue obtained was solubilized in ethyl acetate and the organic layer was washed with a saturated aqueous solution of $NaHCO_3$ and brine, dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 99:1 to 95:5) to afford the product as a colorless oil (8.57 g, 86%). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 37.9 (s, 42%), 37.2 (s, 58%).
To a solution of the crude obtained (8.5 g, 24 mmol, 1 equiv, 52:48 dr) in methanol (50 mL) was added potassium carbonate (331 mg, 2.4 mmol, 0.1 equiv.) and the mixture was stirred for 20 h at rt. The solvent was removed under vacuum and the crude obtained was purified by column chromatography (dichloromethane/acetone 80:20) to afford the product as a white solid (6.58 g, 87%). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 39.2 (s, 44%), 38.5 (s, 56%).
To a suspension of Pd/C (2.2 g, 2.07 mmol, 10 mol%) in ethanol (25 mL) flushed with N_2 was added a solution of the crude obtained (6.5 g, 20.7 mmol, 1 equiv) in ethanol (50 mL). The flask was placed in a hydrogenator and stirred for 4 days at 50 psi of H_2. The suspension was then filtered through celite and the solid was washed with ethanol three times. The filtrate was concentrated under vacuum to afford the product as a white solid (6.54 g, 100%). This solid was crystallized in ethyl acetate (200 mL) at rt to afford the product as a white solid (1.55 g, 24%, > 99% de, 14% overall yield). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 47.3 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 4.76-4.83 (m, 1H), 4.11-4.22 (m, 1H), 3.78-3.90 (m, 2H), 2.18-2.25 (m, 1H), 1.89-2.06 (m, 3H), 1.76-1.88 (m, 3H), 1.60-1.73 (m, 3H), 1.18-1.51 (m, 7H), 1.11 (q, *J* = 11.0 Hz, 1H), 0.98 (dq, *J* = 2.5 and 12.9 Hz, 1H), 0.90 (d, *J* = 7.0 Hz, 3H), 0.90 (d, *J* = 6.4 Hz, 3H), 0.77-0.90 (m, 1H), 0.79 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 76.5 (d, J_{POC} = 8.0 Hz), 57.5 (d, J_{PC} = 100 Hz), 48.7 (d, J_{POCC} = 5.9 Hz), 44.0, 36.3 (d, J_{PC} = 91.2 Hz), 34.1, 31.5, 26.2 (d, J_{PCC} = 13.6 Hz), 26.2 (d, J_{PCC} = 14.7 Hz), 25.9, 25.5, 25.3 (d, J_{PCCC} = 3.2 Hz), 24.7 (d, J_{PCCC} = 3.1 Hz), 22.7, 22.0, 21.2, 15.6; HRMS (EI+) m/z calcd for $C_{17}H_{33}O_3P$ ([M]$^+$) 316.2167, found 316.2162; $[\alpha]_D^{23}$ = -32.1° (chloroform).

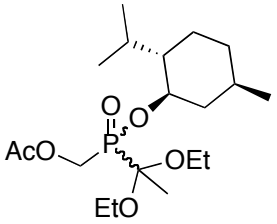
(R_p)-Menthyl (hydroxymethyl)cyclohexylphosphinate 39:



To a solution of *(R_p)/(S_p)-7* (8.28 g, 30 mmol, 1 equiv, 54:46 dr) in DMSO (150 mL) was added $Mn(OAc)_2$ (368 mg, 1.5 mmol, 5 mol%) and cyclohexene (15.2 mL, 150 mmol, 5 equiv.) and the reaction was stirred at 100°C under N_2 for 16 hours. After cooling down the reaction to rt, ethyl acetate (~ 150 mL) and an aqueous solution of $Na_2S_2O_4$ 0.2M saturated with $NaHCO_3$ (~150 mL) were added and the suspension was stirred vigorously for 5 minutes. The 2 layers were separated and the organic layer was washed with an aqueous solution of $Na_2S_2O_4$ 0.2M saturated with $NaHCO_3$ (~150 mL), a saturated aqueous solution of $NaHCO_3$ (~ 150 mL) and brine (~ 150 mL), dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 100:0 to 94:6) to afford the product as a colorless oil (5.37 g, 50%).
To a solution of the crude obtained (5.37 g, 15 mmol, 1 equiv, 54:46 dr) in methanol (50 mL) was added potassium carbonate (207 mg, 1.5 mmol, 0.1 equiv.) and the mixture was stirred for 20 h at rt.

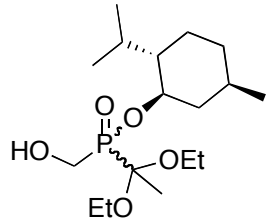
The solvent was removed under vacuum and the crude obtained was purified by column chromatography (dichloromethane/acetone 95:5 to 60:40) to afford the product as a white solid (4.31 g, 91%). This solid was crystallized in acetonitrile at rt to afford the product as a white solid (1.47g, 31%, > 99% de). Mp = 143-144 °C; ^{31}P NMR (162 MHz, $CDCl_3$): δ = 52.3 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 4.35-4.42 (m, 1H), 4.13-4.24 (m, 1H), 3.80-3.91 (m, 2H), 2.19-2.27 (m, 1H), 1.90-2.07 (m, 3H), 1.78-1.88 (m, 3H), 1.62-1.74 (m, 3H), 1.20-1.51 (m, 7H), 1.13 (q, *J* = 11.0 Hz, 1H), 0.99 (dq, *J* = 2.8 and 12.7 Hz, 1H), 0.78-0.95 (m, 1H), 0.92 (d, *J* = 7.1 Hz, 3H), 0.91(d, *J* = 6.4 Hz, 3H), 0.80 (d, *J* = 6.9 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 76.6 (d, J_{POC} = 8.0 Hz), 57.6 (d, J_{PC} = 99.3 Hz), 48.7 (d, J_{POCC} = 5.9 Hz), 44.0, 36.4 (d, J_{PC} = 91.5 Hz), 34.1, 31.5, 26.3 (d, J_{PCC} = 13.7 Hz, 2C), 25.9, 25.5, 25.3 (d, J_{PCCC} = 3.3 Hz), 24.8 (d, J_{PCCC} = 3.0 Hz), 22.8, 22.0, 21.2, 15.6; HRMS (EI+) m/z calcd for $C_{17}H_{33}O_3P$ ($[M]^+$) 316.2167, found 316.2162; $[\alpha]_D^{23}$ = -32.1° (chloroform).

(R_p)/(S_p) Menthyl acetoxymethyl(1,1-diethoxyethyl)phosphinate 40:



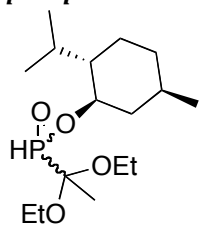
(R_p)/(S_p)-7 (4.95 g, 17.9 mmol, 1 equiv, 51:49 dr), triethyl orthoformate (19.7 mL, 107.5 mmol, 6 equiv) and boron trifluoride diethyl etherate (0.45 mL, 3.6 mmol, 0.2 equiv.) were introduced in a flask and the reaction mixture was stirred at rt under N_2 for 24 hours. Ethyl acetate (~ 150 mL) and an aqueous solution of $NaHCO_3$ (~150 mL) were added and the 2 layers were separated. The organic layer was washed with brine (~ 150 mL), dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (hexane/ethyl acetate 90:10 to 70:30) to afford the product as a colorless oil (5.17 g, 74%). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 35.9 (s, 52%) and 35.1 (s, 48%).

(R_p)/(S_p) Menthyl hydroxymethyl(1,1-diethoxyethyl)phosphinate 41:



To a solution of *40* (6.6 g, 17 mmol, 1 equiv) in methanol (30 mL) was added potassium carbonate (235 mg, 1.7 mmol, 0.1 equiv.) and the mixture was stirred for 20 h at rt. The solvent was removed under vacuum to afford the product as a colorless oil (5.95 g, 100%). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 42.0 (s, 53%) and 41.7 (s, 47%).

(R_p)/(S_p) Menthyl (1,1-diethoxyethyl)-H-phosphinate 43:

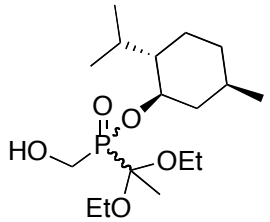


To a solution of concentrated H_3PO_2 (3.3 g, 50 mmol, 1 equiv) was added slowly, at room temperature under nitrogen, trifluoroacetic acid (1.14 mL, 10 mmol, 0.2 equiv) followed by triethyl orthoformate (20 mL, 110 mmol, 2.2 equiv.). After 4 hours of stirring at room temperature, chloroform was added (100 mL) as well as a saturated aqueous solution of $NaHCO_3$ (~100 mL). The 2

layers were separated and the organic layer was washed with brine (~ 100 mL), dried over $MgSO_4$, filtered and concentrated under vacuum to afford the product as a yellow oil (9.87 g, 84% purity). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 31.0 (d, *J* = 543 Hz, 84%), 17.4 (s, 3%) and 7.3 (d, 13%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.96 (d, *J* = 543 Hz, 1H), 4.09-4.32 (m, 2H), 3.63-3.82 (m, 4H), 1.50 (d, *J* = 12.6 Hz, 3H), 1.40 (t, *J* = 7.0 Hz, 3H), 1.24 (t, *J* = 7.1 Hz, 3H), 1.24 (t, *J* = 7.0 Hz, 3H).

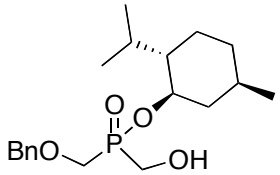
To a solution of *ethyl (1,1-diethoxyethyl)-H-phosphinate* (6 g, 28.6 mmol, 1 equiv, 84% purity) in toluene (40 mL) was added L-menthol (17.85 g, 114 mmol, 4 equiv) followed by $Ti(OiPr)_4$ (0.85 mL, 2.86 mmol, 10 mol%). The reaction mixture was stirred for 24 hours under N_2 at a slow reflux with a Dean-Stark trap to remove the etanol generated during the reaction. After cooling down the reaction to rt, the solvent was removed under vacuum and the residue was purified by column chromatography (dichloromethane/acetone 100:0 to 95:5) to afford the product a colorless oil (5.44 g, 72%). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 29.8 (s, 51%), 25.3 (s, 49%).

(R_p)/(S_p) Menthyl hydroxymethyl(1,1-diethoxyethyl)phosphinate 41:



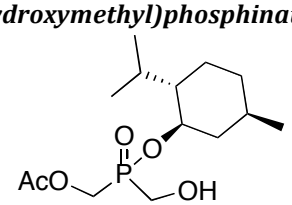
To a solution of *menthyl (1,1-diethoxyethyl)-H-phosphinate* (5.12 g, 16 mmol, 1 equiv, 51:49 dr) in toluene (30 mL) was added paraformaldehyde (0.53 g, 17.6 mmol, 1.1 equiv). The reaction mixture was stirred in a sealed tube at reflux for 20 hours under N_2. After cooling down the reaction to rt, the solvent was removed under vacuum and the residue was purified by column chromatography (dichloromethane/acetone 100:0 to 90:10) to afford the product a colorless oil (2.58 g, 46%). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 42.0 (s, 54%) and 41.7 (s, 46%).

(R_p)/(S_p) Menthyl benzoxymethyl (hydroxymethyl)phosphinate 44:



To a solution of *(R_p)/(S_p)-2* (4.68 g, 20 mmol, 1 equiv, 54:46 dr) in dichloromethane (30 mL) at 0°C and under N_2 was added bis(trimethylsilyl)acetamide (10 mL, 40 mmol, 2 equiv) followed by benzyl chloromethyl ether (5.6 mL, 40 mmol, 2 equiv). The ice-bath was removed and the reaction mixture was then stirred for 20 h at rt. Methanol was added (1.62 mL, 40 mmol, 2 equiv) and the reaction mixture was concentrated under vacuum. The residue obtained was dissolved in ethyl acetate and the organic layer was washed with a saturated aqueous solution of $NaHCO_3$ and brine. The organic layer was dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was purified by column chromatography (dichloromethane/acetone 10:0 to 9:1) to afford the product as white solid (5.27 g, 74%). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 41.8 (s, 40%), 41.7 (s, 60%).

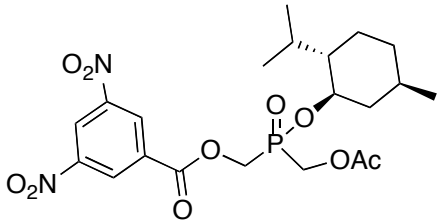
(R_p)/(S_p) Menthyl acetoxymethyl(hydroxymethyl)phosphinate 45:



Paraformaldehyde (4.95 g, 165 mmol, 1.1 equiv) and hypophosphorous acid (9.9 g, 150 mmol, 1 equiv, 50% in water) were introduced in a round bottom flask and the reaction mixture was stirred for 24h at 75°C. The reaction was cooled down to rt and the crude was diluted in toluene (150 mL). L-

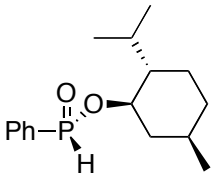
menthol (23.44 g, 150 mmol, 1 equiv) was added and the reaction mixture was stirred for 24h at reflux under N_2 in a flask equipped with a Dean-Stark trap. The solvent was then removed under vacuum and the residue obtained was dissolved in dichloromethane (300 mL). triethylamine (26 mL, 187.5 mmol, 1.25 equiv) and acetic anhydride (17.1 mL, 180 mmol, 1.2 equiv) was then added at 0°C under N_2. The ice-bath was removed and the reaction mixture was stirred for 16h at rt. The solvent was removed under vacuum and the residue obtained was solubilized in ethyl acetate. The organic layer was washed with $NaHCO_3$ and brine, dried over $MgSO_4$, filtered and concentrated under vacuum to afford the product as a white solid (38.7 g, 93%, 46:54 dr). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 25.9 (dm, *J* = 567 Hz, 46%), 21.9 (dm, *J* = 567 Hz, 54%).
To a solution of *menthyl (acetoxymethyl)-H-phosphinate* (38.7 g, 140 mmol, 1 equiv, 46:54 dr) in toluene (250 mL) was added paraformaldehyde (5.11 g, 170 mmol, 1.2 equiv) and the reaction was stirred at reflux under N_2 for 16 hours. The solvent was removed under vacuum to afford the product as a white solid (42.7 g, 100%, 52:48 dr). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 43.8 (s, 52%), 43.3 (s, 48%).

Menthyl [(3,5-dinitrobenzoyloxy)methyl](acetoxymethyl)phenylphosphinate 46:



To a solution of *(R_p)/(S_p)-45* (9.18 g, 30 mmol, 1 equiv, 54:46 dr) in dichloromethane (60 mL) was added at 0^0C under N_2 triethylamine (4.81 mL, 34.5 mmol, 1.15 equiv) followed by 3,5-dinitrobenzoyl chloride (7.61 g, 33 mmol, 1.1 equiv) in dichloromethane (20 mL). The reaction mixture was then stirred for 20 hours at rt. The reaction mixture was concentrated under vacuum. The residue was dissolved in ethyl acetate (~ 100 mL) and the organic layer was washed with an aqueous solution of $NaHCO_3$ (~ 100 mL) and brine (~ 100 mL), dried over $MgSO_4$, filtered and concentrated under vacuum. The crude obtained was crystallized in toluene (80 mL) to obtain the product as yellow needles (4.35 g, 29%, >99% de). Mp = 139-140^{0}C; ^{31}P NMR (121.47 MHz, $CDCl_3$): δ = 34.9 (s); ^1H NMR (400 MHz, $CDCl_3$): δ = 9.27 (s, 1H), 9.17 (s, 2H), 4.71-4.84 (m, 2H), 4.48-4.57 (m, 2H), 4.36-4.47 (m, 1H), 2.13-2.22 (m, 1H), 2.15 (s, 3H), 2.02-2.11 (m, 1H), 1.65-1.74 (m, 2H), 1.36-1.57 (m, 2H), 1.25 (d, *J* = 11.8 Hz, 1H), 1.04 (d, *J* = 13.2 Hz, 1H), 0.85-0.97 (m, 1H), 0.94 (d, *J* = 6.2 Hz, 3H), 0.90 (d, *J* = 6.3 Hz, 3H), 0.82 (d, *J* = 6.7 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$): δ = 169.9 (d, J_{PCOC} = 7.2 Hz), 162.0 (d, J_{PCOC} = 7.1 Hz), 148.8 (2C), 132.6, 129.6 (2C), 123.0, 79.1 (d, J_{POC} = 7.8 Hz), 60.0 (d, J_{PC} = 112 Hz), 59.0 (d, J_{PC} = 114 Hz), 48.4 (d, J_{POCC} = 5.9 Hz), 43.4, 33.8, 31.6, 25.9, 22.8, 21.9, 20.8, 20.5, 15.6; HRMS (EI+) m/z calcd for $C_{21}H_{30}N_2O_{10}P$ ($[M+H]^+$) 501.1638, found 501.1621; $[\alpha]_D^{25}$ = -18.2^o (chloroform).

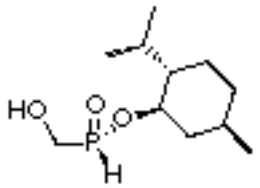
(R_p)-Menthyl phenyl-H-phosphinate 1:[1]



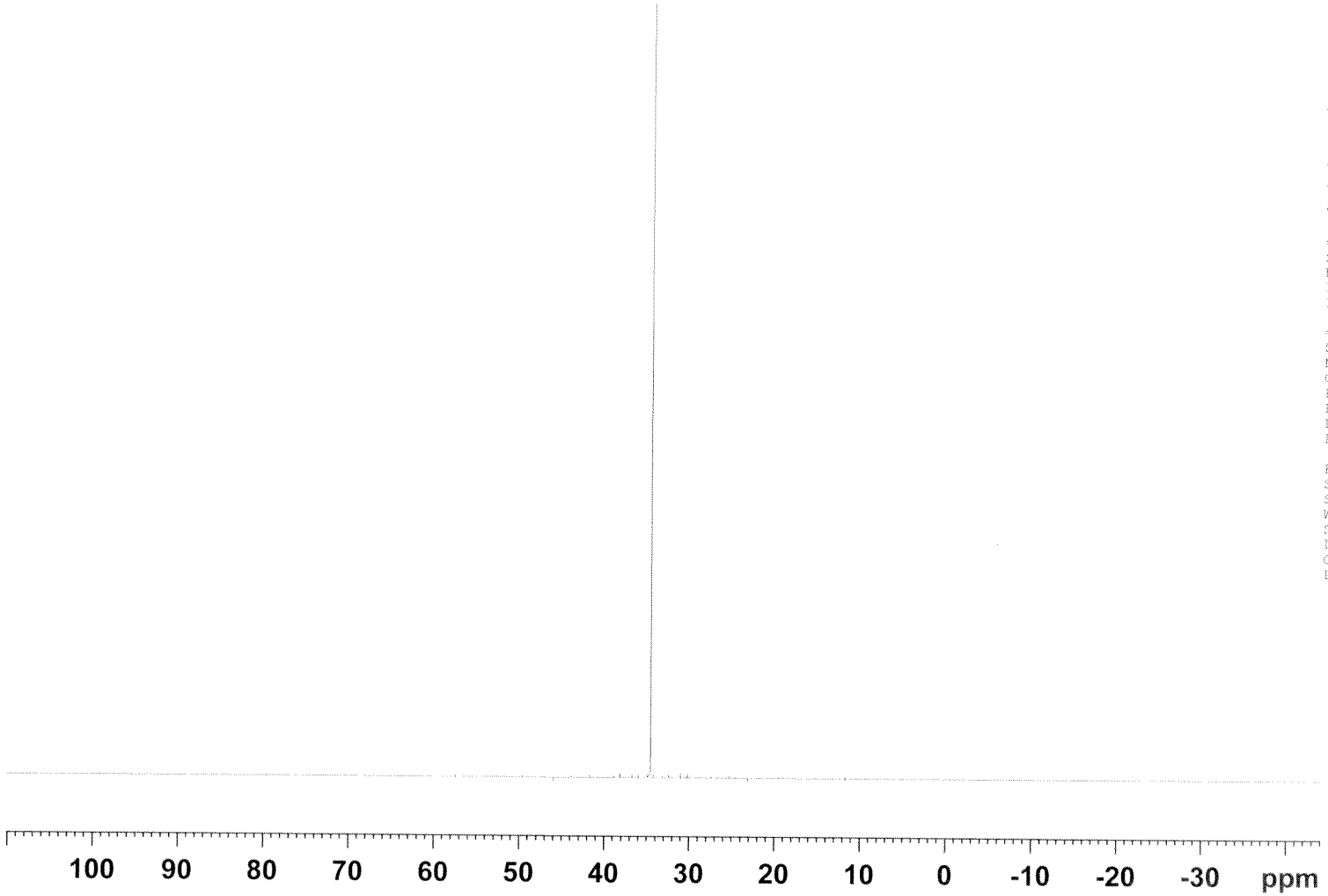
To a solution of *(R_p)/(S_p)-1* (8.4 g, 30 mmol, 1 equiv, 50:50 dr) in diethylether (200 mL) was slowly added at rt under N_2 phosphorus trichloride (3.14 mL, 36 mmol, 1.2 equiv) followed by pyridine (2.91 mL, 36 mmol, 1.2 equiv). After 2 hours at rt, the reaction was cooled down to -78^{o}C and then a mixture of diethylether – water (50 mL) was added over 20 minutes. After 4 hours at -78^{o}C, the reaction was allowed to warm up to rt. Brine was added and the 2 layers were separated. The organic layer was dried over magnesium sulfate, filtered and concentrated. The crude obtained was purified by column chromatography (hexane/ethyl acetate 7:3) to afford the product as a colorless oil (7.3 g, 65%, 63% de). The oil obtained was crystallized in petroleum ether (7.5 mL) at -30^{o}C to afford the product as a colorless oil (2.5 g, 22%, 96% de). ^{31}P NMR (162 MHz, $CDCl_3$): δ = 24.7 (dm, *J* = 553 Hz);

^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.73-7.84 (m, 2H), 7.66 (d, *J* = 553 Hz, 1H), 7.46-7.64 (m, 3H), 4.22-4.36 (m, 1H), 2.14-2.27 (m, 2H), 1.62-1.75 (m, 2H), 1.38-1.54 (m, 2H), 1.24 (q, *J* = 11.2 Hz, 1H), 0.78-1.13 (m, 2H), 0.96 (d, *J* = 7.0 Hz, 3H), 0.90 (d, *J* = 6.4 Hz, 3H), 0.86 (d, *J* = 7.0 Hz, 3H); $[\alpha]_D^{23}$ = -35.5° (chloroform, literature with 90% de: -21.0° in benzene).

[1] O. Berger, J.-L. Montchamp *Angew. Chem. Int. Ed.* **2013**, *52*, 11377-11380.
[2] O. Berger, J.-L. Montchamp *Chemistry - A European Journal* **2014**, *20*, 12385-12388.
[3] H. C. Fisher, O. Berger, F. Gelat, J.-L. Montchamp *Adv. Synth. Catal.* **2014**, *356*, 1199-1204.
[4] J.-L. Montchamp, O. Berger U.S. Pat. Appl. Publ. 2013, US 20130331594 A1 20131212.
[5] Y.-R. Chen, W.-L. Duan *J. Am. Chem. Soc.* **2013**, *135*, 16754-16757.



Compound (R_p)-2
$^{31}P\{^1H\}$ NMR decoupled

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PROCNO                 1

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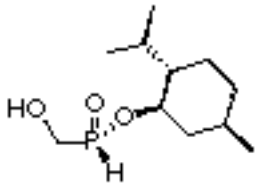

Compound (R_p)-2
$^{31}P\{^1H\}$ NMR coupled

BRUKER

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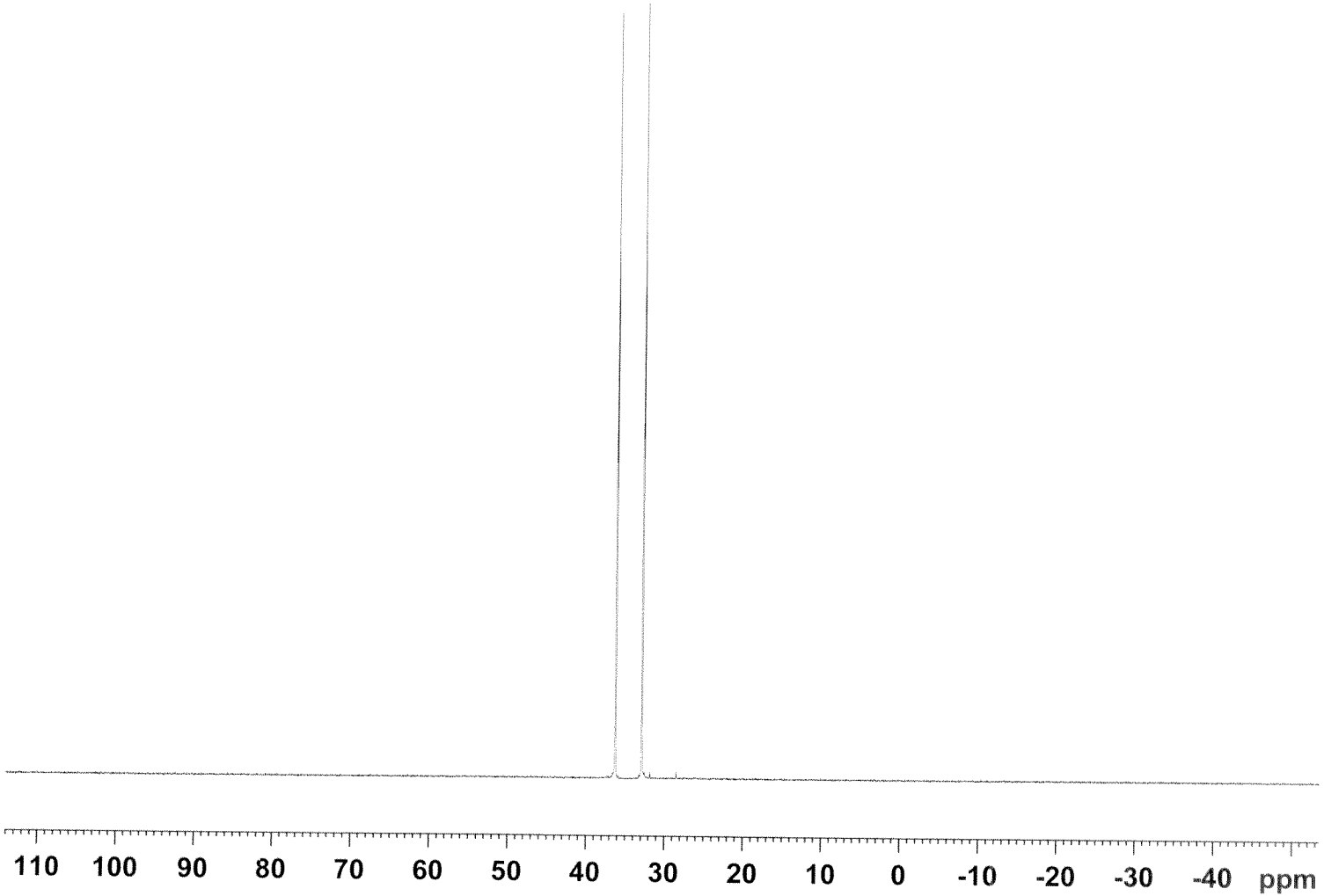
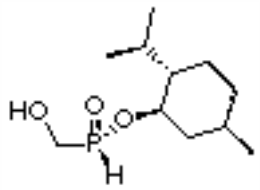
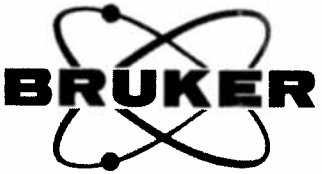




Compound (R_p)-2
^{1}H NMR

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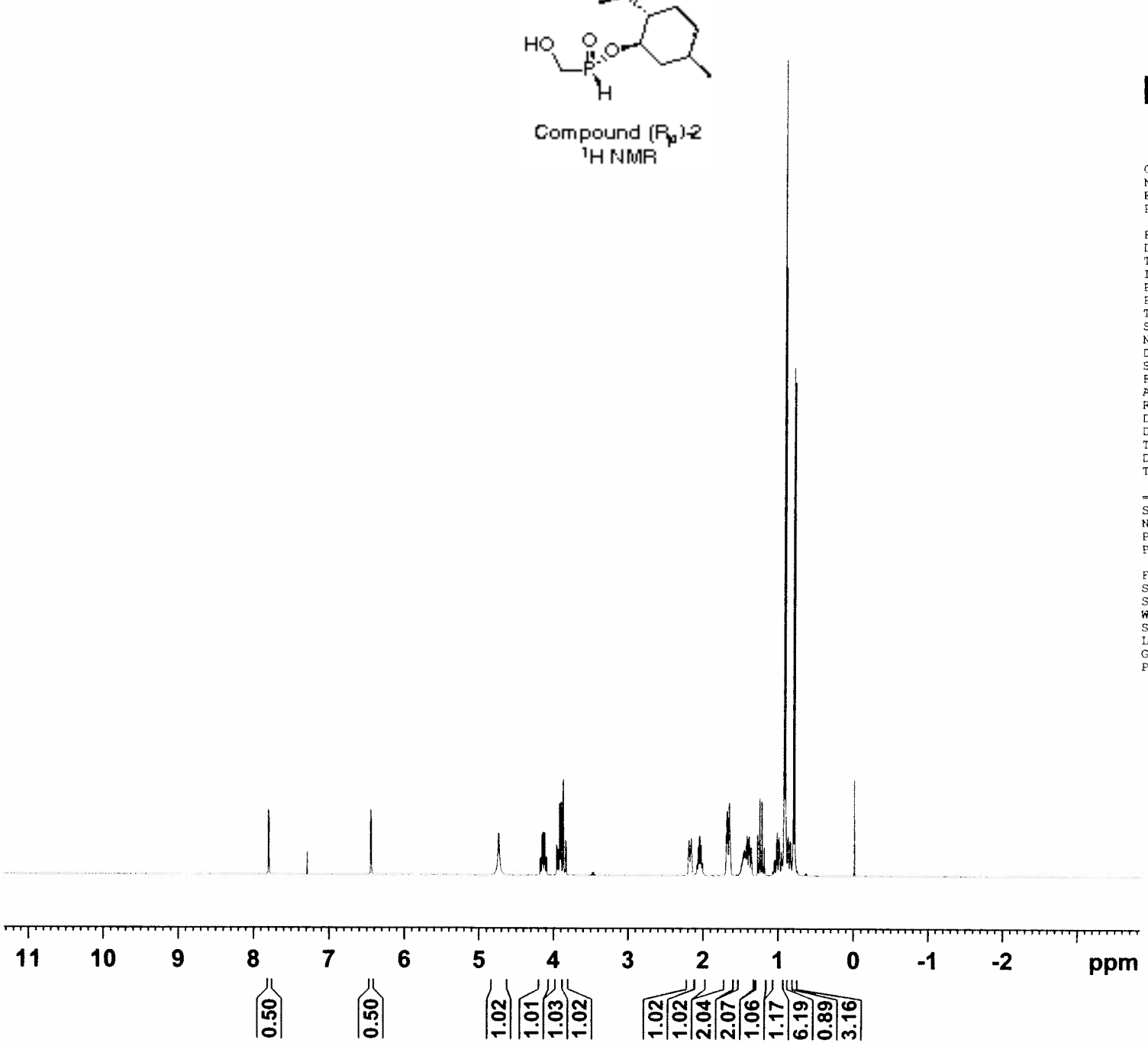
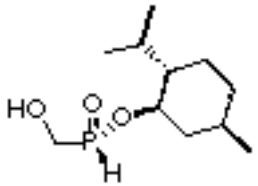
TD                65536
SOLVENT           CDCl3
NS                   11
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                18.98
DW               62.400 usec
DE                6.50 usec
TE               298.0 K
D1            1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                 1H
P1                10.00 usec
PLW1         25.00300026 W

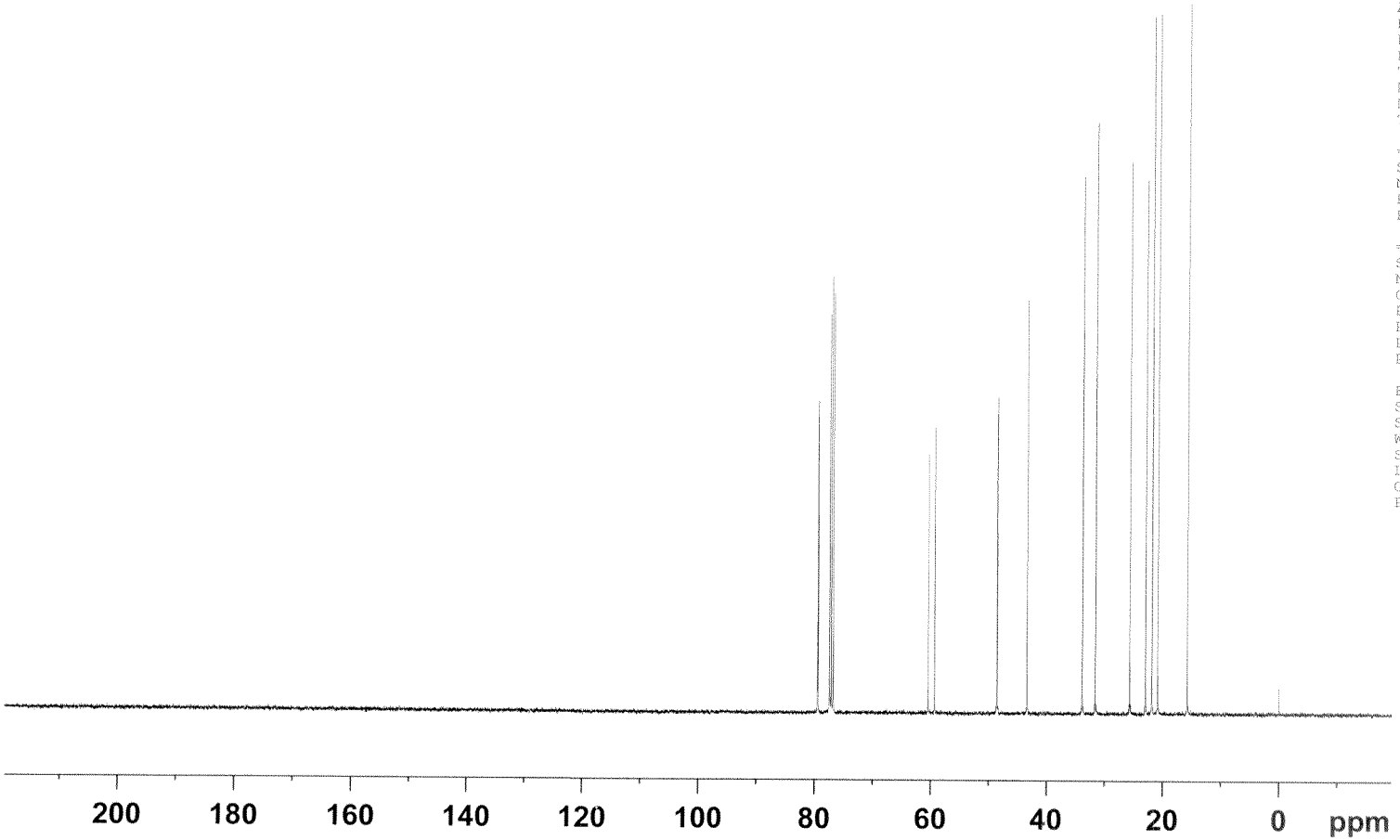
F2 - Processing parameters
SI                65536
SF           400.1300000 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```





Compound (R_p)-2

^{13}C NMR



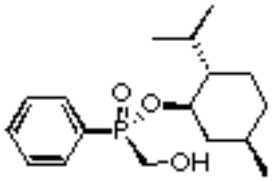

```
Current Data Parameters
NAME      OB 1883 after crystallization
EXPNO                 4
PROCNO                1

F2 - Acquisition Parameters
Date_          20150114
Time               10.46
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  545
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                299.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

========== CHANNEL f1 ==========
SFO1         100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1         45.00000000 W

========== CHANNEL f2 ==========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



Compound (S_p)-3
$^{31}P\{^1H\}$ NMR decoupled

BRUKER

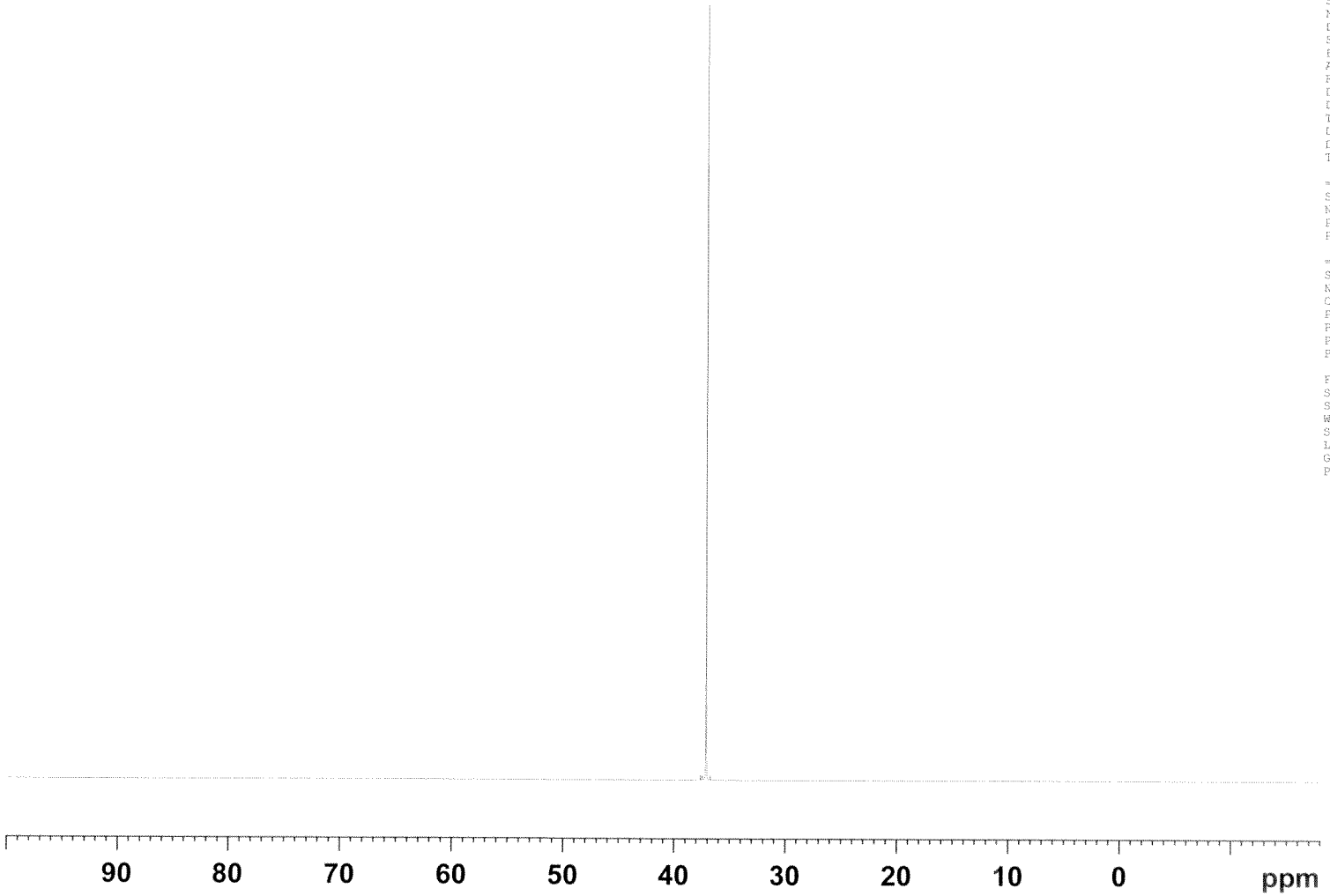
```
Current Data Parameters
NAME      OB 1888 2nd crystallization in Et2O
EXPNO                  1
PROCNO                 1

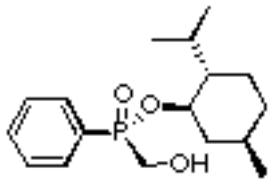
F2 - Acquisition Parameters
Date_           20150212
Time               12.16
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                 7.800 usec
DE                 6.50 usec
TE                297.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1         15.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                 32768
SF           161.9755930 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```





Compound (S_p)-3
$^{31}P\{^1H\}$ NMR coupled

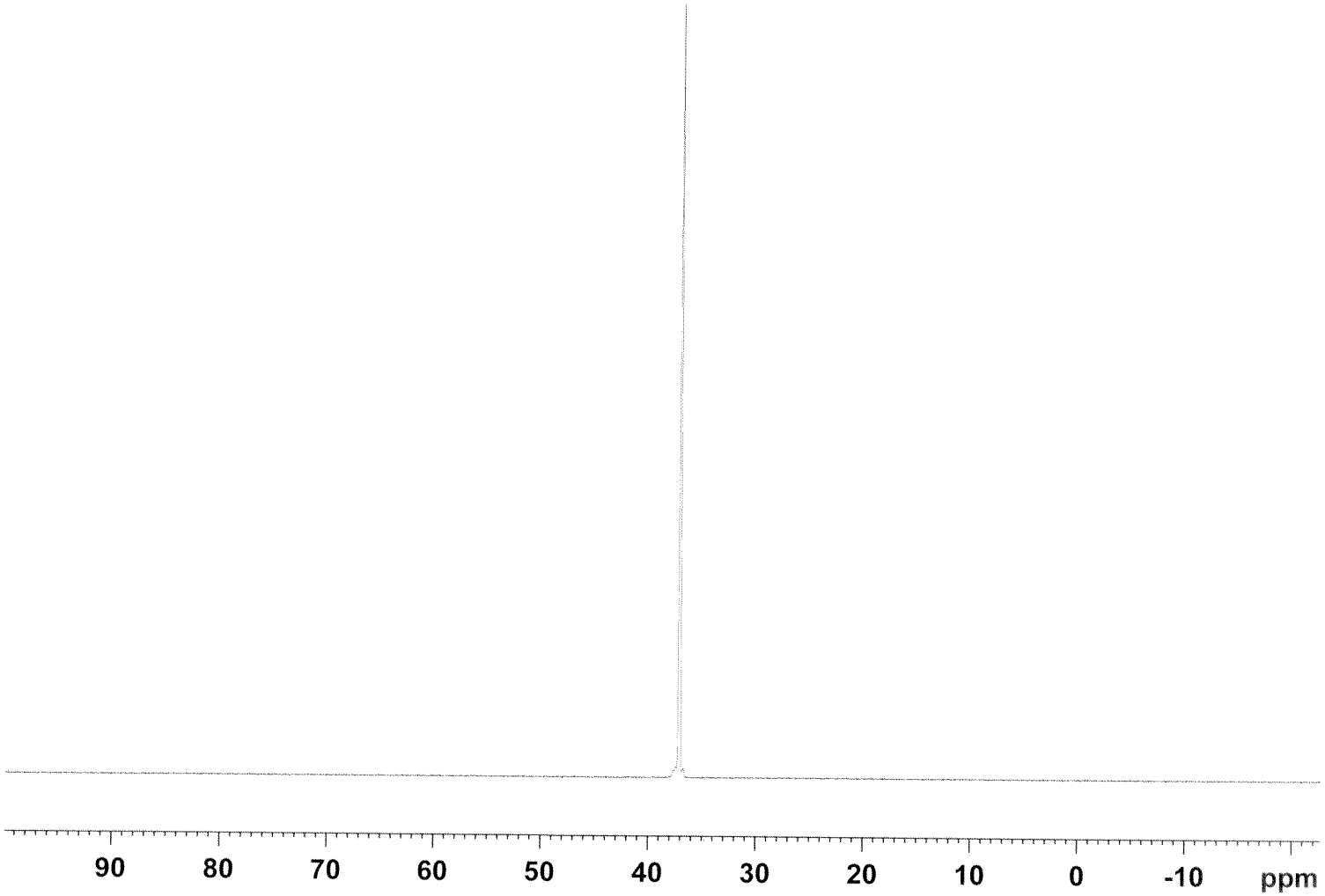
BRUKER

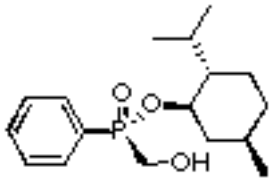
```
Current Data Parameters
NAME      OB 1888 2nd crystallization in Et2O
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150212
Time                12.17
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH             64102.563 Hz
FIDRES          0.978127 Hz
AQ              0.5111808 sec
RG                 203.57
DW                  7.800 usec
DE                  6.50 usec
TE                297.1 K
D1             2.00000000 sec
TD0                    1

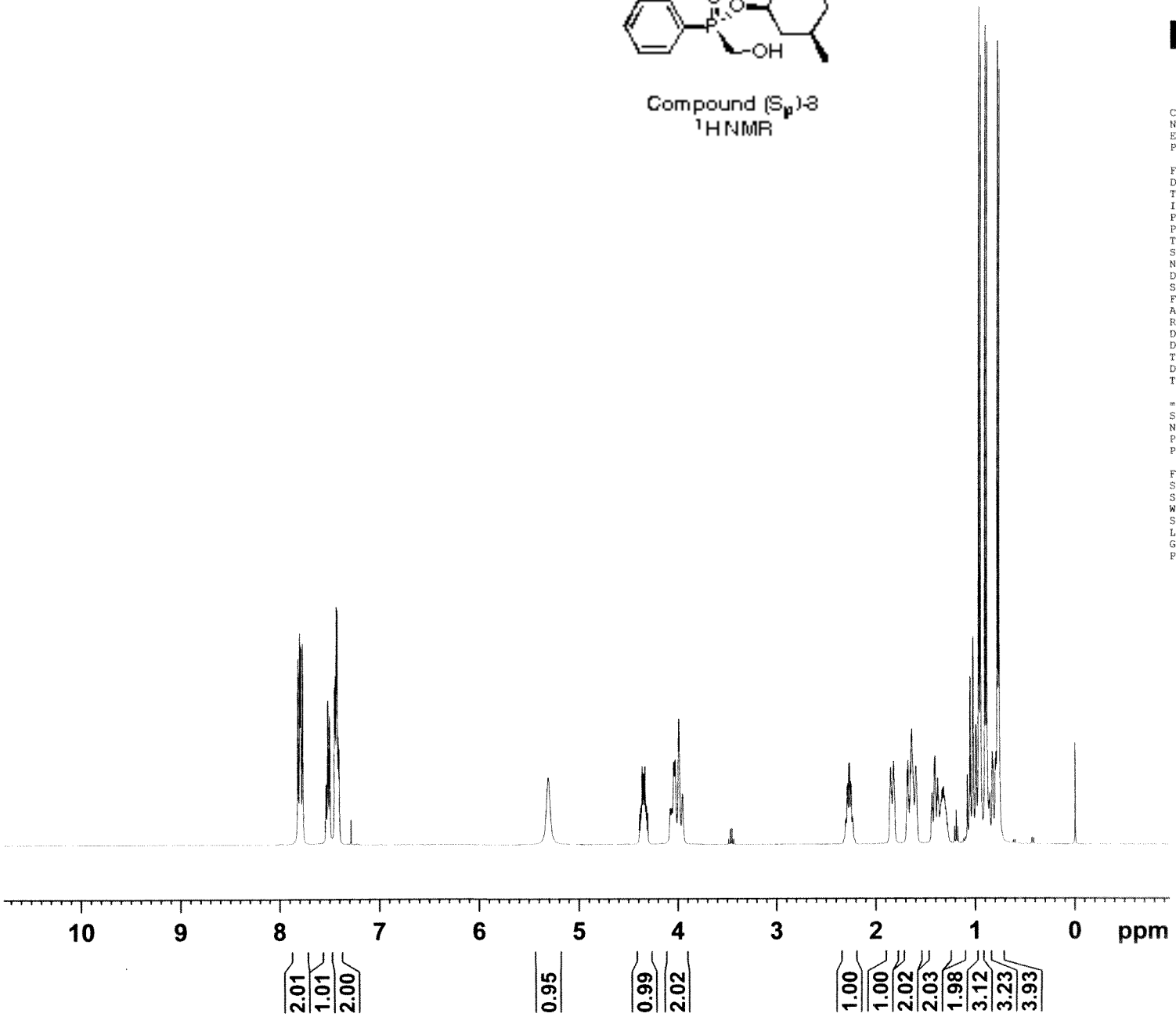
======== CHANNEL f1 ========
SFO1          161.9674942 MHz
NUC1                  31P
P1                  14.25 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                 32768
SF            161.9755930 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```





Compound (S_p)-3
^{1}H NMR

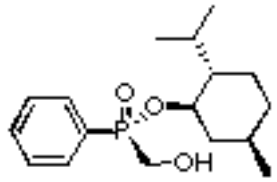



```
Current Data Parameters
NAME      OB 1888 2nd crystallization in Et2O
EXPNO                 3
PROCNO                1

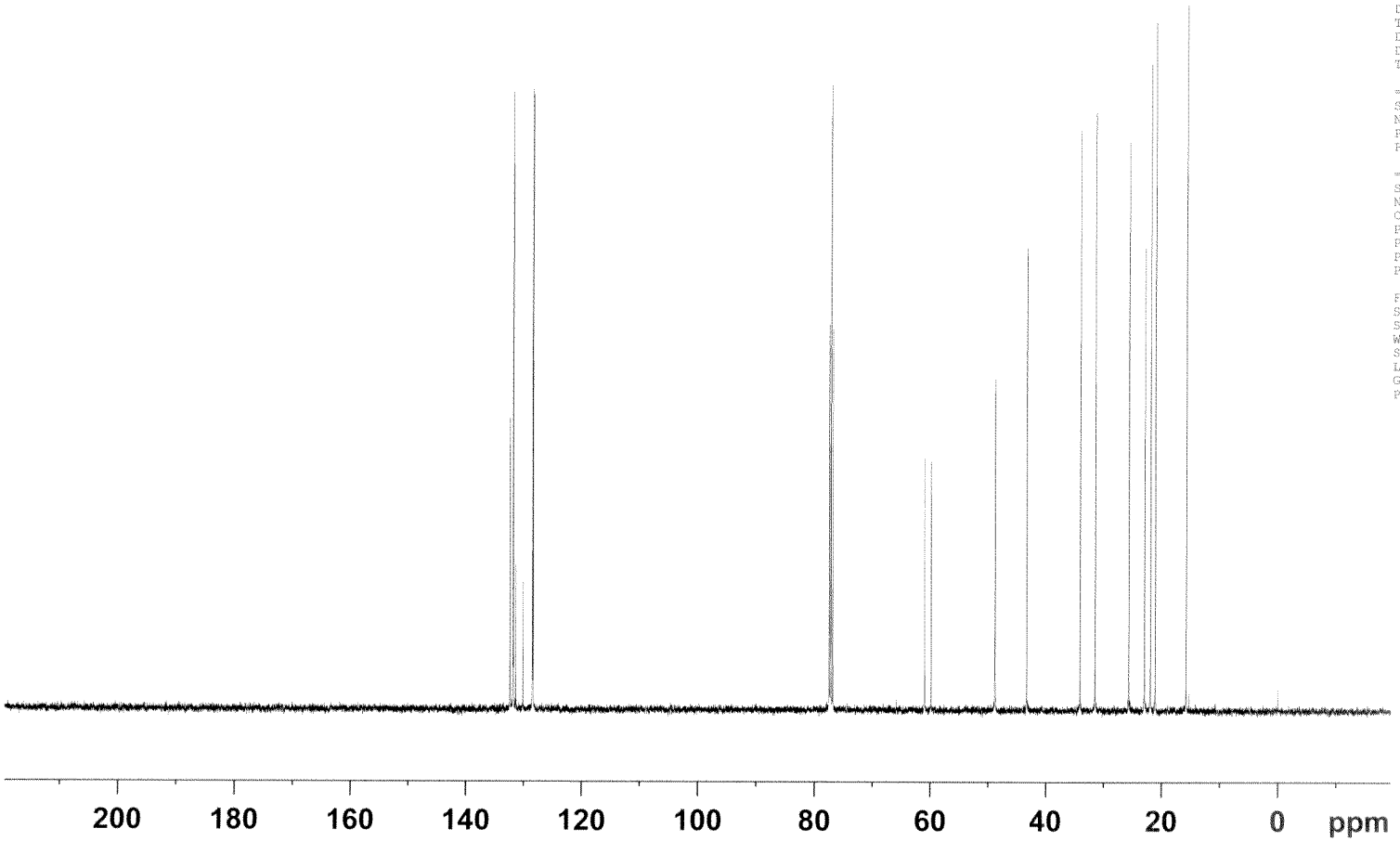
F2 - Acquisition Parameters
Date_          20150212
Time_             12.21
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  13
DS                   2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                16.39
DW              62.400 usec
DE               6.50 usec
TE              297.0 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1       400.1324710 MHz
NUC1               1H
P1              10.00 usec
PLW1       25.00300026 W

F2 - Processing parameters
SI               65536
SF         400.1300000 MHz
WDW                EM
SSB       0
LB              0.30 Hz
GB        0
PC              1.00
```



Compound (S_p)-3
^{13}C NMR




```
Current Data Parameters
NAME      OB 1888 2nd crystallization in Et2O
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150212
Time               12.30
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  120
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                298.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1               13C
P1                10.00 usec
PLW1        45.00000000 W

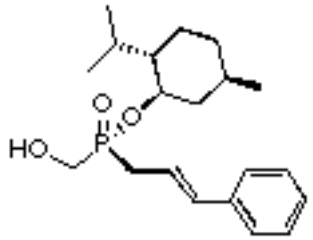
======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                32768
SF          100.6127685 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

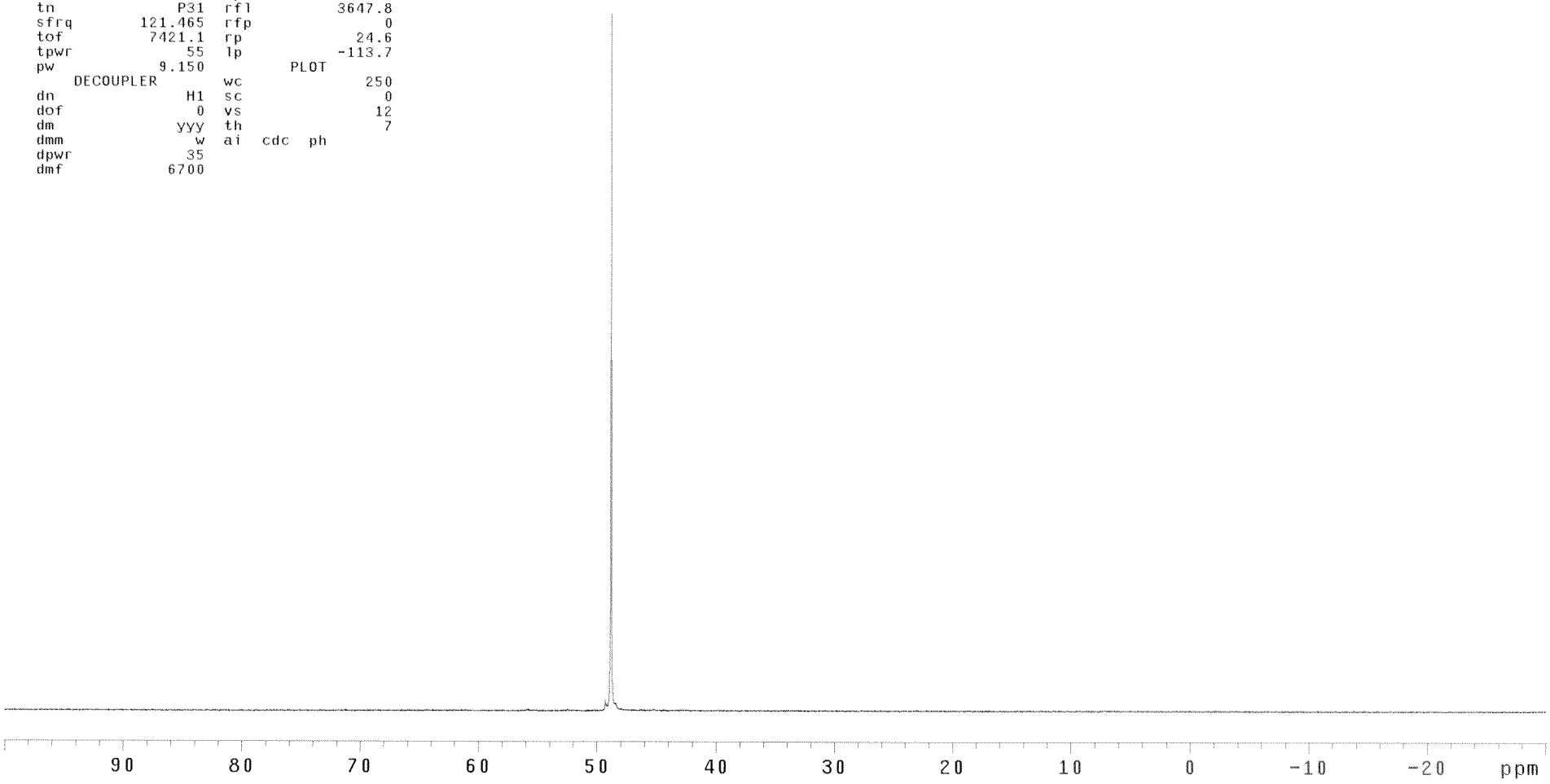
OB 900
pad=10 run with findz0 before acquisitio
n

exp1 Phosphorus

SAMPLE		SPECIAL	
date	Feb 1 2013	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2013.01.20/s_2013~		pw90	18.300
0201_12/data/cdcl3~		alfa	10.000
_02.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	24.6
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	12
dm	yyy	th	7
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



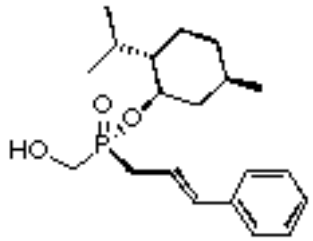
Compound (R_p)-4
$^{31}P\{^{1}H\}$ NMR decoupled



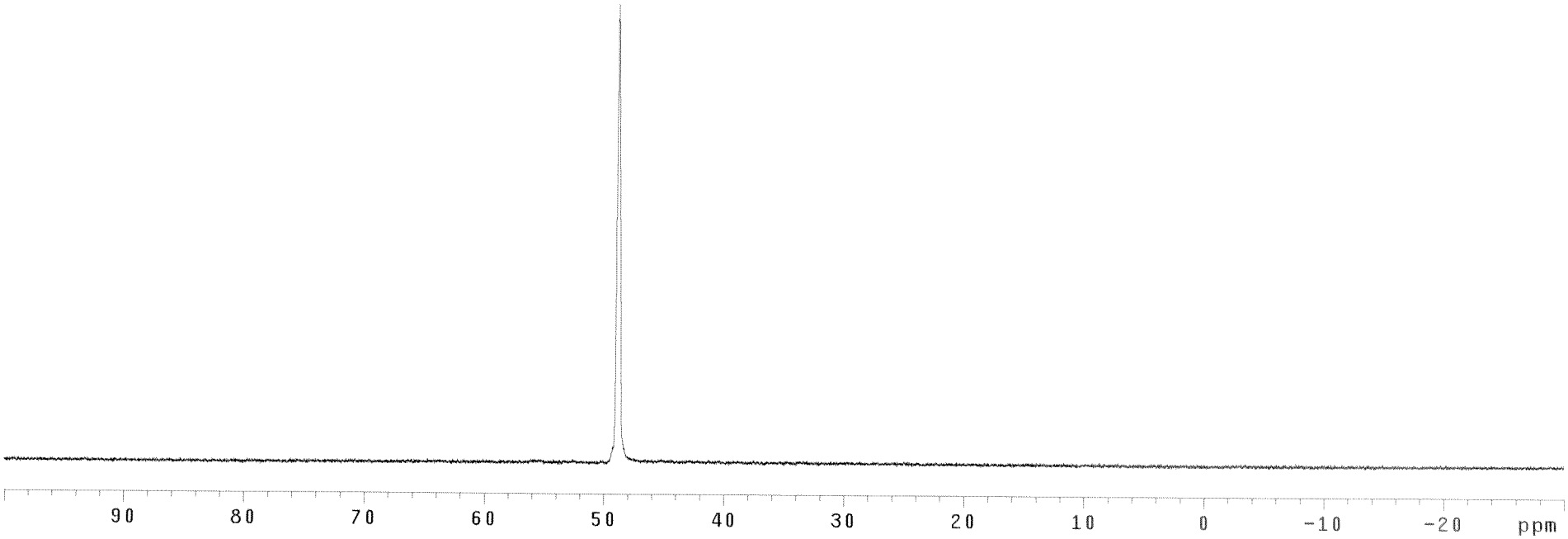
OB 900

exp1 Phosphorus

SAMPLE		SPECIAL	
date	Feb 1 2013	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
	/vnmrsys/data/auto~	hst	0.008
	_2013.01.20/s_2013~	pw90	18.300
	0201_12/data/cdcl3~	alfa	10.000
	_04.fid	FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	15.6
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	35
dm	ynn	th	3
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



Compound (R_p)-4
$^{31}P\{^{1}H\}$ NMR coupled



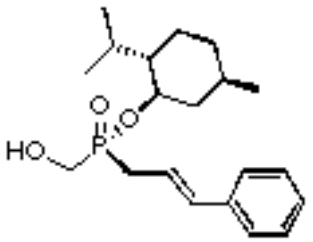
```
OB 900
pad=10 run with findz0 before acquisitio
n

exp1  Proton

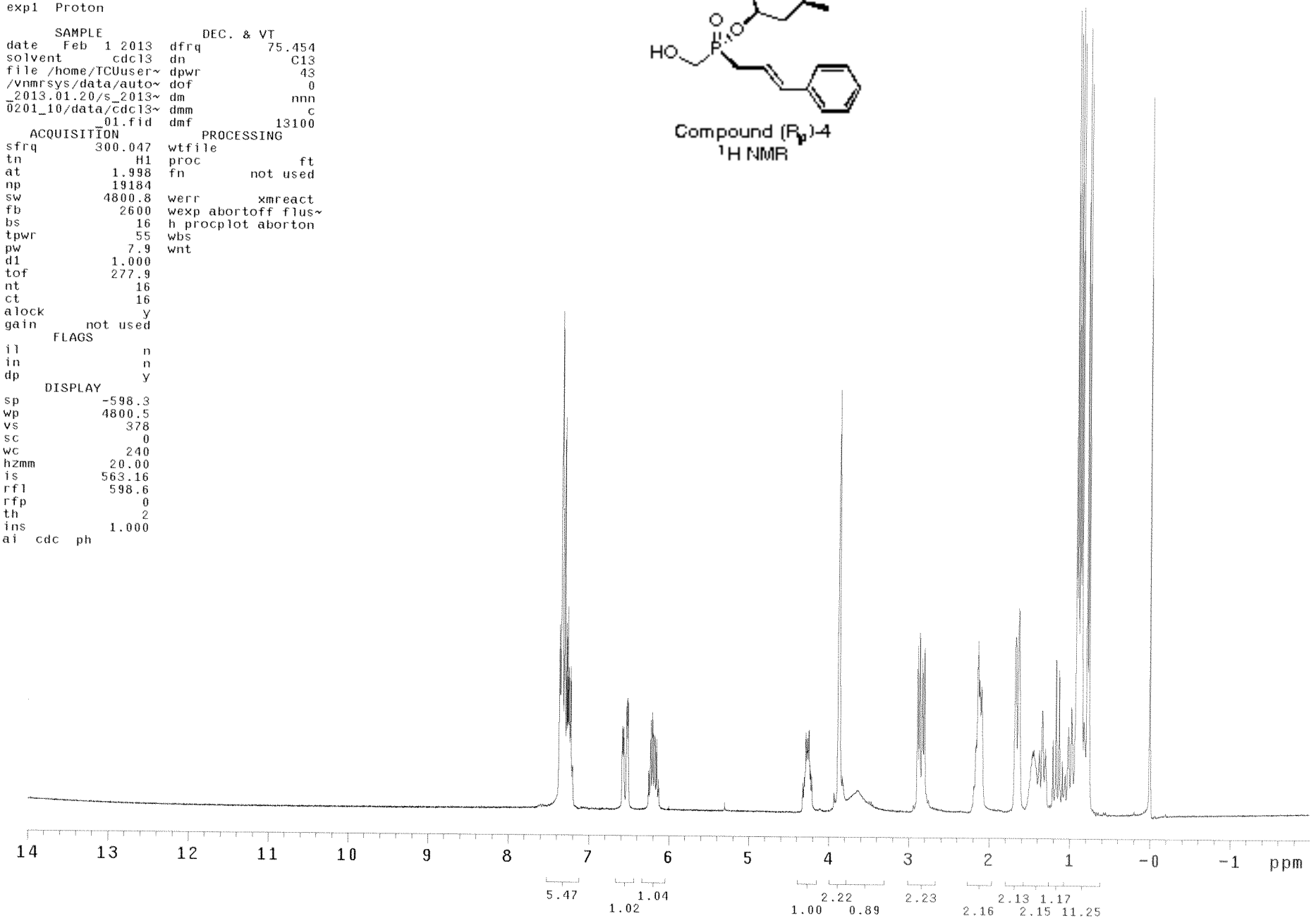
      SAMPLE
date   Feb  1 2013
solvent      cdcl3
file /home/TCUuser~
/vnmrsys/data/auto~
_2013.01.20/s_2013~
0201_10/data/cdcl3~
           _01.fid
   ACQUISITION
sfrq       300.047
tn              H1
at            1.998
np            19184
sw           4800.8
fb             2600
bs               16
tpwr             55
pw              7.9
d1            1.000
tof           277.9
nt               16
ct               16
alock             y
gain      not used
      FLAGS
il                n
in                n
dp                y
     DISPLAY
sp          -598.3
wp          4800.5
vs             378
sc               0
wc             240
hzmm         20.00
is          563.16
rfl          598.6
rfp              0
th               2
ins          1.000
ai  cdc  ph

      DEC. & VT
dfrq        75.454
dn             C13
dpwr            43
dof              0
dm             nnn
dmm              c
dmf          13100
     PROCESSING
wtfile
proc            ft
fn       not used

werr       xmreact
wexp abortoff flus~
h procplot aborton
wbs
wnt
```



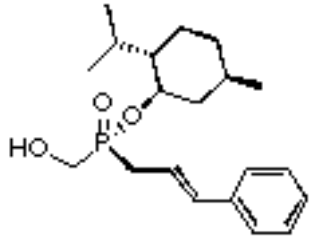
Compound (R_p)-4
^{1}H NMR



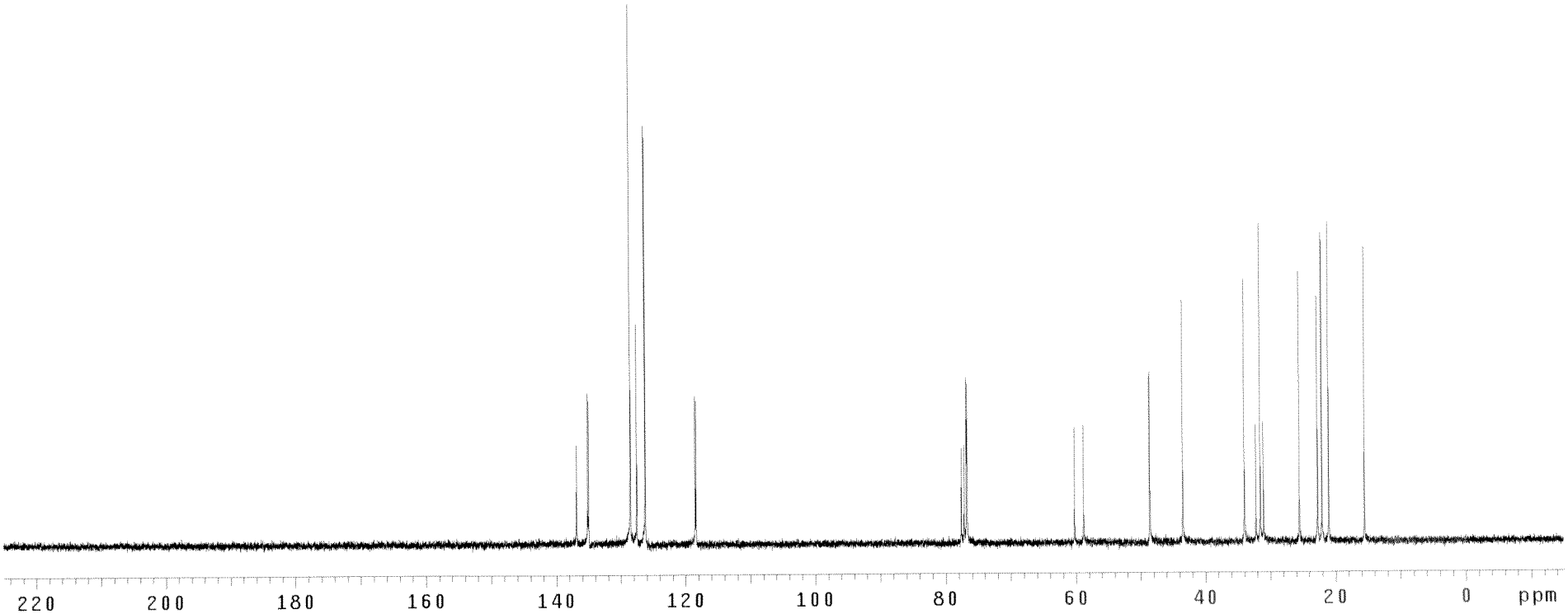
OB 900

exp1 Carbon

SAMPLE		SPECIAL	
date	Feb 1 2013	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2013.01.20/s_2013~		pw90	18.500
0201_12/data/cdcl3~		alfa	10.000
05.fid		FLAGS	
ACQUISITION		il	n
sw	18115.9	in	n
at	1.301	dp	y
np	47120	hs	nn
fb	10000	PROCESSING	
bs	64	lb	0.50
d1	2.000	fn	not used
nt	750	DISPLAY	
ct	750	sp	-1135.5
TRANSMITTER		wp	18115.4
tn	C13	rfl	1136.1
sfrq	75.454	rfp	0
tof	766.0	rp	9.2
tpwr	58	lp	-182.4
pw	9.250	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	332
dm	yyy	th	6
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



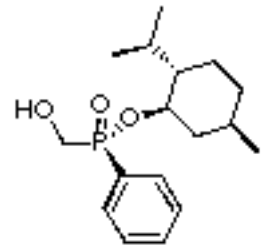
Compound (R_p)-4
^{13}C NMR



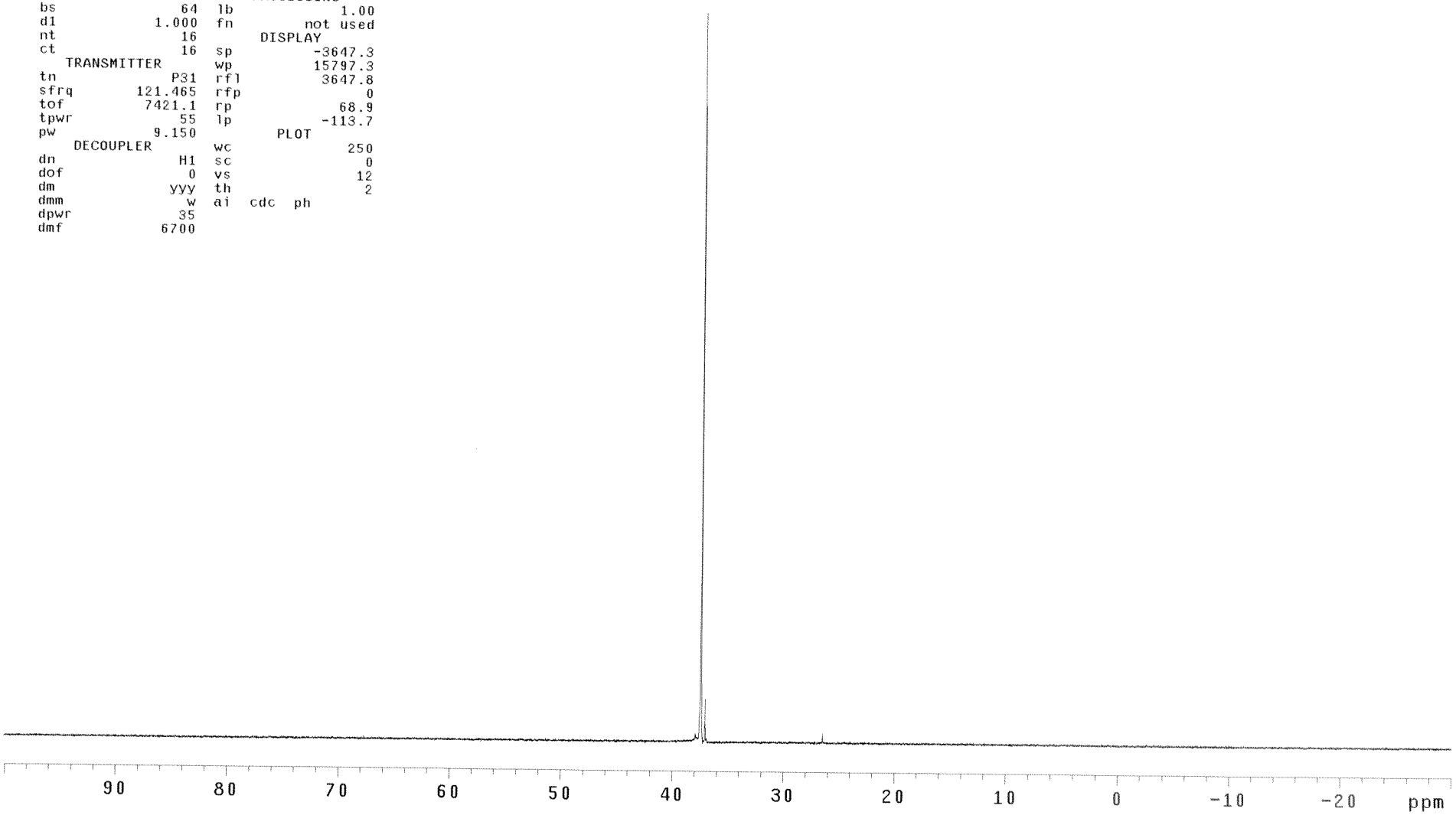
OB 876
pad=10 run with findz0 before acquisitio
n

exp1 Phosphorus

SAMPLE		SPECIAL	
date	Dec 18 2012	temp	not used
solvent	cdcl3	gain	25
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2012.12.14/s_2012~		pw90	18.300
1218_04/data/cdcl3~		alfa	10.000
_01.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	68.9
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	12
dm	yyy	th	2
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



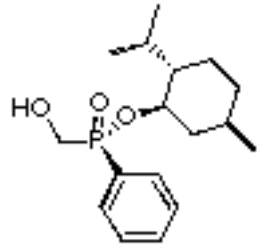
Compound (R_p)-3
$^{31}P/^{1}H$ NMR decoupled



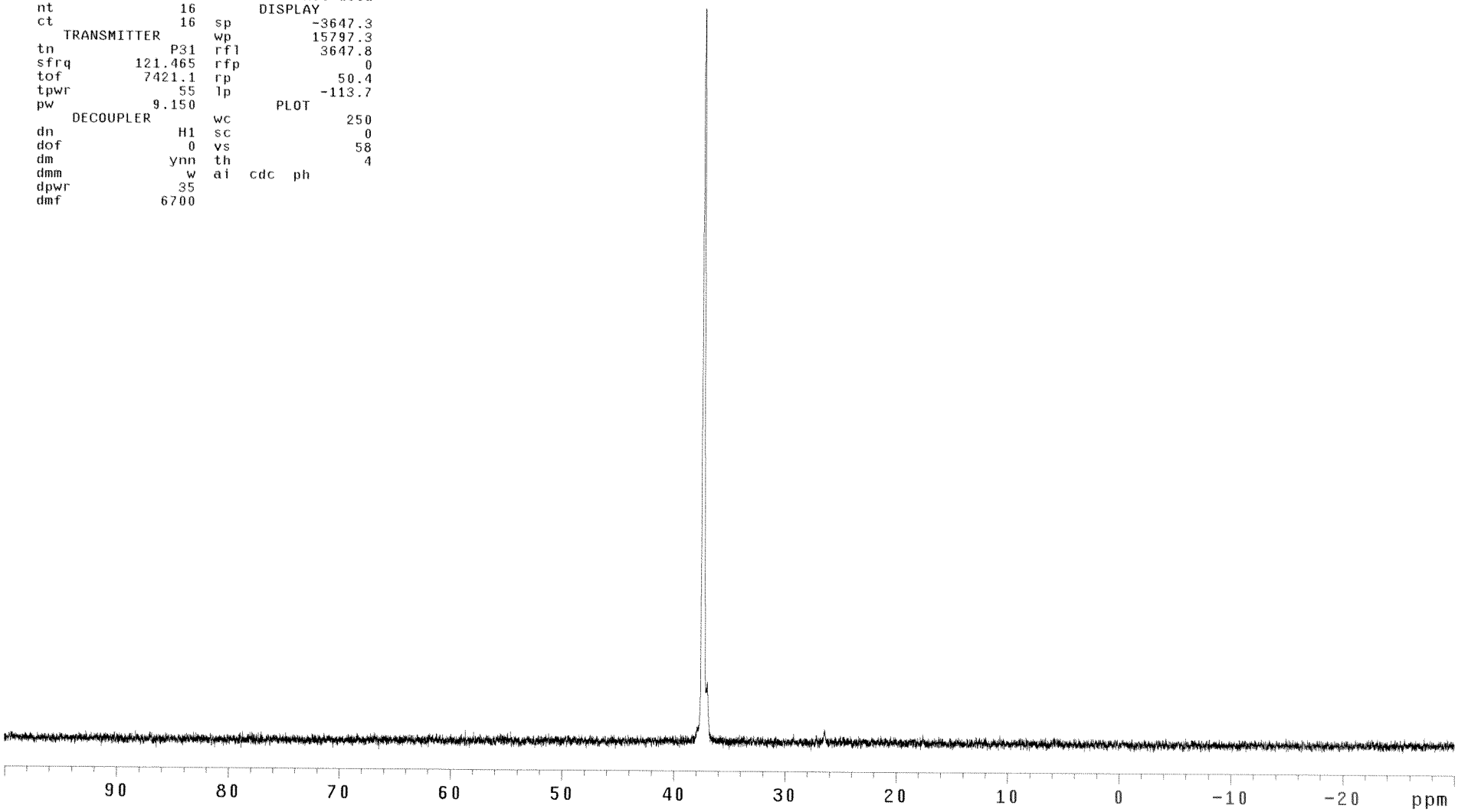
OB 876

exp1 Phosphorus

SAMPLE		SPECIAL	
date	Dec 18 2012	temp	not used
solvent	cdcl3	gain	25
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2012.12.14/s_2012~		pw90	18.300
1218_04/data/cdcl3~		alfa	10.000
_02.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	50.4
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	58
dm	ynn	th	4
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



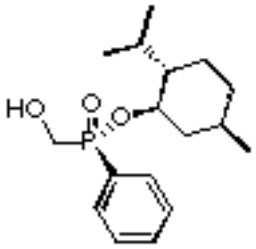
Compound (R_p)-3
$^{31}P/^{1}H$ NMR coupled



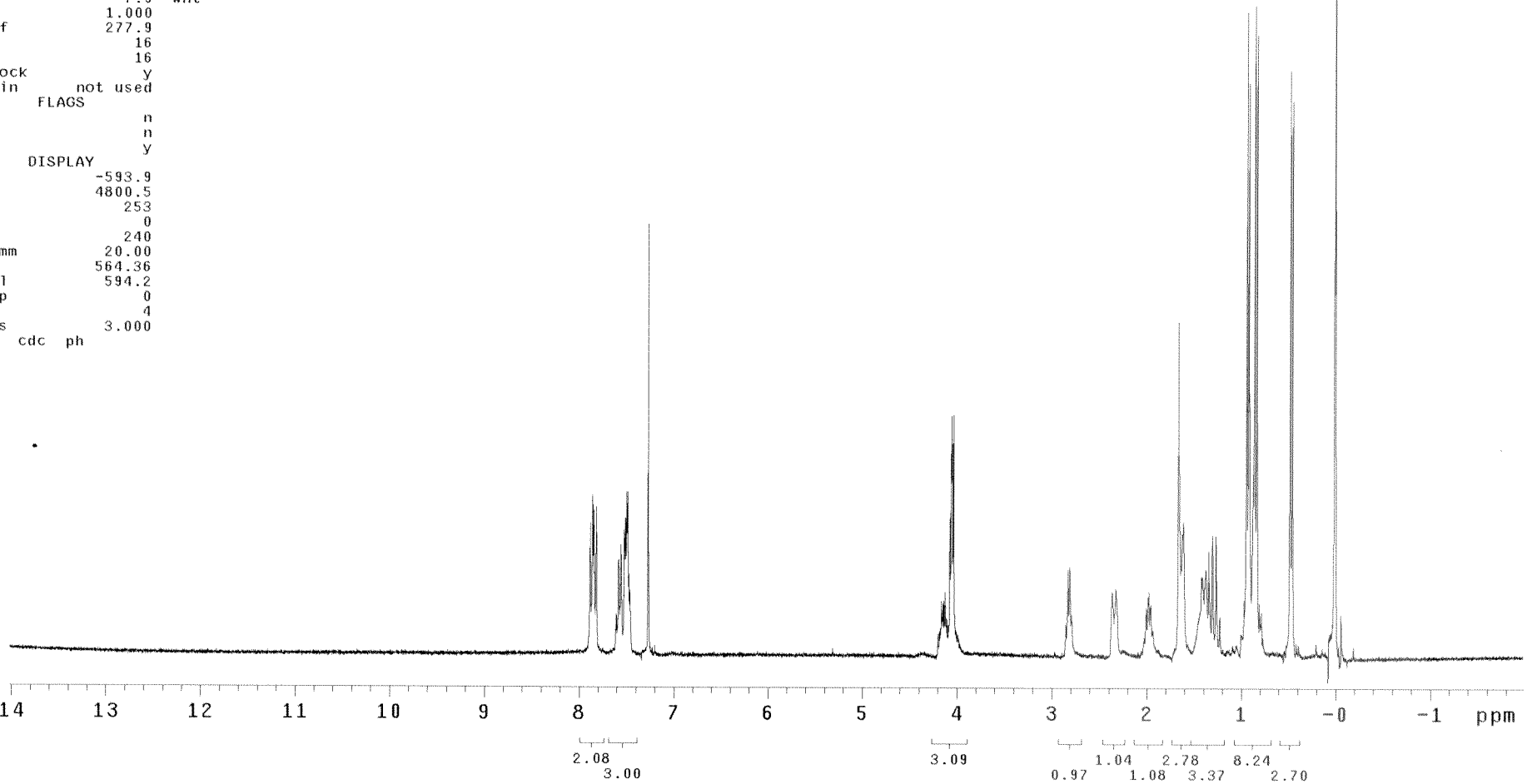
OB 876
pad=10 run with findz0 before acquisitio
n

exp1 Proton

SAMPLE		DEC. & VT	
date	Dec 18 2012	dfrq	75.454
solvent	cdcl3	dn	C13
file	/home/TCUuser~	dpwr	43
	/vnmrsys/data/auto~	dof	0
	_2012.12.14/s_2012~	dm	nnn
	1218_02/data/cdcl3~	dmm	c
	_01.fid	dmf	13100
ACQUISITION		PROCESSING	
sfrq	300.047	wtfile	
tn	H1	proc	ft
at	1.998	fn	not used
np	19184		
sw	4800.8	werr	xmreact
fb	2600	wexp	abortoff flus~
bs	16	h procplot	aborton
tpwr	55	wbs	
pw	7.9	wnt	
d1	1.000		
tof	277.9		
nt	16		
ct	16		
alock	y		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-593.9		
wp	4800.5		
vs	253		
sc	0		
wc	240		
hzmm	20.00		
is	564.36		
rfl	594.2		
rfp	0		
th	4		
ins	3.000		
ai cdc ph			



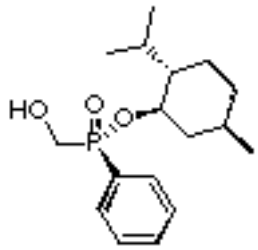
Compound (R_p)-3
^{1}H NMR



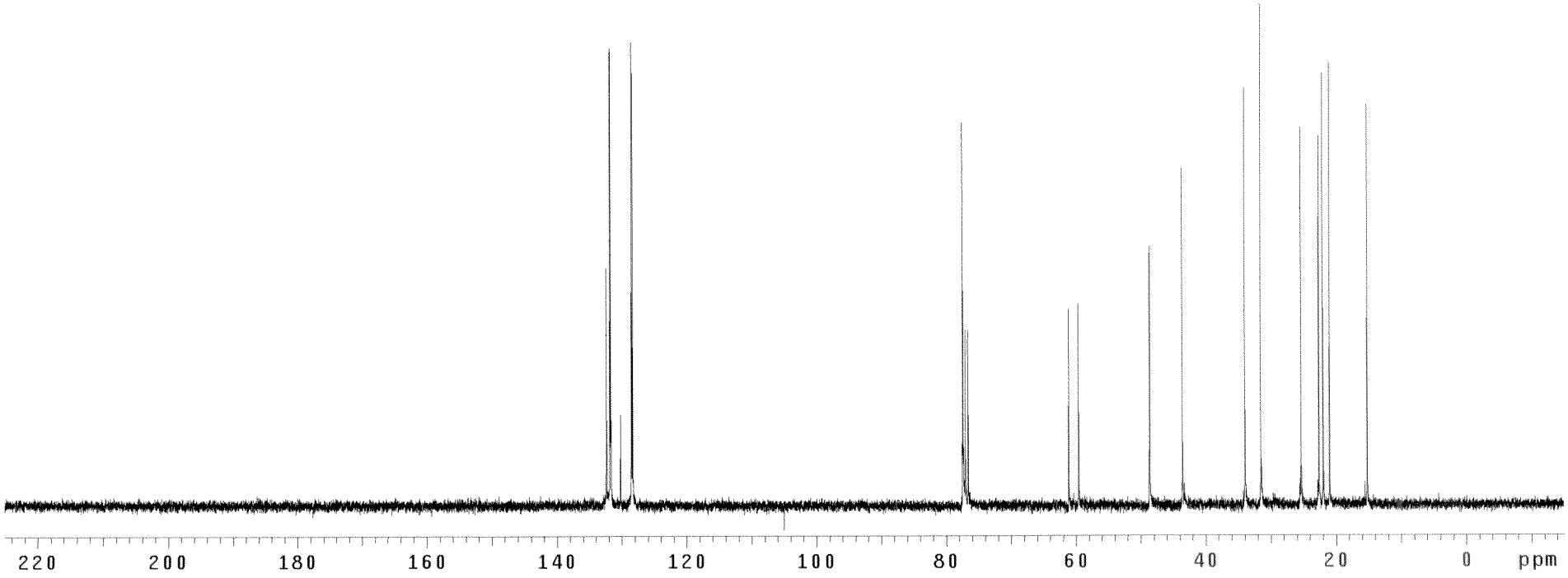
OB 876

exp1 Carbon

SAMPLE		SPECIAL	
date	Dec 18 2012	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2012.12.14/s_2012~		pw90	18.500
1218_04/data/cdcl3~		alfa	10.000
03.fid		FLAGS	
ACQUISITION		il	n
sw	18115.9	in	n
at	1.301	dp	y
np	47120	hs	nn
fb	10000	PROCESSING	
bs	64	lb	0.50
d1	2.000	fn	not used
nt	800	DISPLAY	
ct	800	sp	-1135.5
TRANSMITTER		wp	18115.4
tn	C13	rfl	1136.1
sfrq	75.454	rfp	0
tof	766.0	rp	43.1
tpwr	58	lp	-199.8
pw	9.250	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	483
dm	yyy	th	7
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



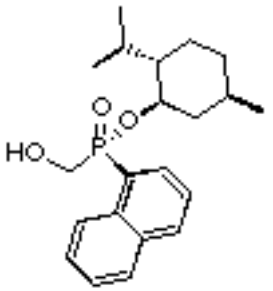
Compound (R_p)-3
^{13}C NMR



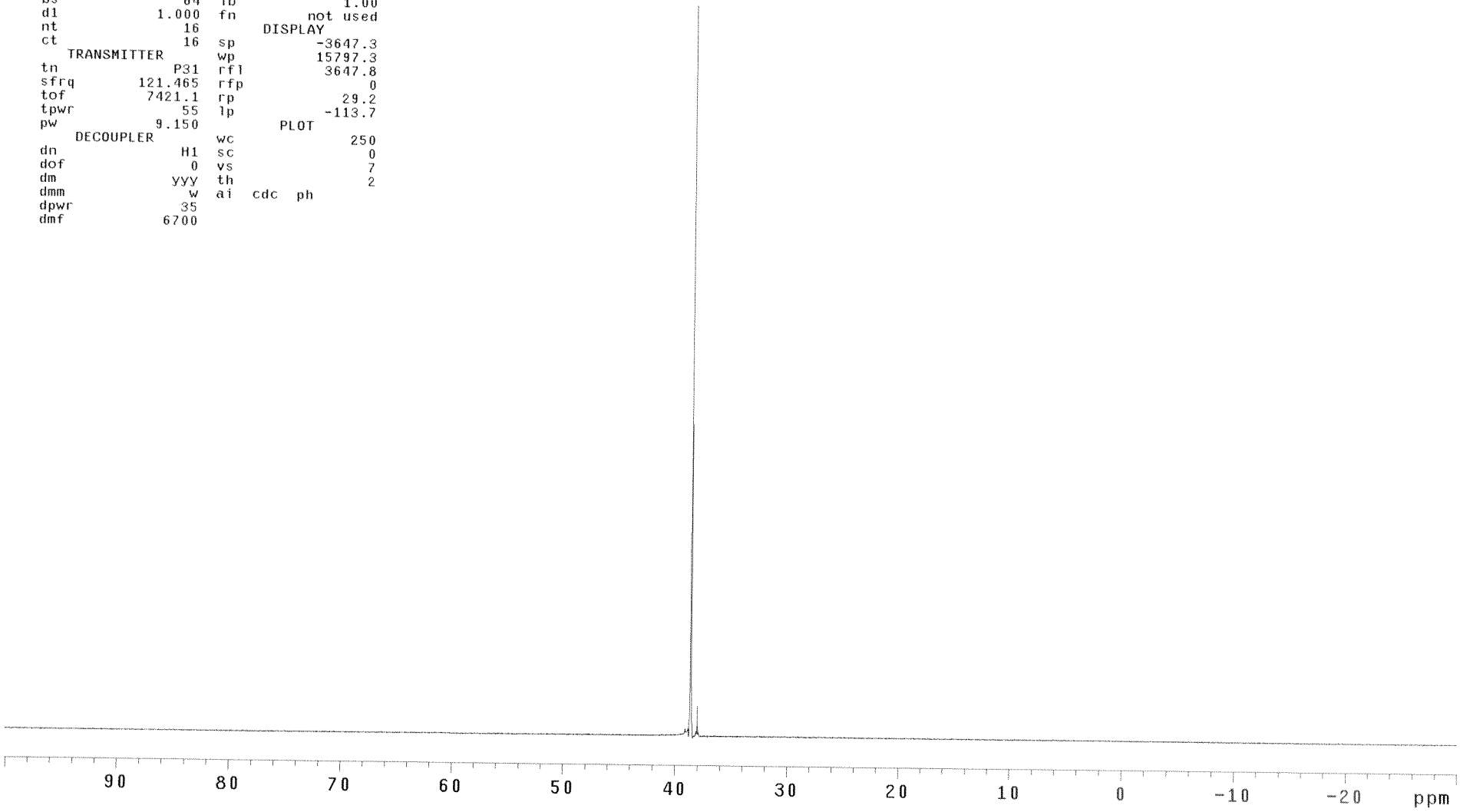
OB 882
pad=10 run with findz0 before acquisitio
n

exp1 Phosphorus

SAMPLE		SPECIAL	
date	Dec 18 2012	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2012.12.14/s_2012~		pw90	18.300
1218_31/data/cdcl3~		alfa	10.000
_02.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	29.2
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	7
dm	yyy	th	2
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



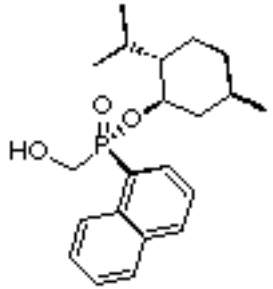
Compound (R_p)-5
$^{31}P\{^1H\}$ NMR decoupled



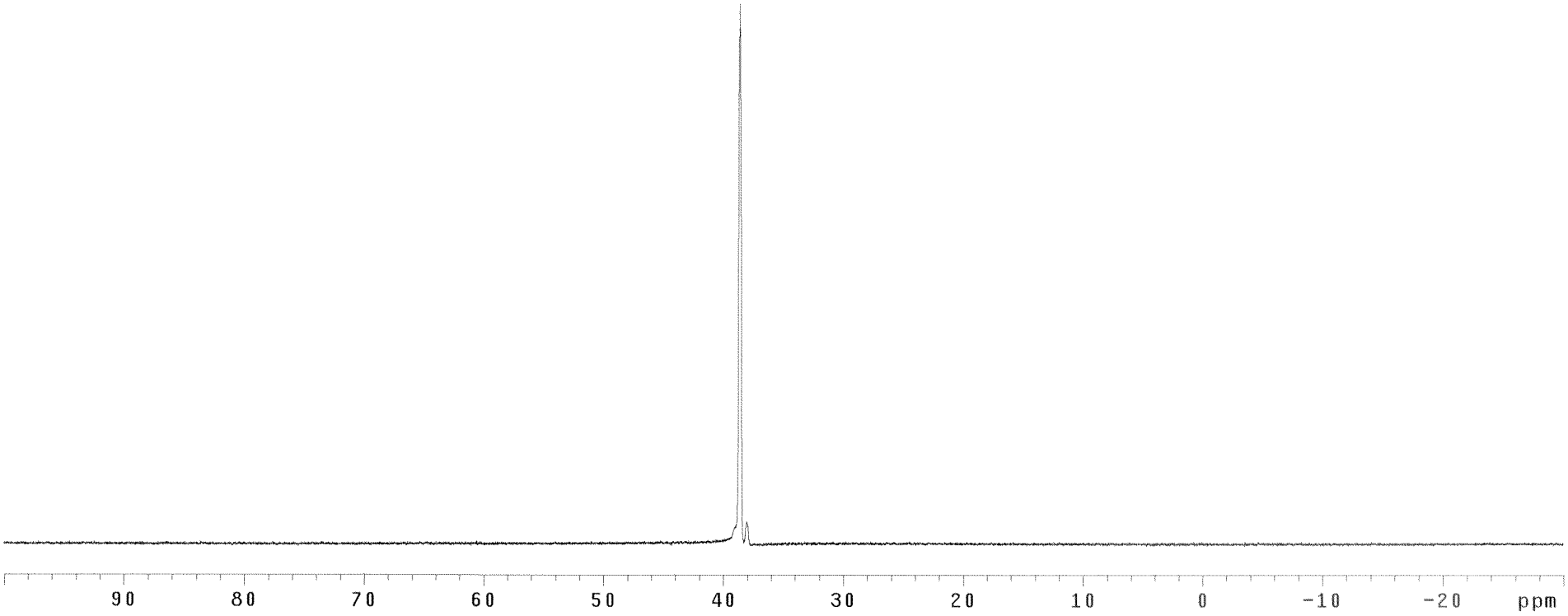
OB 882

exp1 Phosphorus

SAMPLE		SPECIAL	
date	Dec 18 2012	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2012.12.14/s_2012~		pw90	18.300
1218_31/data/cdcl3~		alfa	10.000
04.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	31.8
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	26
dm	ynn	th	2
dmm	w	ai cdc ph	
dpwr	35		
dmf	6700		



Compound (R_p)-5
$^{31}P\{^1H\}$ NMR coupled

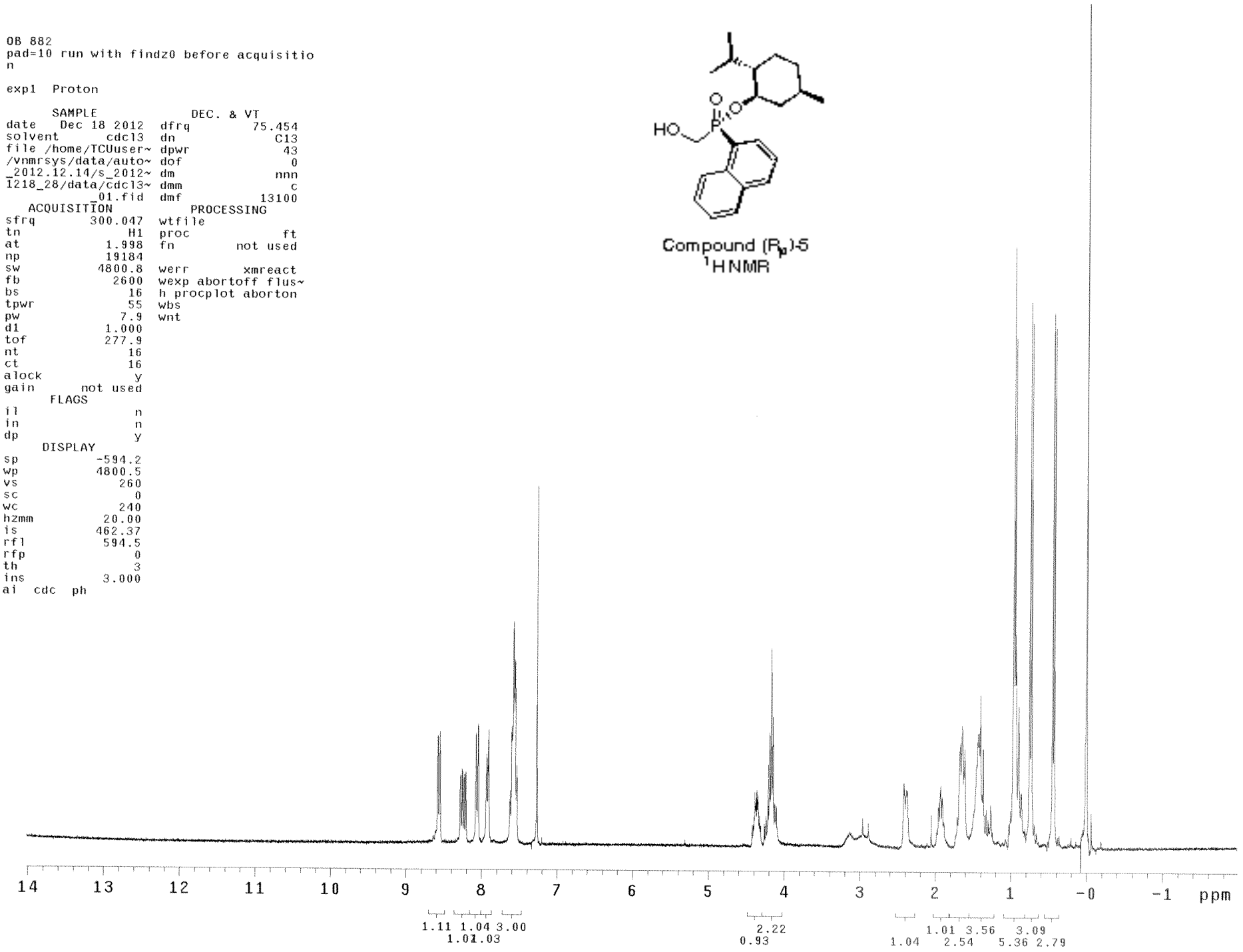


OB 882
pad=10 run with findz0 before acquisitio
n

exp1 Proton

SAMPLE		DEC. & VT	
date	Dec 18 2012	dfrq	75.454
solvent	cdcl3	dn	C13
file	/home/TCUuser~	dpwr	43
	/vnmrsys/data/auto~	dof	0
	_2012.12.14/s_2012~	dm	nnn
	1218_28/data/cdcl3~	dmm	c
	01.fid	dmf	13100
ACQUISITION		PROCESSING	
sfrq	300.047	wtfile	
tn	H1	proc	ft
at	1.998	fn	not used
np	19184		
sw	4800.8	werr	xmreact
fb	2600	wexp	abortoff flus~
bs	16	h procplot	aborton
tpwr	55	wbs	
pw	7.9	wnt	
d1	1.000		
tof	277.9		
nt	16		
ct	16		
alock	y		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-594.2		
wp	4800.5		
vs	260		
sc	0		
wc	240		
hzmm	20.00		
is	462.37		
rfl	594.5		
rfp	0		
th	3		
ins	3.000		
ai cdc ph			

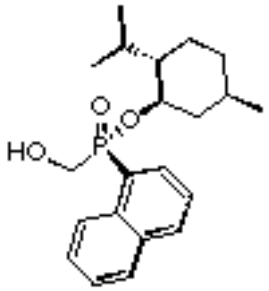
Compound (R_p)-5
^{1}HNMR



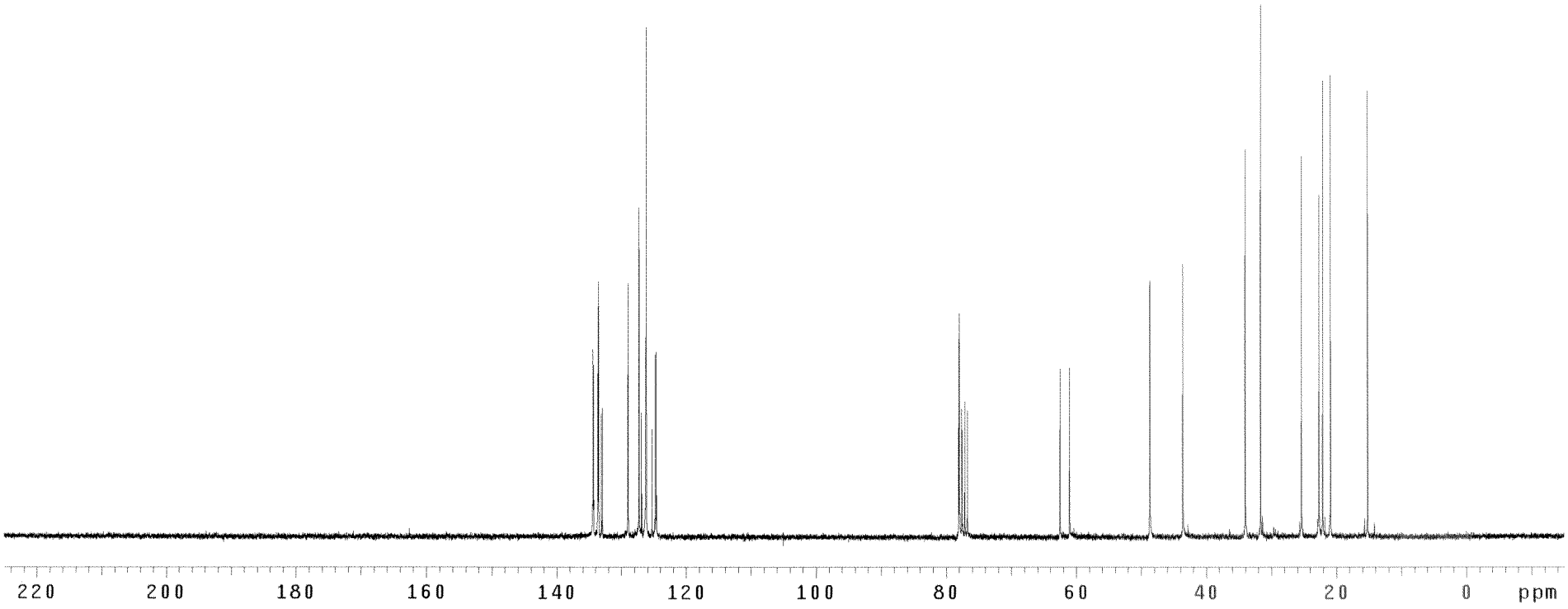
OB 882

exp1 Carbon

SAMPLE		SPECIAL	
date	Dec 18 2012	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2012.12.14/s_2012~		pw90	18.500
1218_31/data/cdcl3~		alfa	10.000
_05.fid		FLAGS	
ACQUISITION		il	n
sw	18115.9	in	n
at	1.301	dp	y
np	47120	hs	nn
fb	10000	PROCESSING	
bs	64	lb	0.50
d1	2.000	fn	not used
nt	1000	DISPLAY	
ct	1000	sp	-1135.5
TRANSMITTER		wp	18115.4
tn	C13	rfl	1136.1
sfrq	75.454	rfp	0
tof	766.0	rp	-7.6
tpwr	58	lp	-201.1
pw	9.250	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	272
dm	yyy	th	6
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



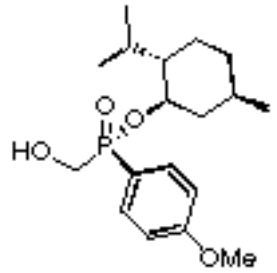
Compound (R_p)-5
^{13}C NMR



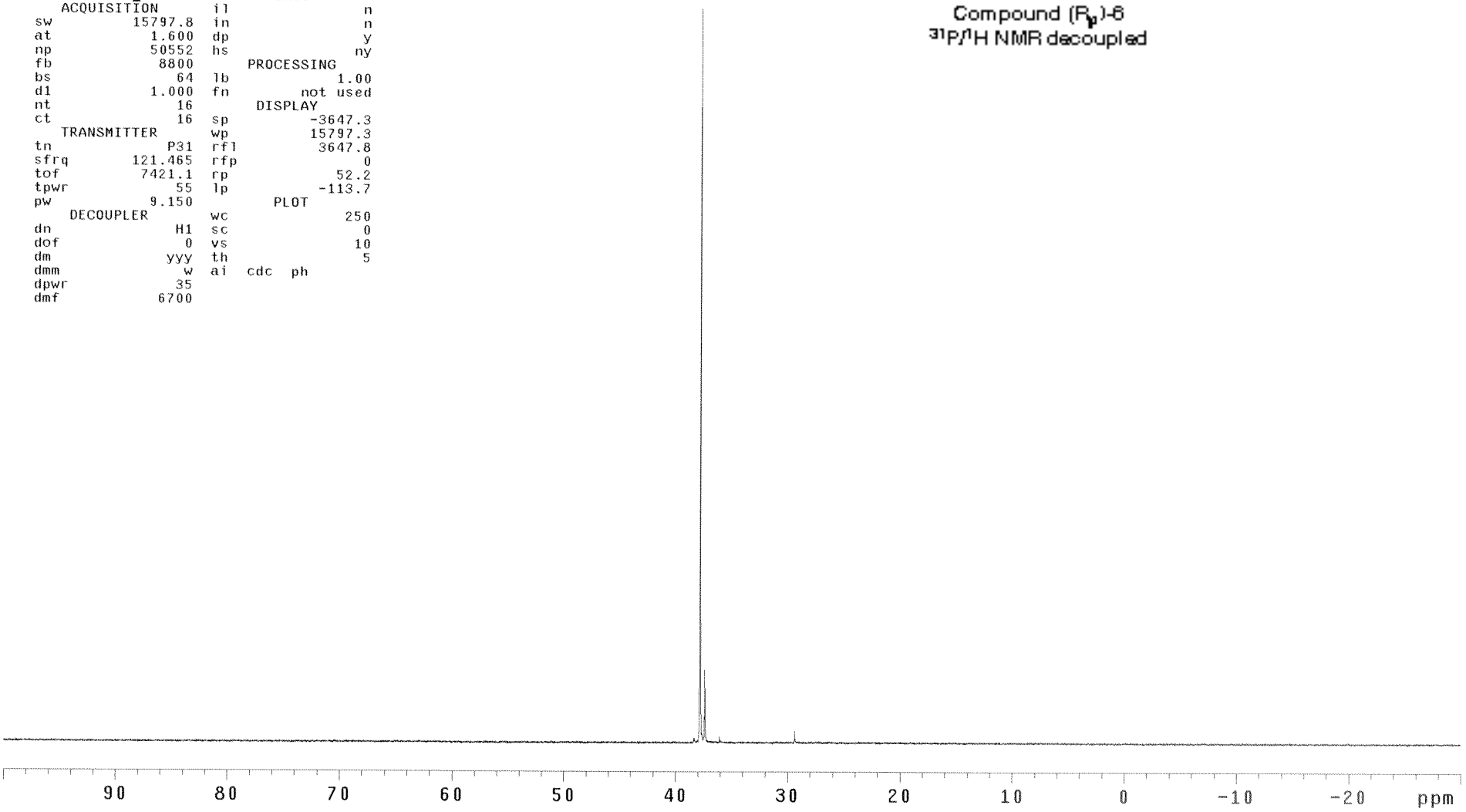
OB 883
pad=10 run with findz0 before acquisitio
n

exp1 Phosphorus

SAMPLE		SPECIAL	
date	Dec 19 2012	temp	not used
solvent	cdcl3	gain	25
file	/home/TCUuser~	spin	20
	/vnmrsys/data/auto~	hst	0.008
	_2012.12.14/s_2012~	pw90	18.300
	1219_02/data/cdcl3~	alfa	10.000
	_01.fid	FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	52.2
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	10
dm	yyy	th	5
dmm	w	ai cdc ph	
dpwr	35		
dmf	6700		



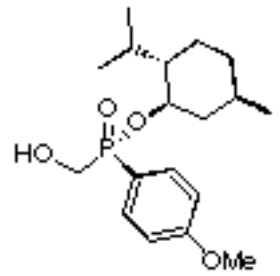
Compound (R_p)-6
$^{31}P\{^{1}H\}$ NMR decoupled



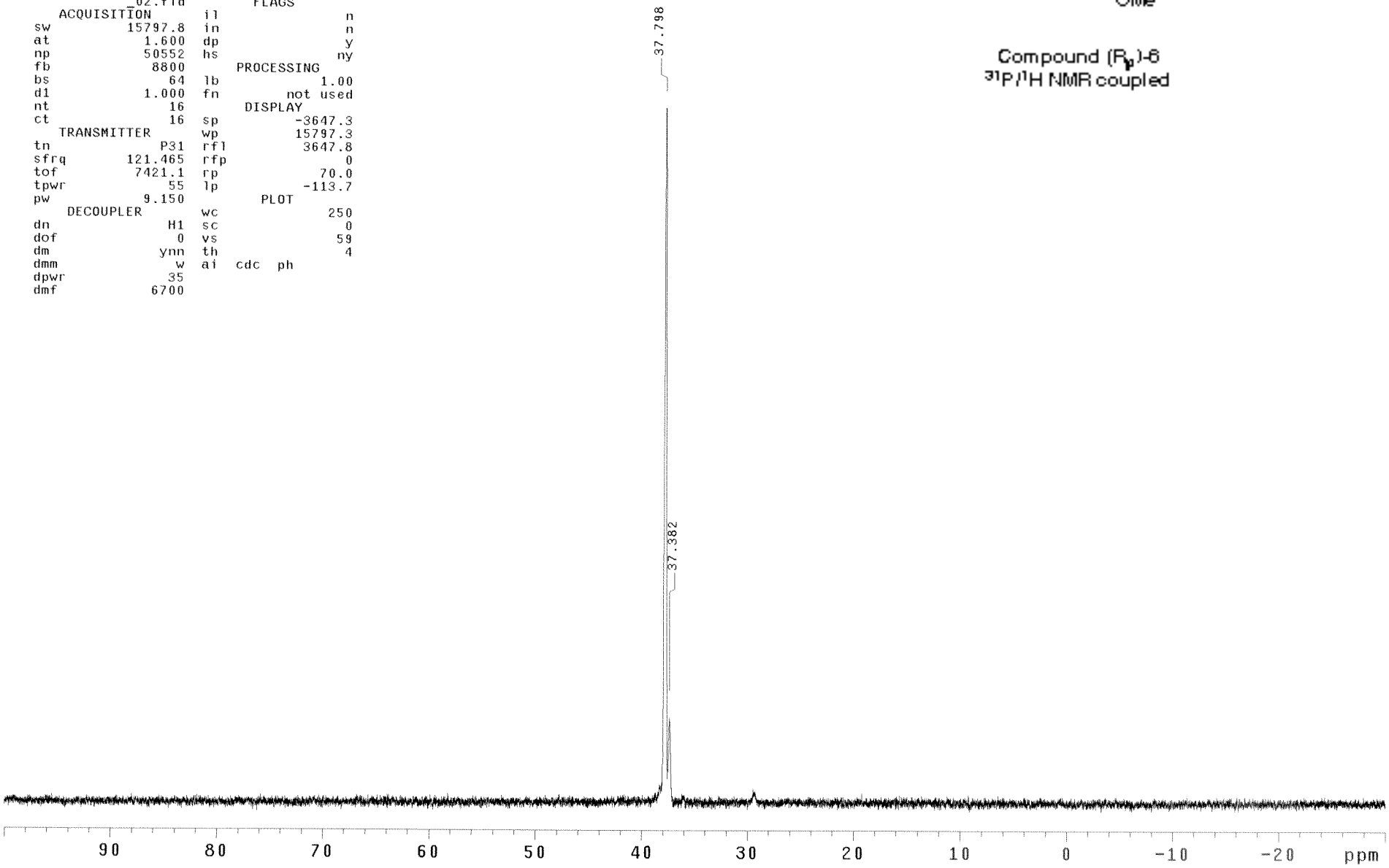
OB 883

exp1 Phosphorus

SAMPLE		SPECIAL	
date	Dec 19 2012	temp	not used
solvent	cdcl3	gain	25
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2012.12.14/s_2012~		pw90	18.300
1219_02/data/cdcl3~		alfa	10.000
_02.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	70.0
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	59
dm	ynn	th	4
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



Compound (R_p)-6
$^{31}P/^{1}H$ NMR coupled

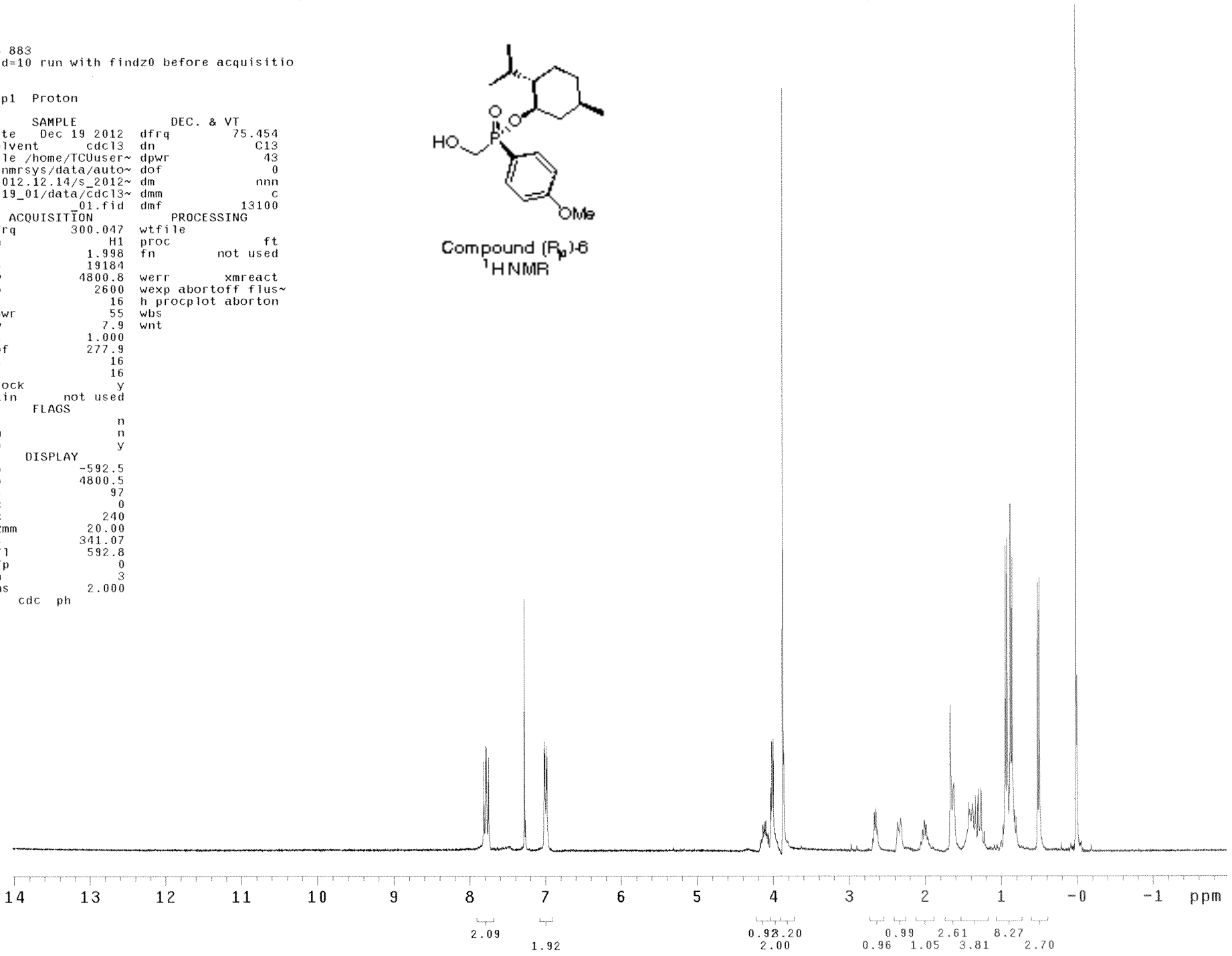


OB 883
pad=10 run with findz0 before acquisitio
n

exp1 Proton

SAMPLE		DEC. & VT	
date	Dec 19 2012	dfrq	75.454
solvent	cdcl3	dn	C13
file	/home/TCUuser~	dpwr	43
	/vnmrsys/data/auto~	dof	0
	_2012.12.14/s_2012~	dm	nnn
	1219_01/data/cdcl3~	dmm	c
	01.fid	dmf	13100
ACQUISITION		PROCESSING	
sfrq	300.047	wtfile	
tn	H1	proc	ft
at	1.998	fn	not used
np	19184		
sw	4800.8	werr	xmreact
fb	2600	wexp	abortoff flus~
bs	16	h procplot	aborton
tpwr	55	wbs	
pw	7.9	wnt	
d1	1.000		
tof	277.9		
nt	16		
ct	16		
alock	y		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-592.5		
wp	4800.5		
vs	97		
sc	0		
wc	240		
hzmm	20.00		
is	341.07		
rfl	592.8		
rfp	0		
th	3		
ins	2.000		
ai cdc ph			

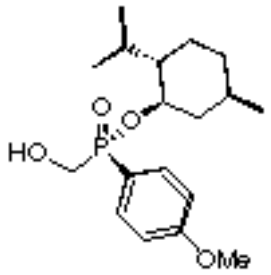
Compound (R_p)-6
^{1}H NMR



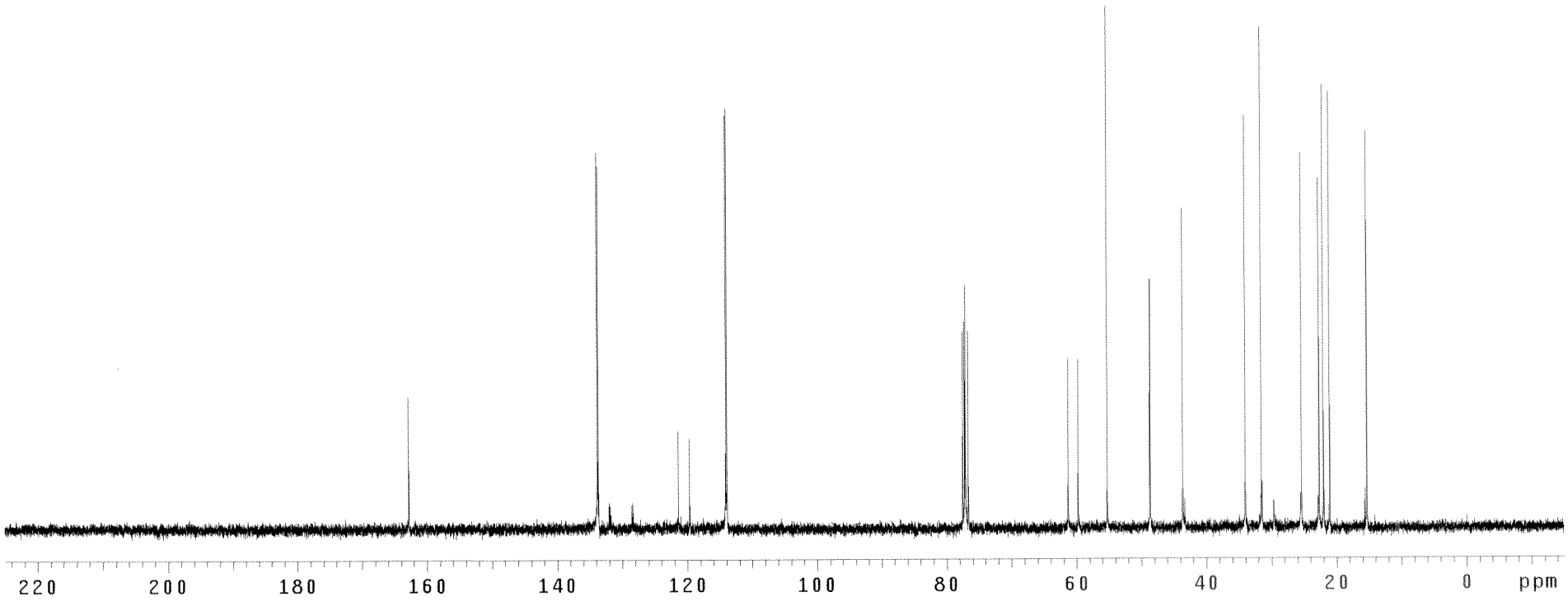
OB 883

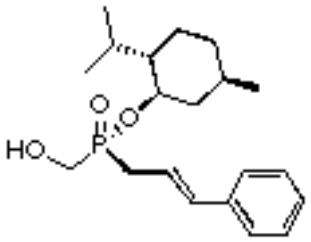
exp1 Carbon

SAMPLE		SPECIAL	
date	Dec 19 2012	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2012.12.14/s_2012~		pw90	18.500
1219_02/data/cdcl3~		alfa	10.000
_03.fid		FLAGS	
ACQUISITION		il	n
sw	18115.9	in	n
at	1.301	dp	y
np	47120	hs	nn
fb	10000	PROCESSING	
bs	64	lb	0.50
d1	2.000	fn	not used
nt	800	DISPLAY	
ct	800	sp	-1135.5
TRANSMITTER		wp	18115.4
tn	C13	rfl	1136.1
sfrq	75.454	rfp	0
tof	766.0	rp	5.2
tpwr	58	lp	-204.1
pw	9.250	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	510
dm	yyy	th	7
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		

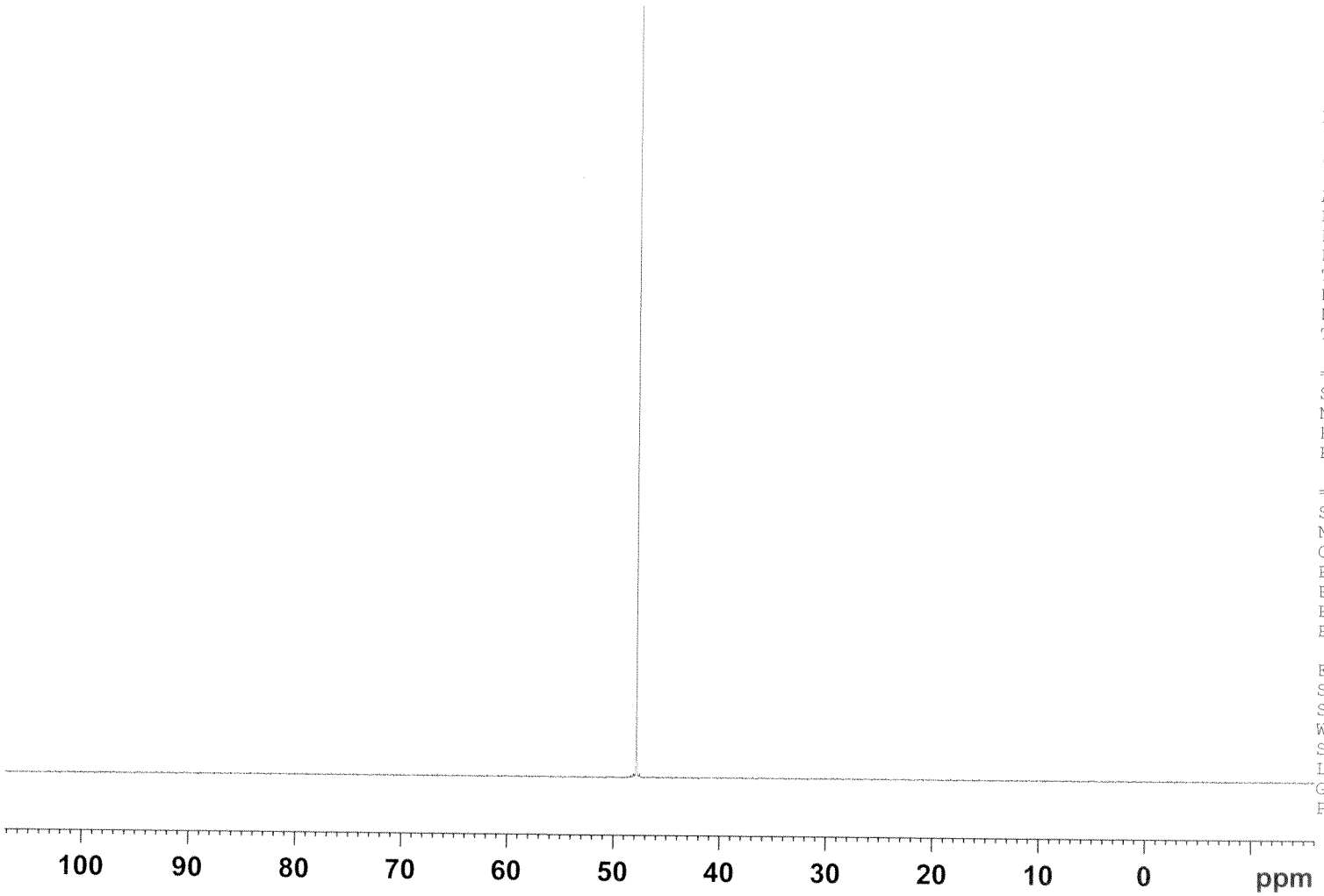


Compound (R_p)-6
^{13}C NMR





Compound (R_p)-4
$^{31}P\{^1H\}$ NMR decoupled



BRUKER

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EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150828
Time                18.10
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     4
SWH            64102.563 Hz
FIDRES          0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                 7.800 usec
DE                 6.50 usec
TE                295.4 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

========== CHANNEL f1 ==========
SFO1         161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1         15.00000000 W

========== CHANNEL f2 ==========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                 32768
SF           161.9755930 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
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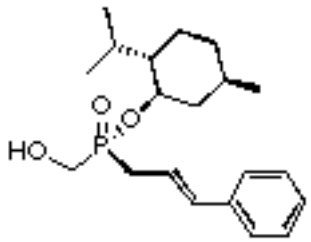

Compound (R_p)-4
$^{31}P/^{1}H$ NMR coupled

BRUKER

```
Current Data Parameters
NAME      OB 2157 after column
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_              20150828
Time                  18.14
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       32
DS                        4
SWH               64102.563 Hz
FIDRES             0.978127 Hz
AQ                0.5111808 sec
RG                   203.57
DW                    7.800 usec
DE                    6.50 usec
TE                   294.9 K
D1               2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1            161.9674942 MHz
NUC1                    31P
P1                    14.25 usec
PLW1            15.00000000 W

F2 - Processing parameters
SI                    32768
SF              161.9755930 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
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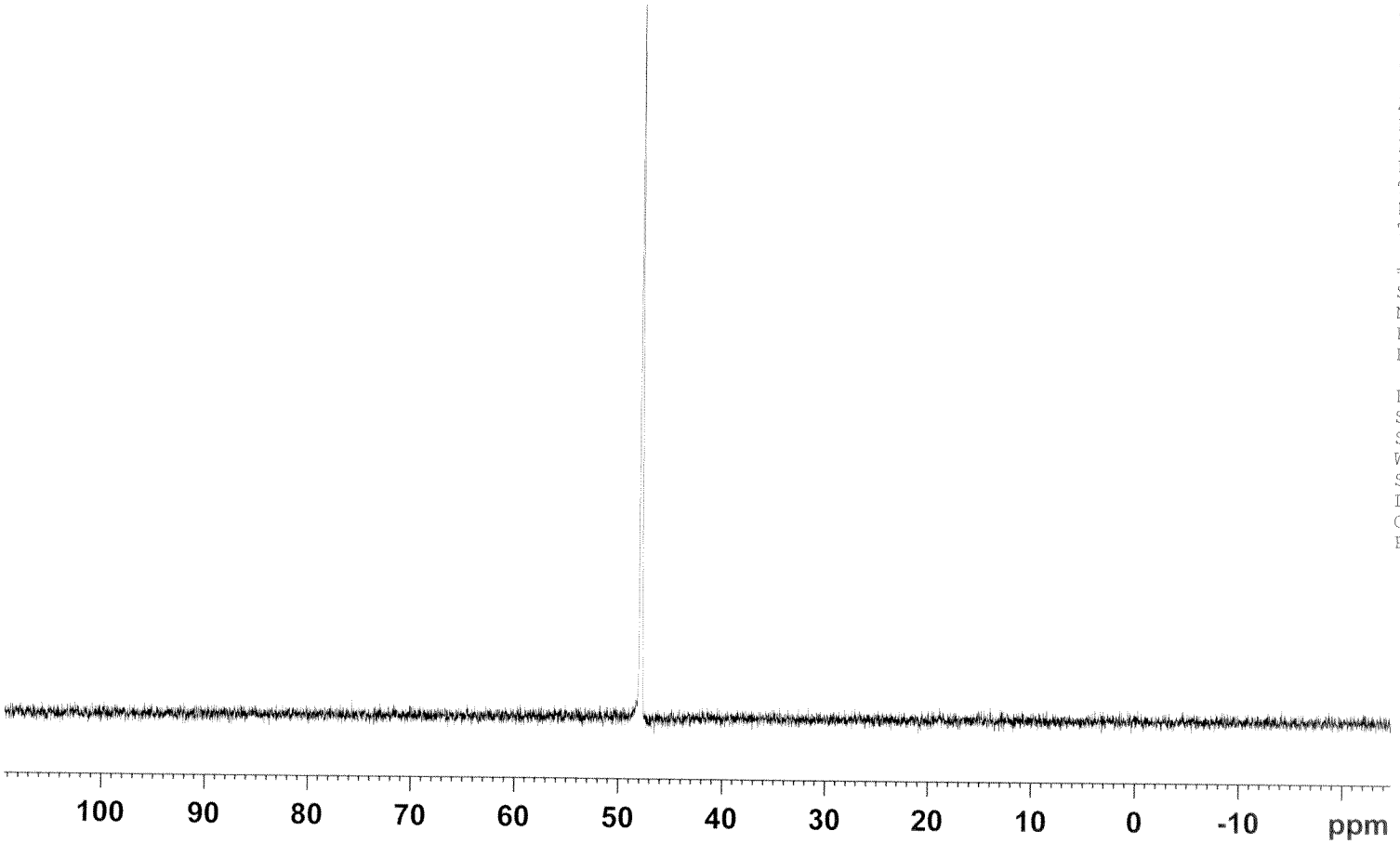
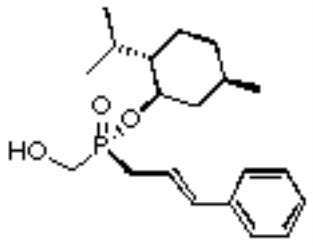
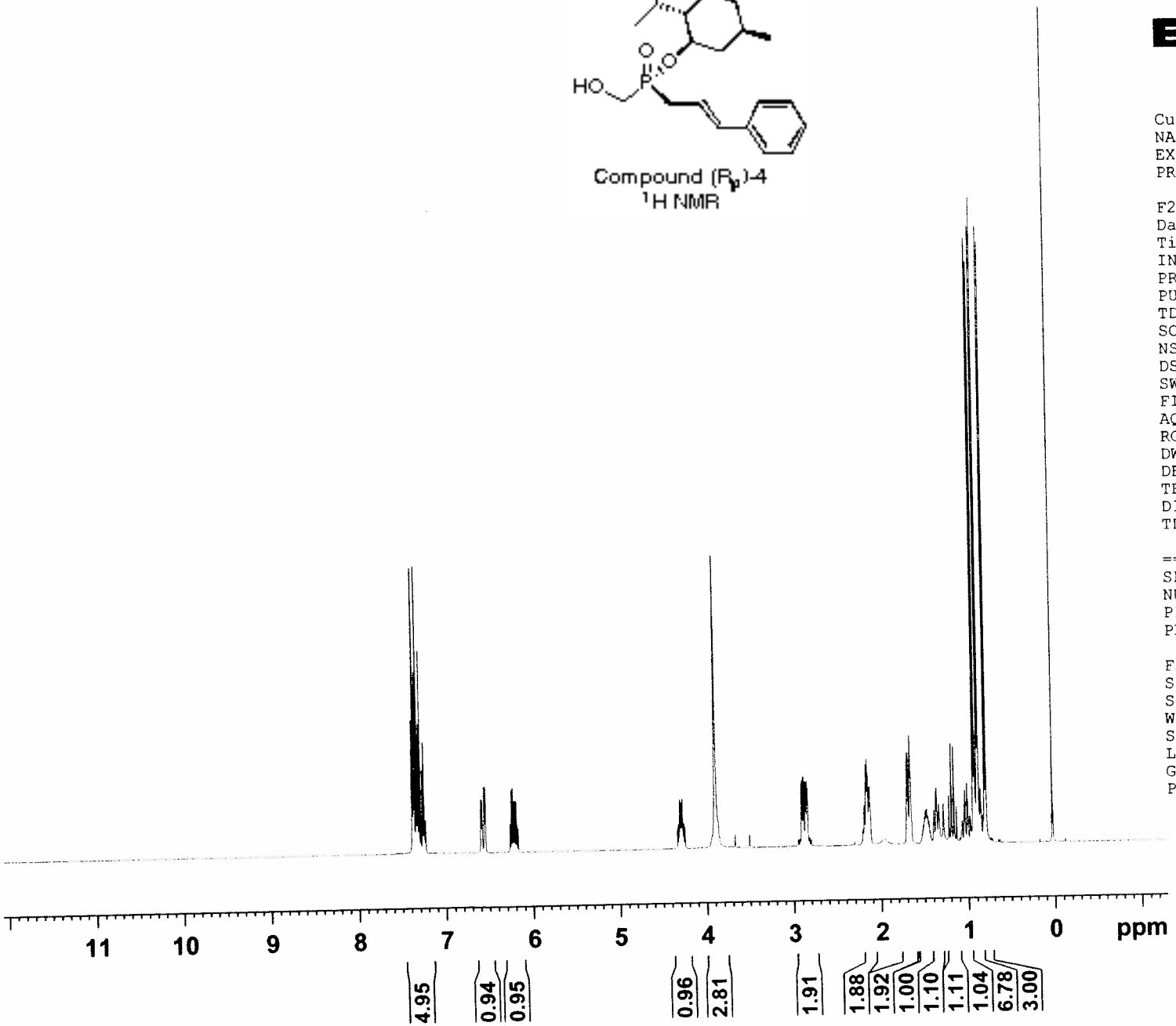




Compound (R_p)-4
^{1}H NMR



BRUKER

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Current Data Parameters
NAME      OB 2157 after column
EXPNO                        3
PROCNO                       1

F2 - Acquisition Parameters
Date_              20150828
Time                  18.16
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       9
DS                       2
SWH               8012.820 Hz
FIDRES               0.122266 Hz
AQ                4.0894465 sec
RG                   81.67
DW                   62.400 usec
DE                    6.50 usec
TE                   294.9 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                    1H
P1                   10.00 usec
PLW1          25.00300026 W

F2 - Processing parameters
SI                   65536
SF            400.1300000 MHz
WDW                     EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
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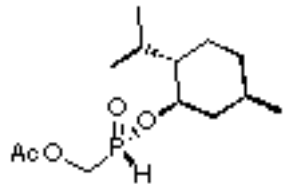
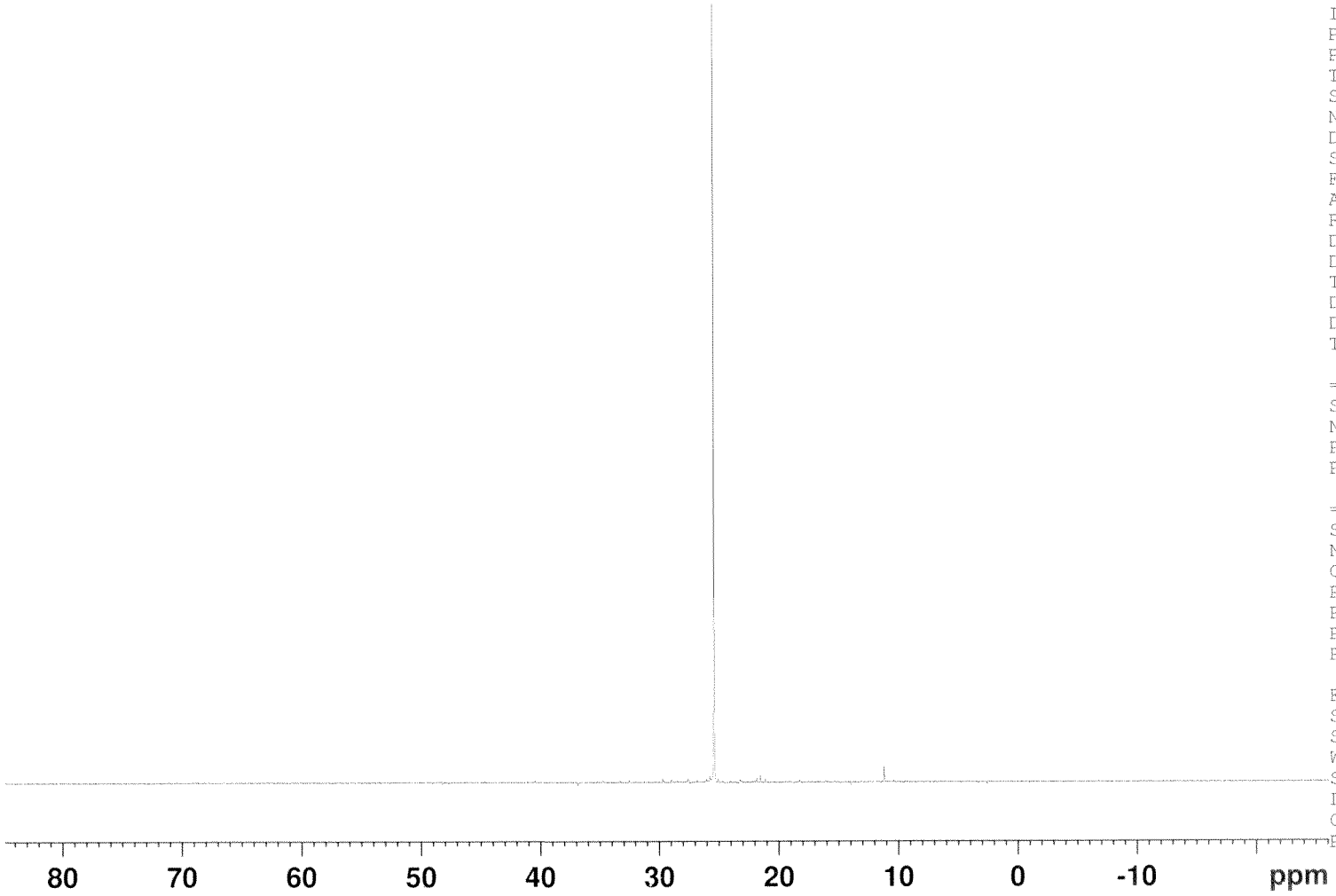


Compound (R_p)-7
$^{31}P\{^{1}H\}$ NMR decoupled

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Current Data Parameters
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EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
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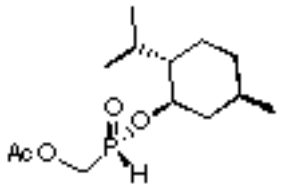
Time                   11.14
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT             Acetone
NS                         16
DS                          4
SWH               64102.563 Hz
FIDRES             0.978127 Hz
AQ                0.5111808 sec
RG                    203.57
DW                   7.800 usec
DE                    6.50 usec
TE                   297.4 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                         1

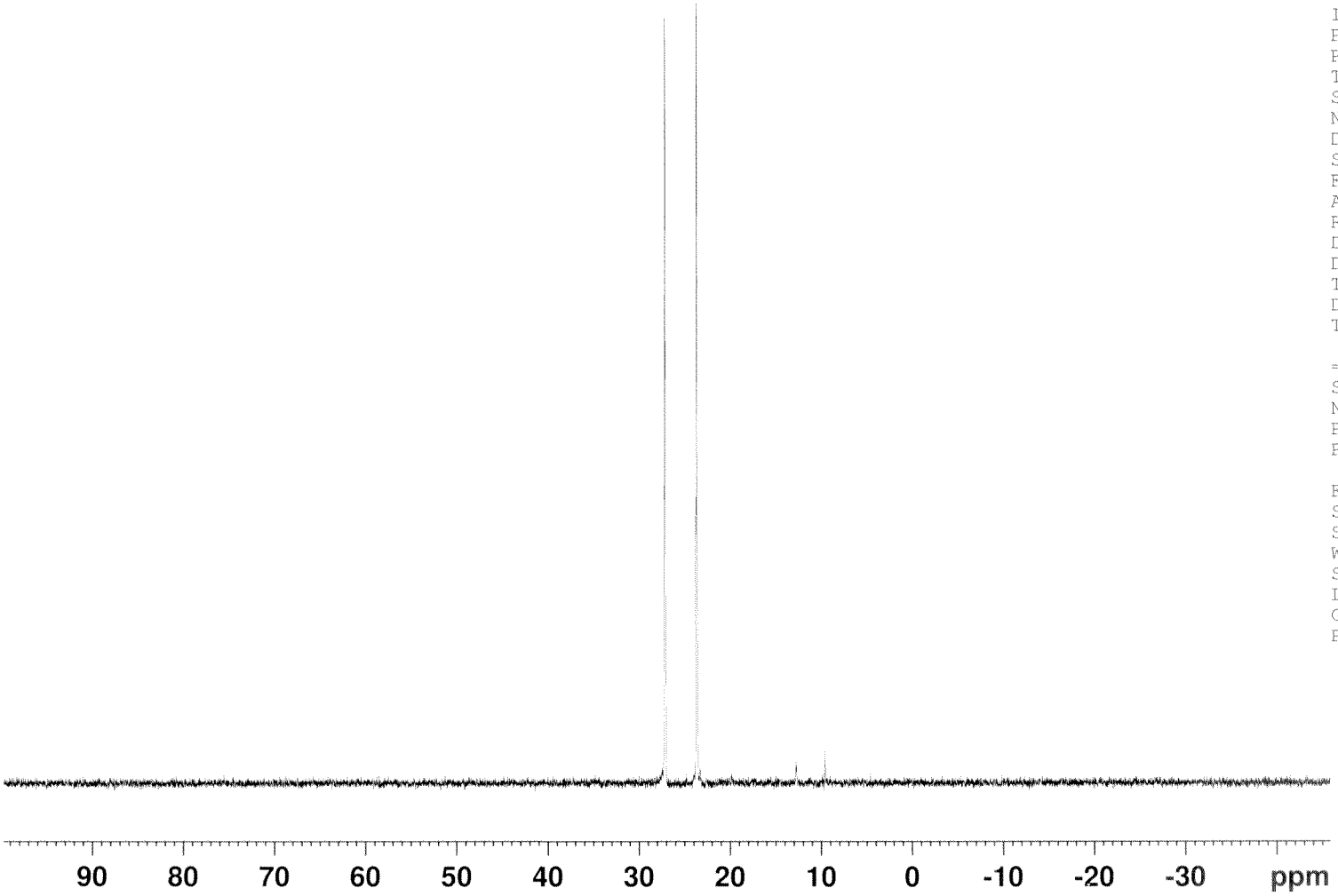
======== CHANNEL f1 ========
SFO1           161.9674942 MHz
NUC1                    31P
P1                    14.25 usec
PLW1           15.00000000 W

======== CHANNEL f2 ========
SFO2           400.1316005 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                 90.00 usec
PLW2           10.00000000 W
PLW12           0.31604999 W
PLW13           0.25600001 W

F2 - Processing parameters
SI                      32768
SF             161.9755930 MHz
WDW                        EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.40
```



Compound (R_p)-7
$^{31}P/^{1}H$ NMR coupled

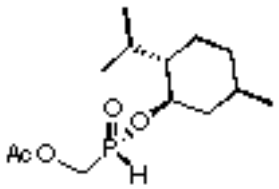



```
Current Data Parameters
NAME                  OB 1904
EXPNO                       2
PROCNO                      1

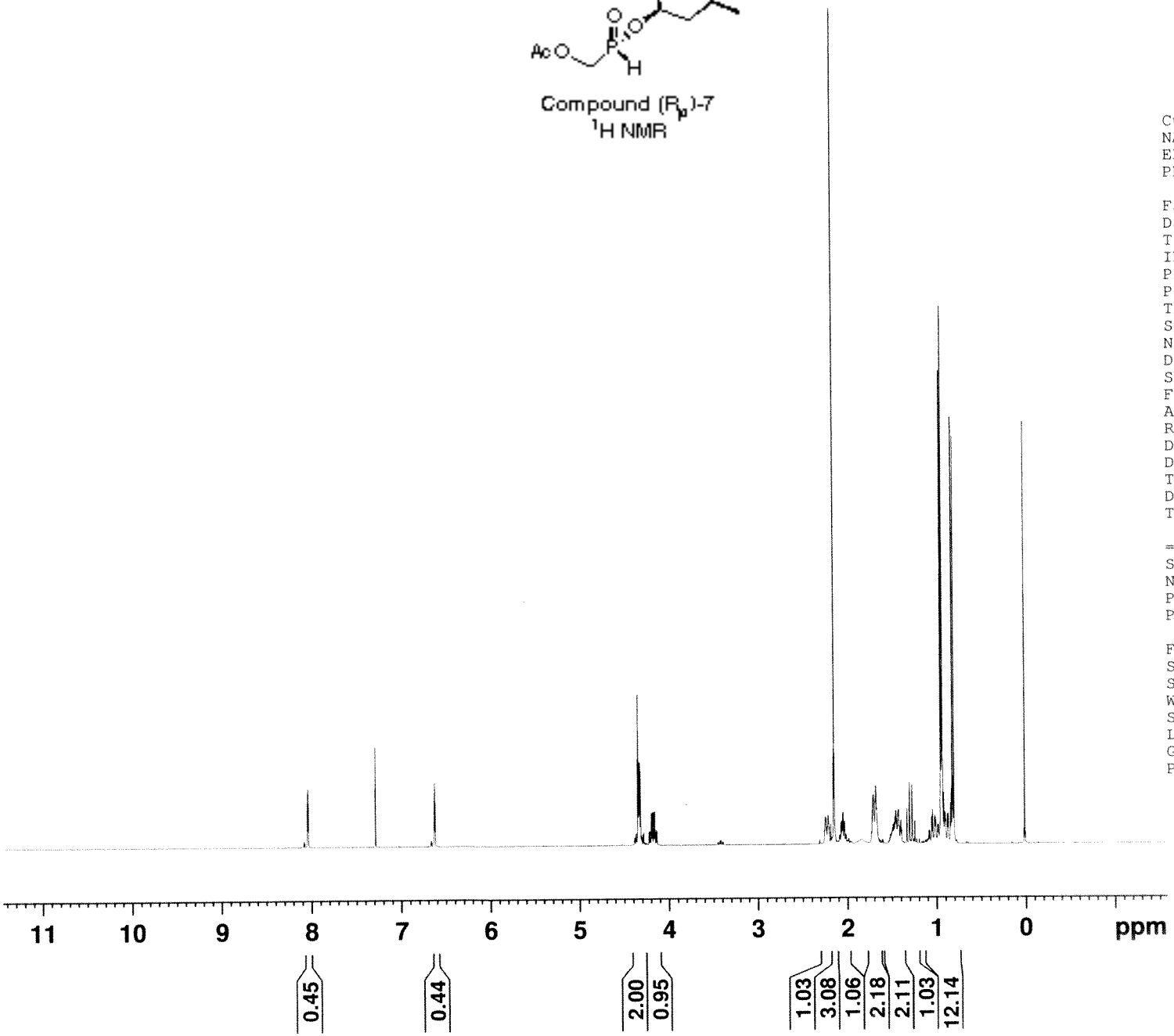
F2 - Acquisition Parameters
Date_                20150121
Time                    11.16
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT              Acetone
NS                          32
DS                           4
SWH                64102.563 Hz
FIDRES              0.978127 Hz
AQ                  0.5111808 sec
RG                     203.57
DW                      7.800 usec
DE                      6.50 usec
TE                     297.0 K
D1                2.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             161.9674942 MHz
NUC1                    31P
P1                     14.25 usec
PLW1             15.00000000 W

F2 - Processing parameters
SI                      32768
SF               161.9755930 MHz
WDW                        EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```



Compound (R_p)-7
^{1}H NMR

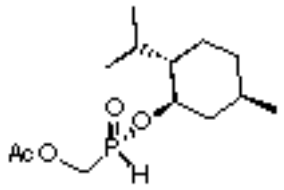



```
Current Data Parameters
NAME      OB 1781 after work-up
EXPNO                          3
PROCNO                         1

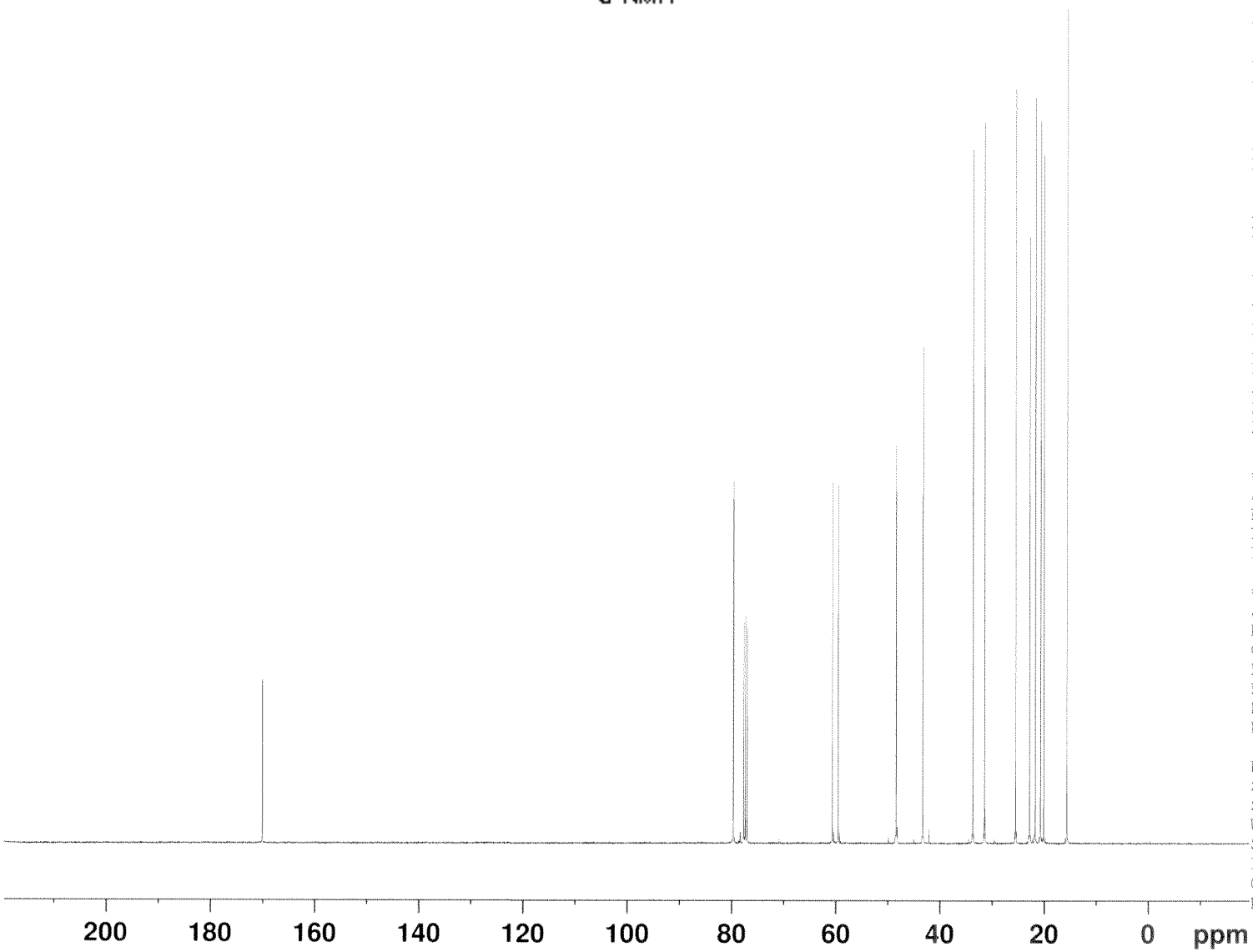
F2 - Acquisition Parameters
Date_               20141015
Time                   18.09
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        7
DS                        2
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894465 sec
RG                     81.67
DW                    62.400 usec
DE                     6.50 usec
TE                    294.9 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                      1H
P1                    10.00 usec
PLW1           25.00300026 W

F2 - Processing parameters
SI                    65536
SF              400.1300000 MHz
WDW                      EM
SSB       0
LB                     0.30 Hz
GB        0
PC                     1.00
```



Compound (R_p)-7
^{13}C NMR



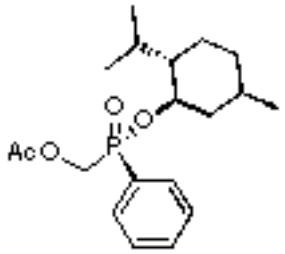

```
Current Data Parameters
NAME                  OB 1169
EXPNO                       4
PROCNO                      1

F2 - Acquisition Parameters
Date_               20130712
Time                   18.02
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      857
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631488 sec
RG                   203.57
DW                   20.800 usec
DE                    6.50 usec
TE                   295.2 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

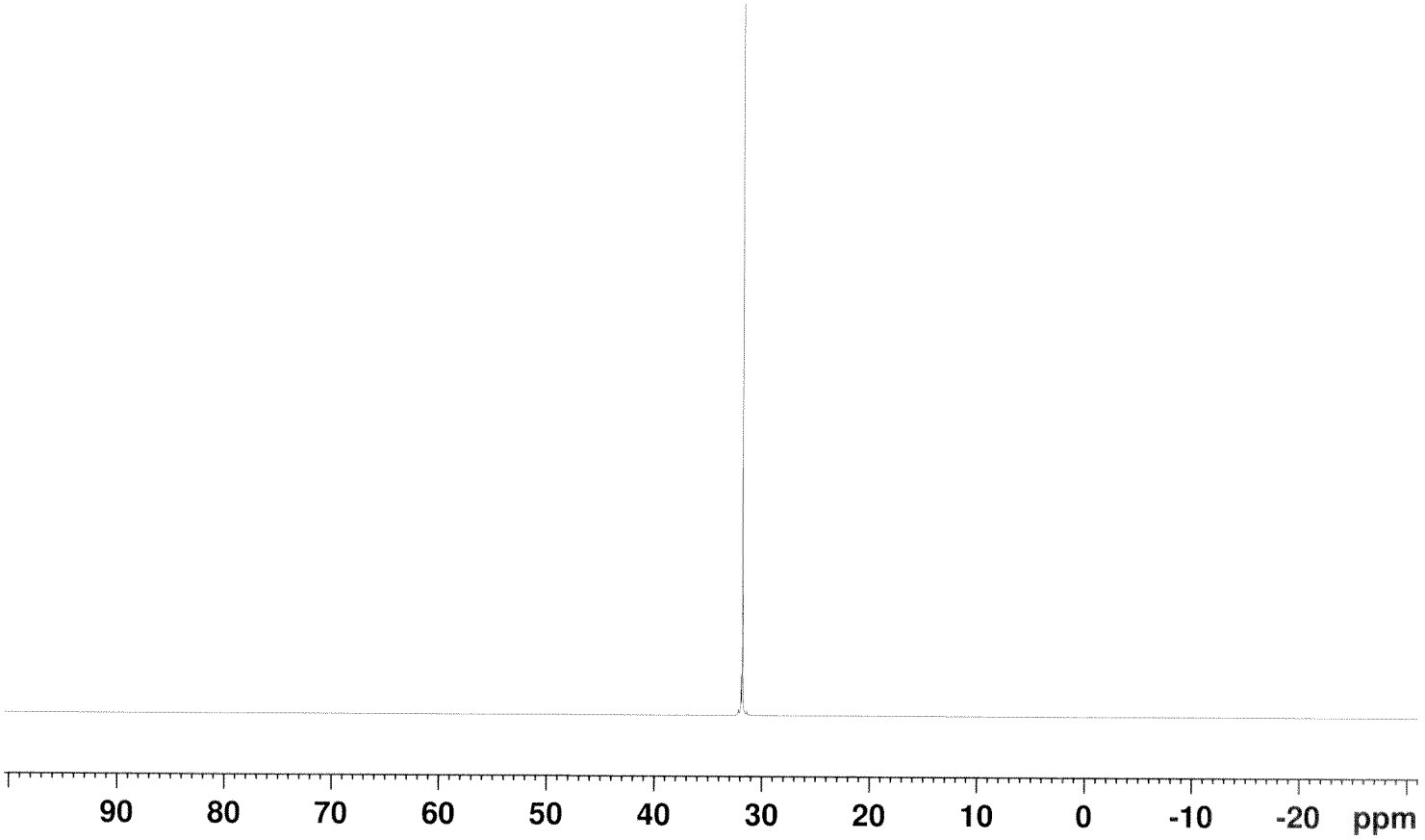
======== CHANNEL f1 ========
SFO1            100.6228293 MHz
NUC1                    13C
P1                   10.00 usec
PLW1           45.00000000 W

======== CHANNEL f2 ========
SFO2            400.1316005 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                90.00 usec
PLW2           10.00000000 W
PLW12           0.31604999 W
PLW13           0.25600001 W

F2 - Processing parameters
SI                      32768
SF              100.6127690 MHz
WDW                        EM
SSB         0
LB                    1.00 Hz
GB          0
PC                    1.40
```



Compound (R_p)-3a
$^{31}P\{^1H\}$ NMR decoupled



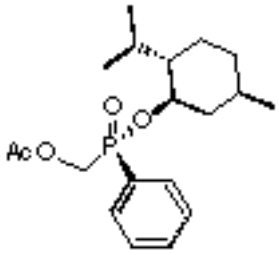

```
Current Data Parameters
NAME        OB 1379 pure
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140320
Time               18.02
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE                296.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

========== CHANNEL f1 =========
SFO1        161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1        15.00000000 W

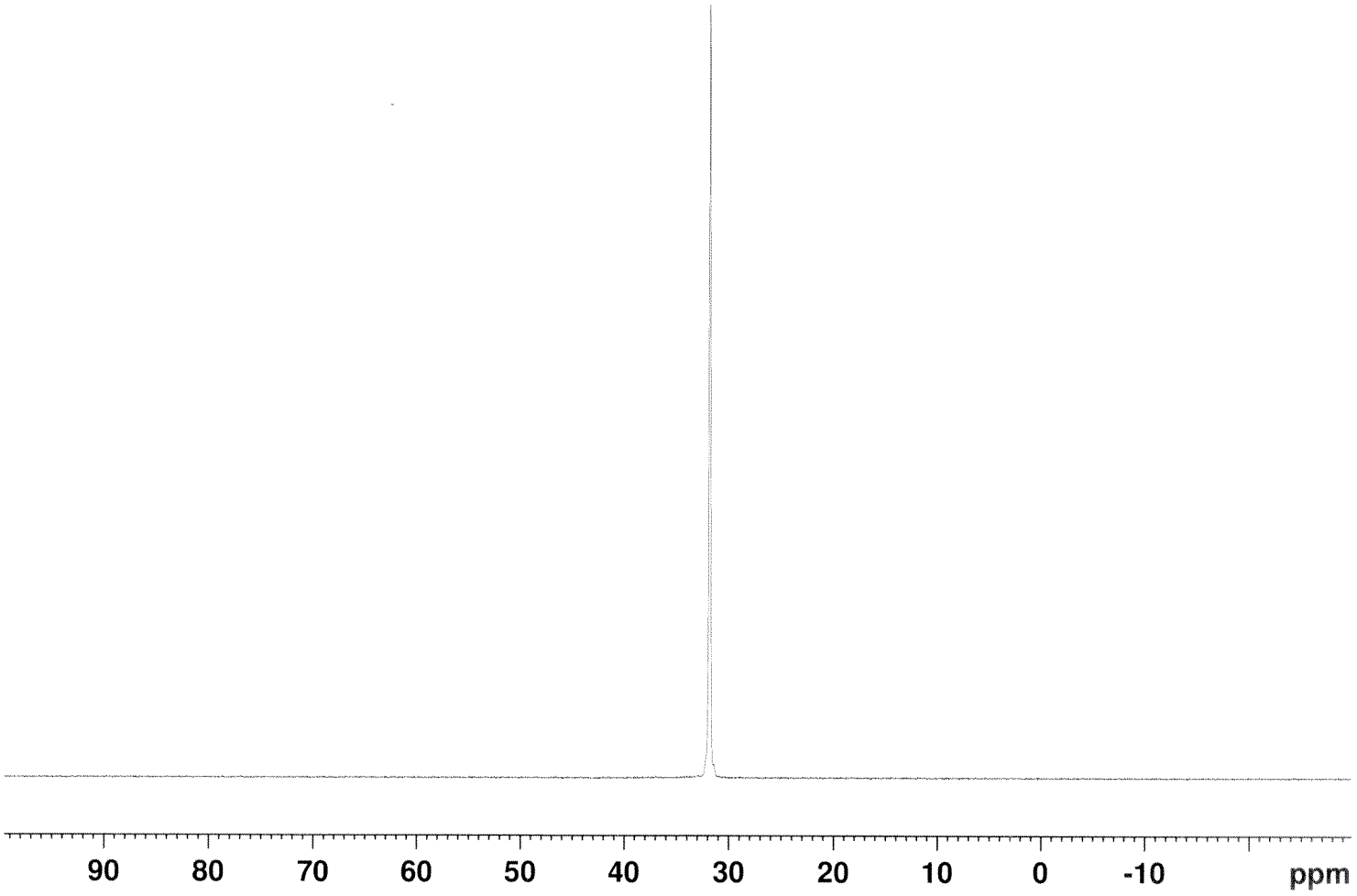
========== CHANNEL f2 ==========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                 32768
SF          161.9755930 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



Compound (R_p)-3a

$^{31}P\{^1H\}$ NMR coupled

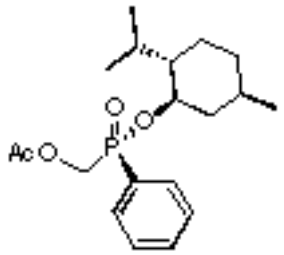



```
Current Data Parameters
NAME        OB 1379 pure
EXPNO                  2
PROCNO                 1

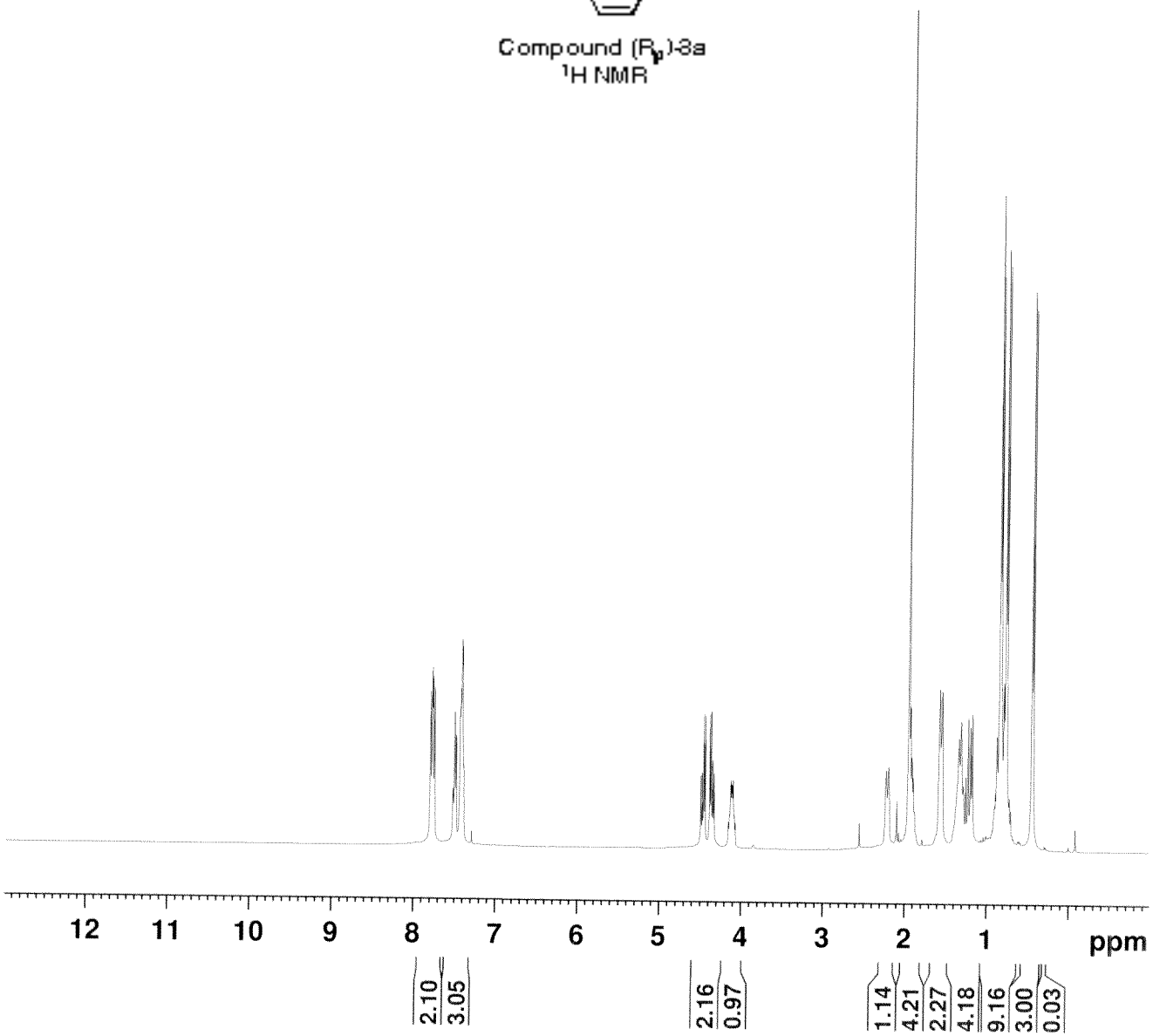
F2 - Acquisition Parameters
Date_           20140320
Time               18.06
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE               295.7 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1               31P
P1               14.25 usec
PLW1        15.00000000 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



Compound (R_p)-3a
^{1}H NMR

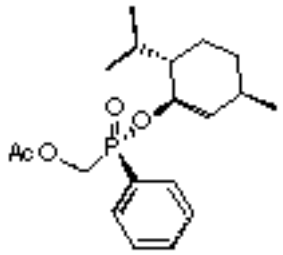



```
Current Data Parameters
NAME        OB 1379 pure
EXPNO                  3
PROCNO                 1

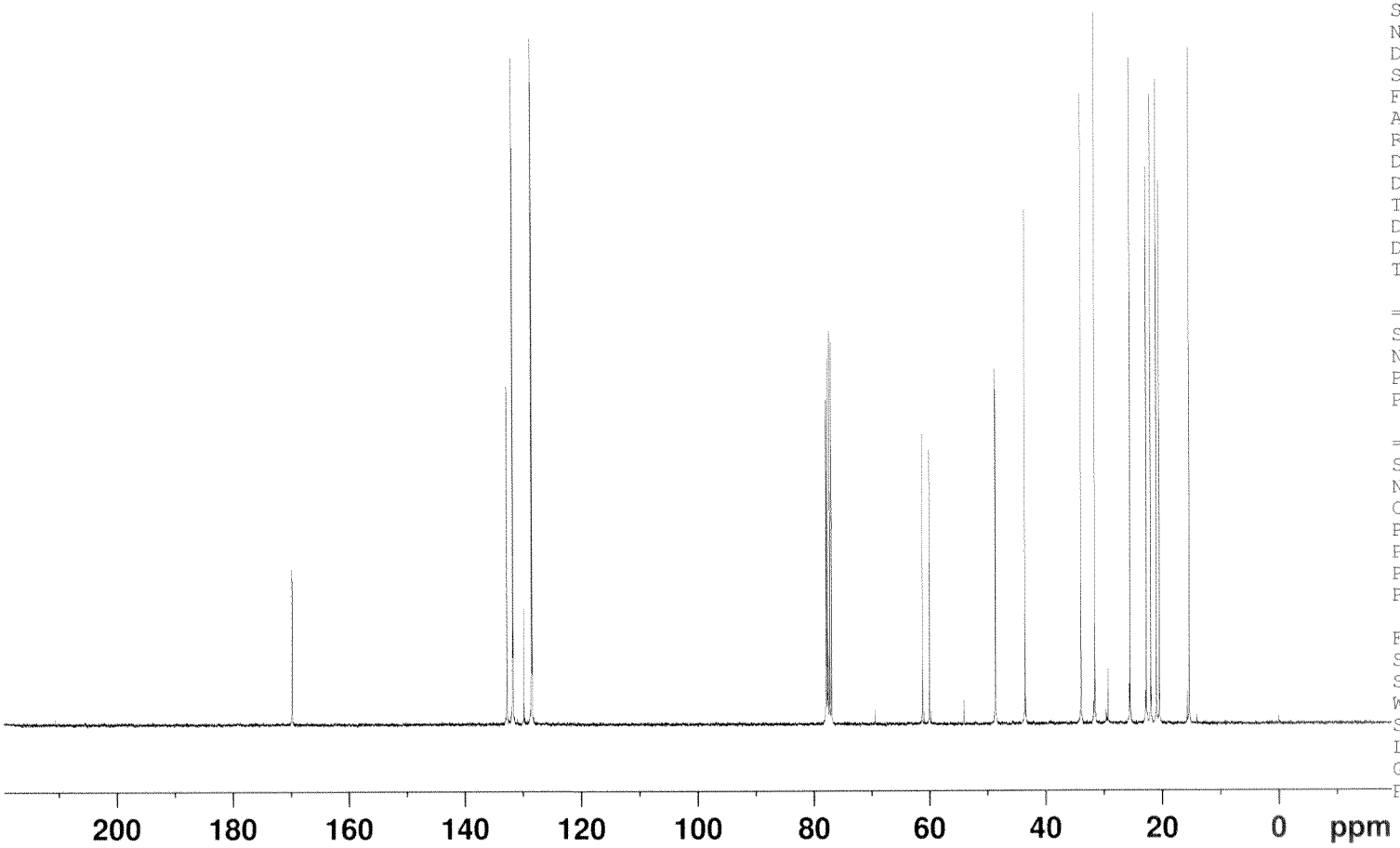
F2 - Acquisition Parameters
Date_           20140320
Time               18.09
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           8012.820 Hz
FIDRES        0.122266 Hz
AQ           4.0894465 sec
RG                12.96
DW               62.400 usec
DE                6.50 usec
TE               295.6 K
D1          1.00000000 sec
TD0                   1

========== CHANNEL f1 ==========
SFO1        400.1324710 MHz
NUC1                  1H
P1                10.00 usec
PLW1        25.00300026 W

F2 - Processing parameters
SI                65536
SF          400.1300000 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



Compound (R_p)-3a
^{13}C NMR



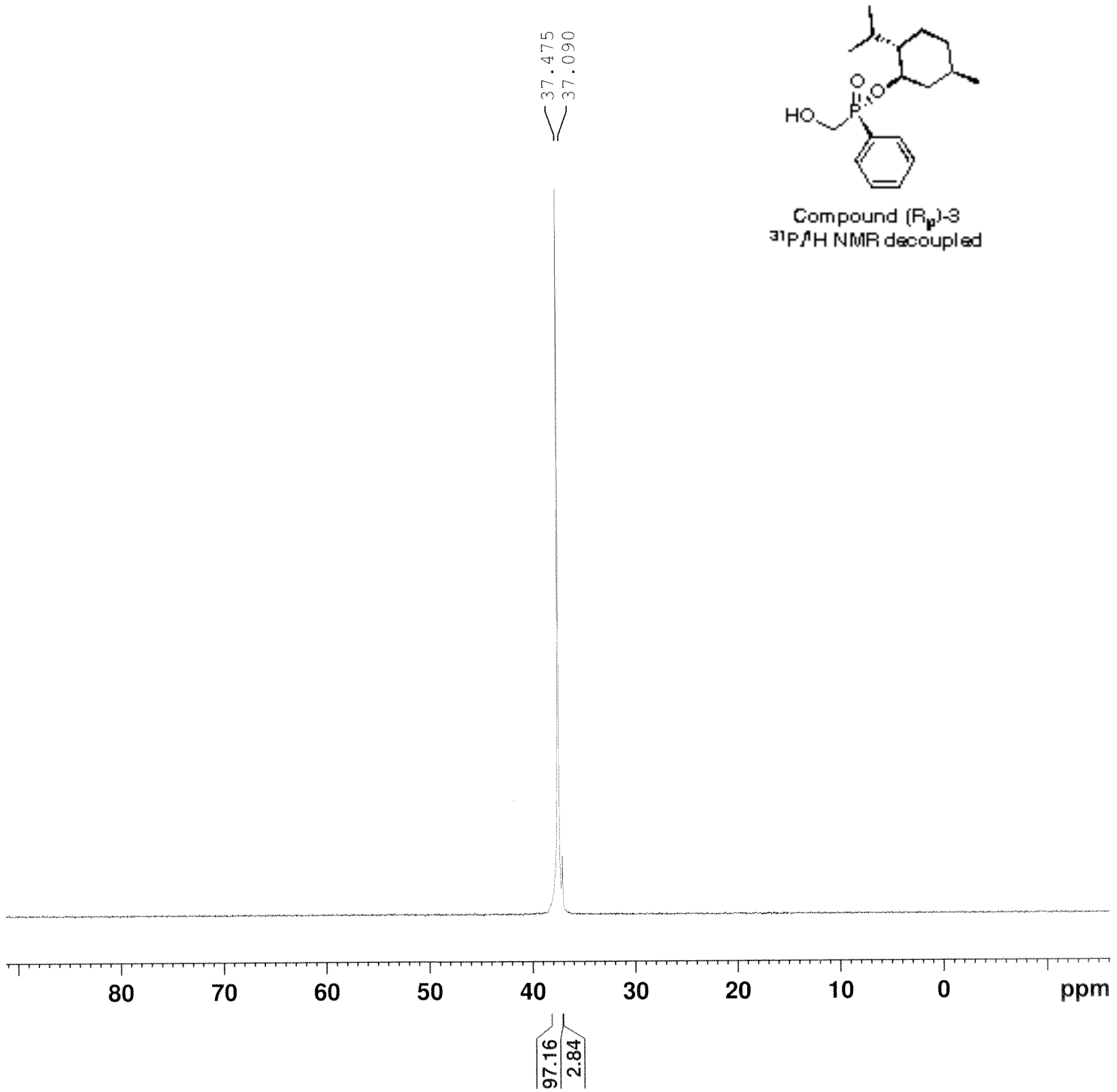

```
Current Data Parameters
NAME        OB 1379 pure
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140320
Time               18.52
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  733
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                296.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         45.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



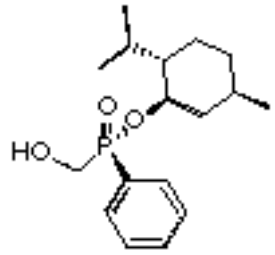

```
Current Data Parameters
NAME        OB 1438 pure
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140424
Time              17.03
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE                295.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

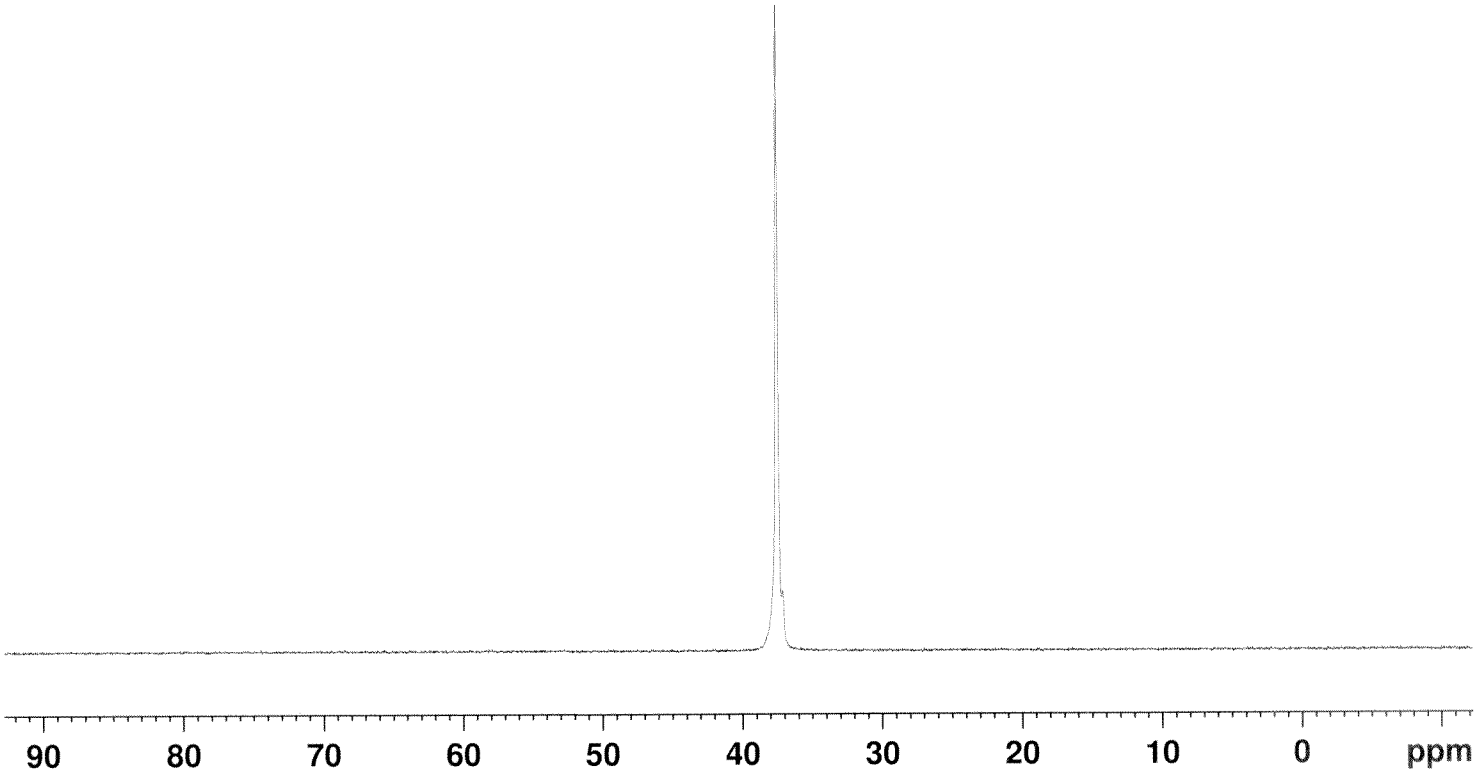
========== CHANNEL f1 =========
SFO1        161.9674942 MHz
NUC1               31P
P1                14.25 usec
PLW1         15.00000000 W

========== CHANNEL f2 =========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



Compound (R_p)-3
$^{31}P\{^1H\}$ NMR coupled

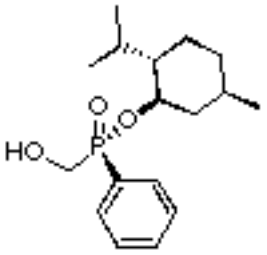



```
Current Data Parameters
NAME        OB 1438 pure
EXPNO                  2
PROCNO                 1

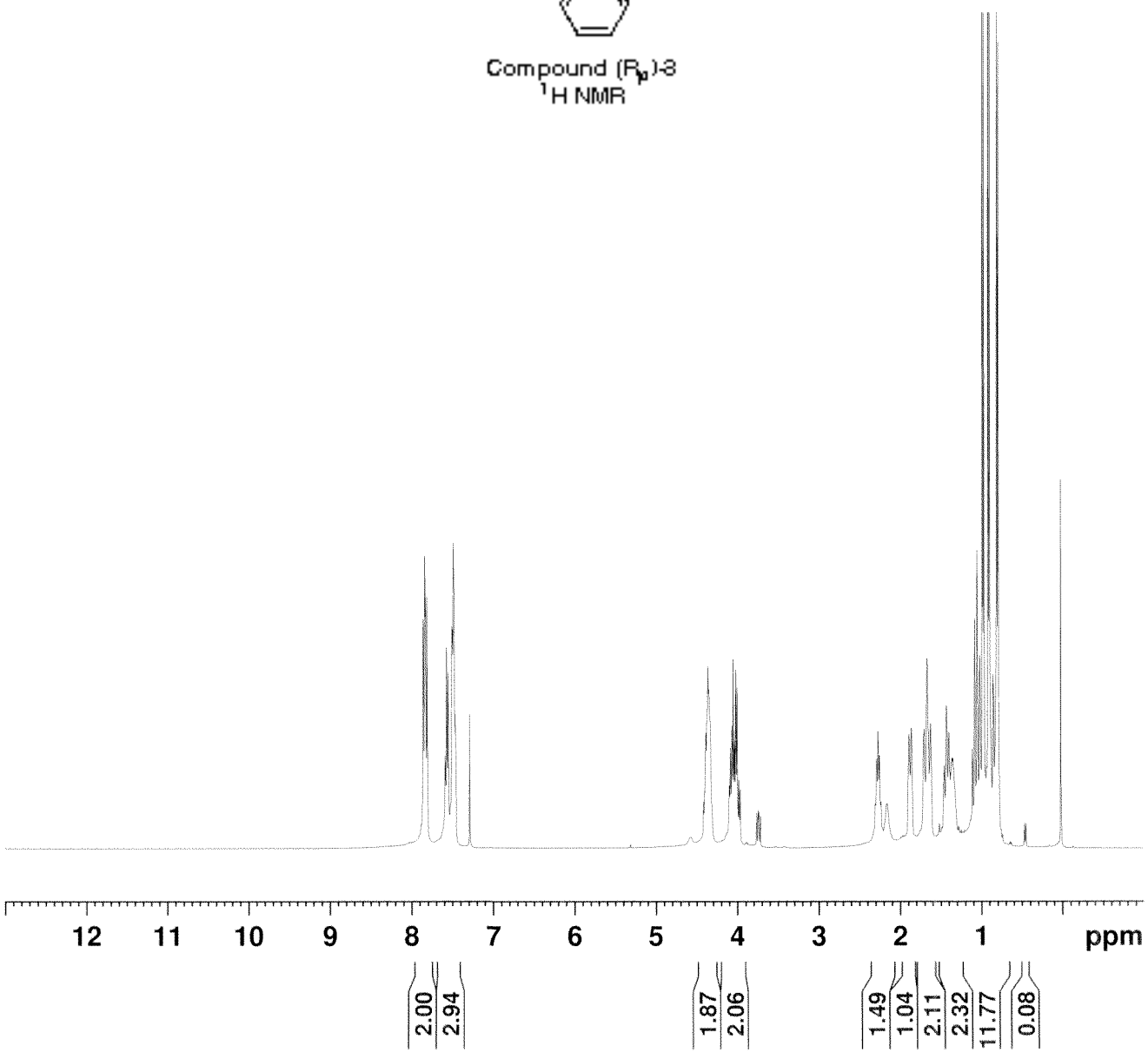
F2 - Acquisition Parameters
Date_           20140424
Time               17.05
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDC13
NS                   32
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE               295.2 K
D1           2.00000000 sec
TD0                    1

========== CHANNEL f1 ==========
SFO1         161.9674942 MHz
NUC1                 31P
P1                14.25 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 32768
SF           161.9755930 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



Compound (R_p)-3
^{1}H NMR

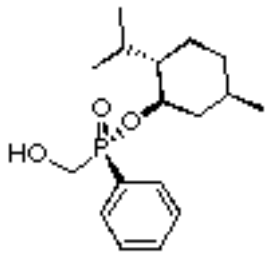



```
Current Data Parameters
NAME      OB 1387 after precipitation
EXPNO                 3
PROCNO                1

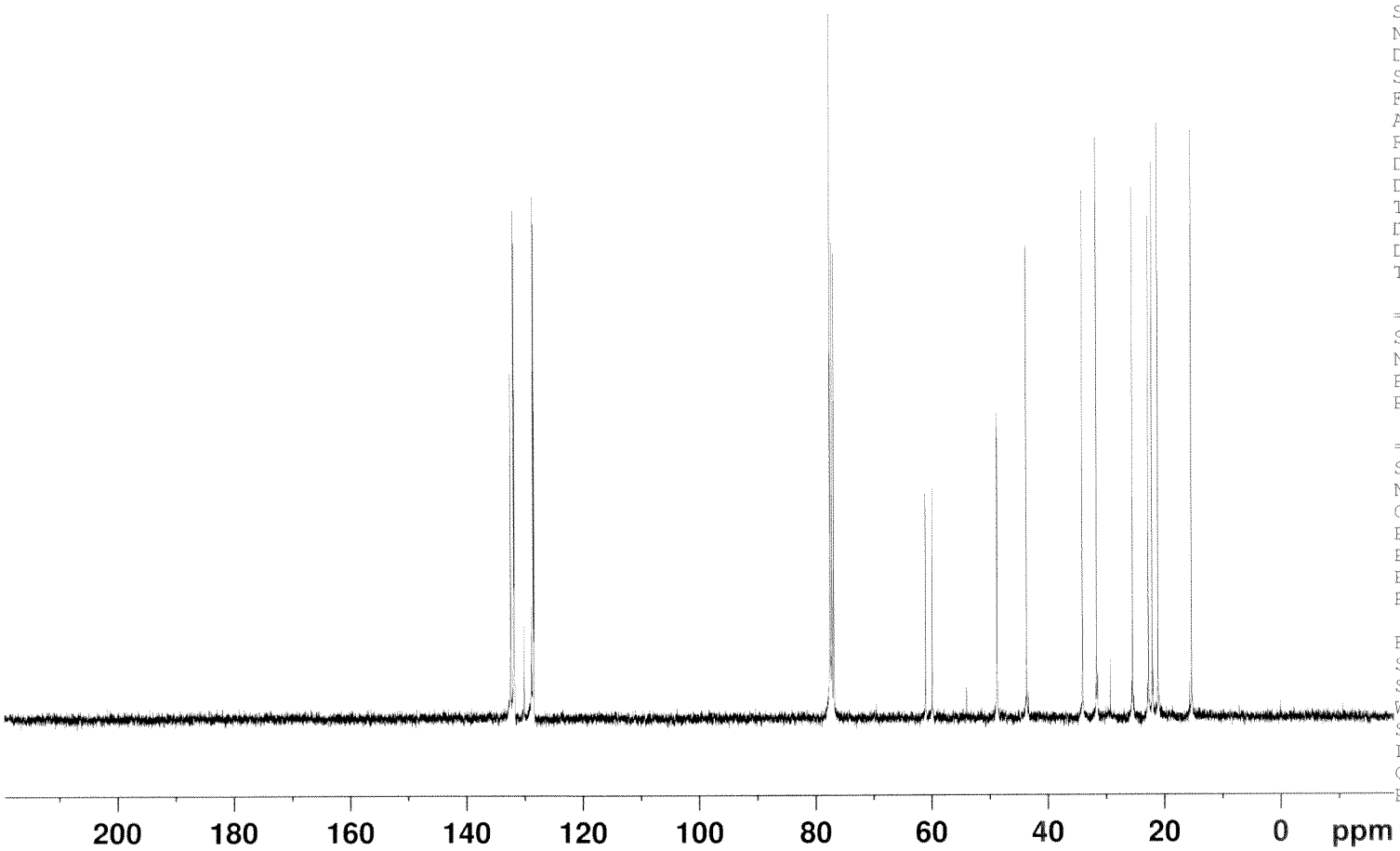
F2 - Acquisition Parameters
Date_          20140328
Time              14.26
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                66.4
DW              62.400 usec
DE               6.50 usec
TE              297.1 K
D1           1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1               1H
P1               10.00 usec
PLW1         25.00300026 W

F2 - Processing parameters
SI               65536
SF           400.1300000 MHz
WDW                EM
SSB       0
LB               0.30 Hz
GB        0
PC               1.00
```



Compound (R_p)-3
^{13}C NMR



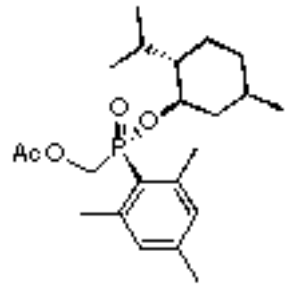

```
Current Data Parameters
NAME        OB 1438 pure
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140424
Time               17.01
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDC13
NS                  300
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                296.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         45.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



Compound (R_p)-8a

$^{31}P\{^1H\}$ NMR decoupled

BRUKER

Current Data Parameters

NAME	OB 1846 after column
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20141121
Time	9.22
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	4
SWH	64102.563 Hz
FIDRES	0.978127 Hz
AQ	0.5111808 sec
RG	203.57
DW	7.800 usec
DE	6.50 usec
TE	296.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

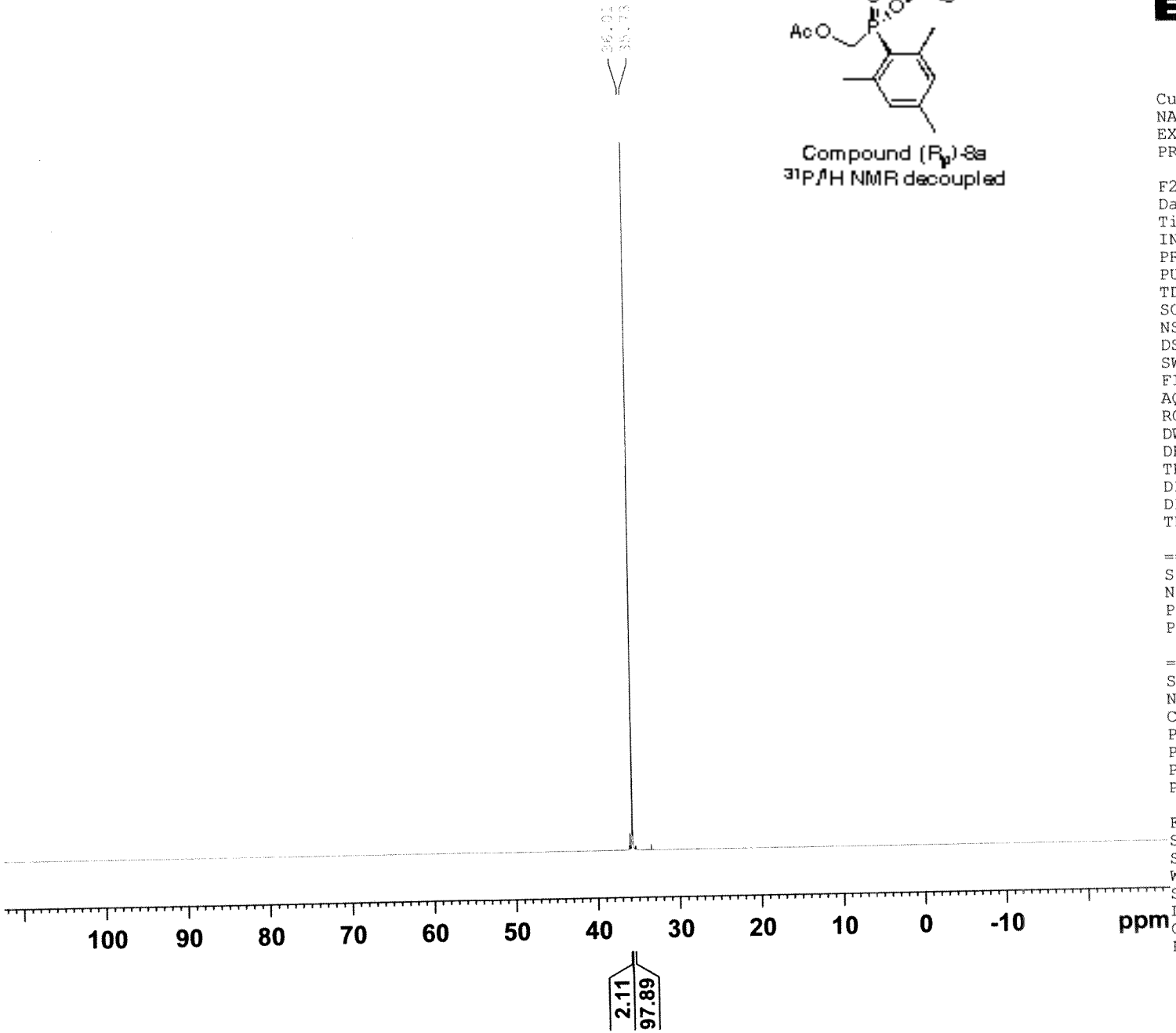
SFO1	161.9674942 MHz
NUC1	31P
P1	14.25 usec
PLW1	15.00000000 W

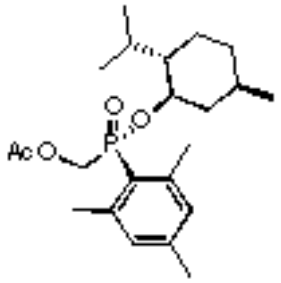
======== CHANNEL f2 ========

SFO2	400.1316005 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	90.00 usec
PLW2	10.00000000 W
PLW12	0.31604999 W
PLW13	0.25600001 W

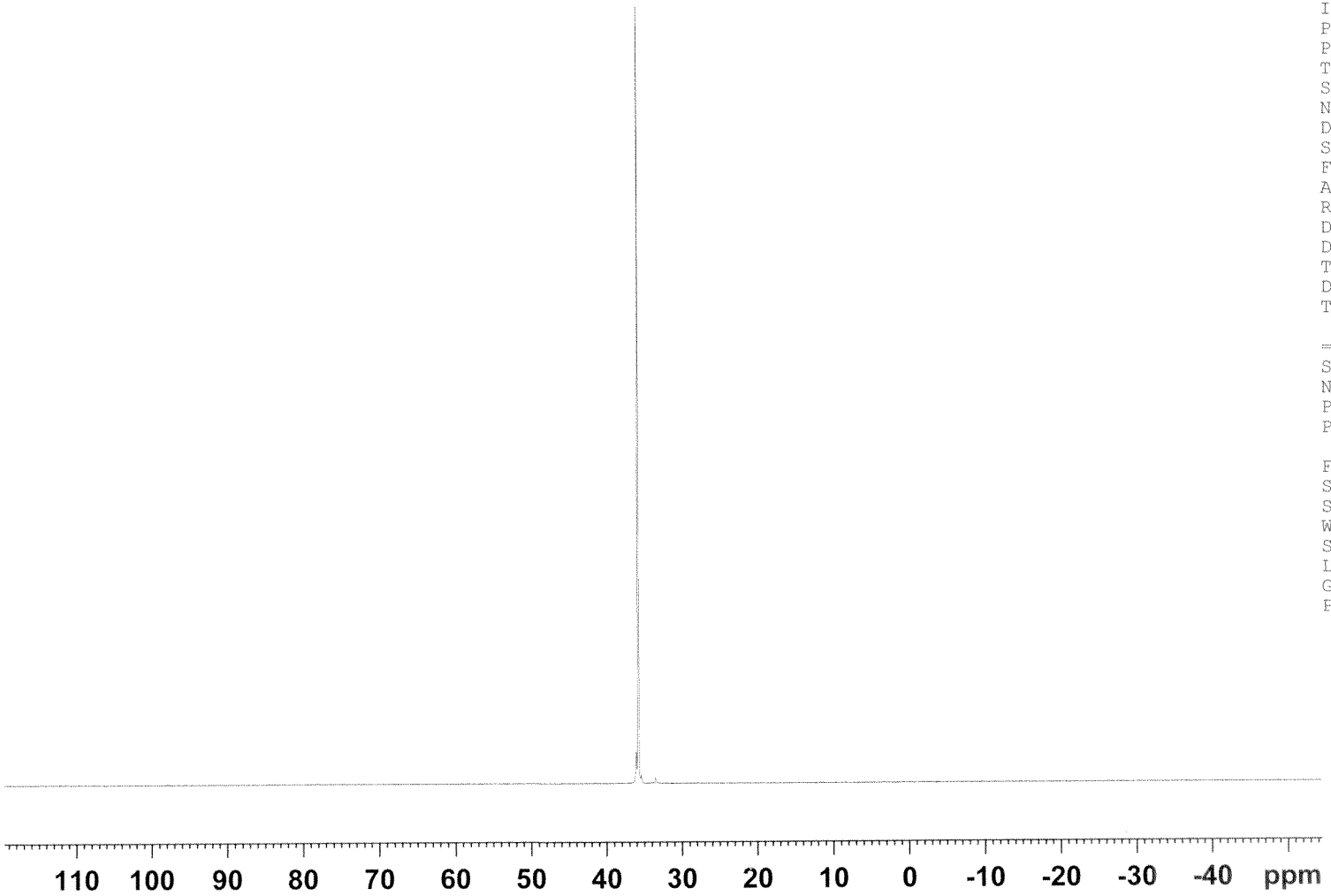
F2 - Processing parameters

SI	32768
SF	161.9755930 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40





Compound (R_p)-8a
$^{31}P\{^1H\}$ NMR coupled



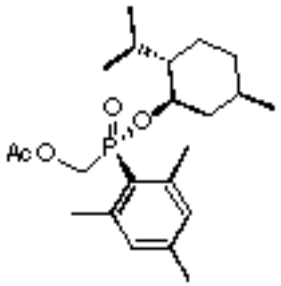
BRUKER

```
Current Data Parameters
NAME      OB 1843 after column
EXPNO                    2
PROCNO                   1

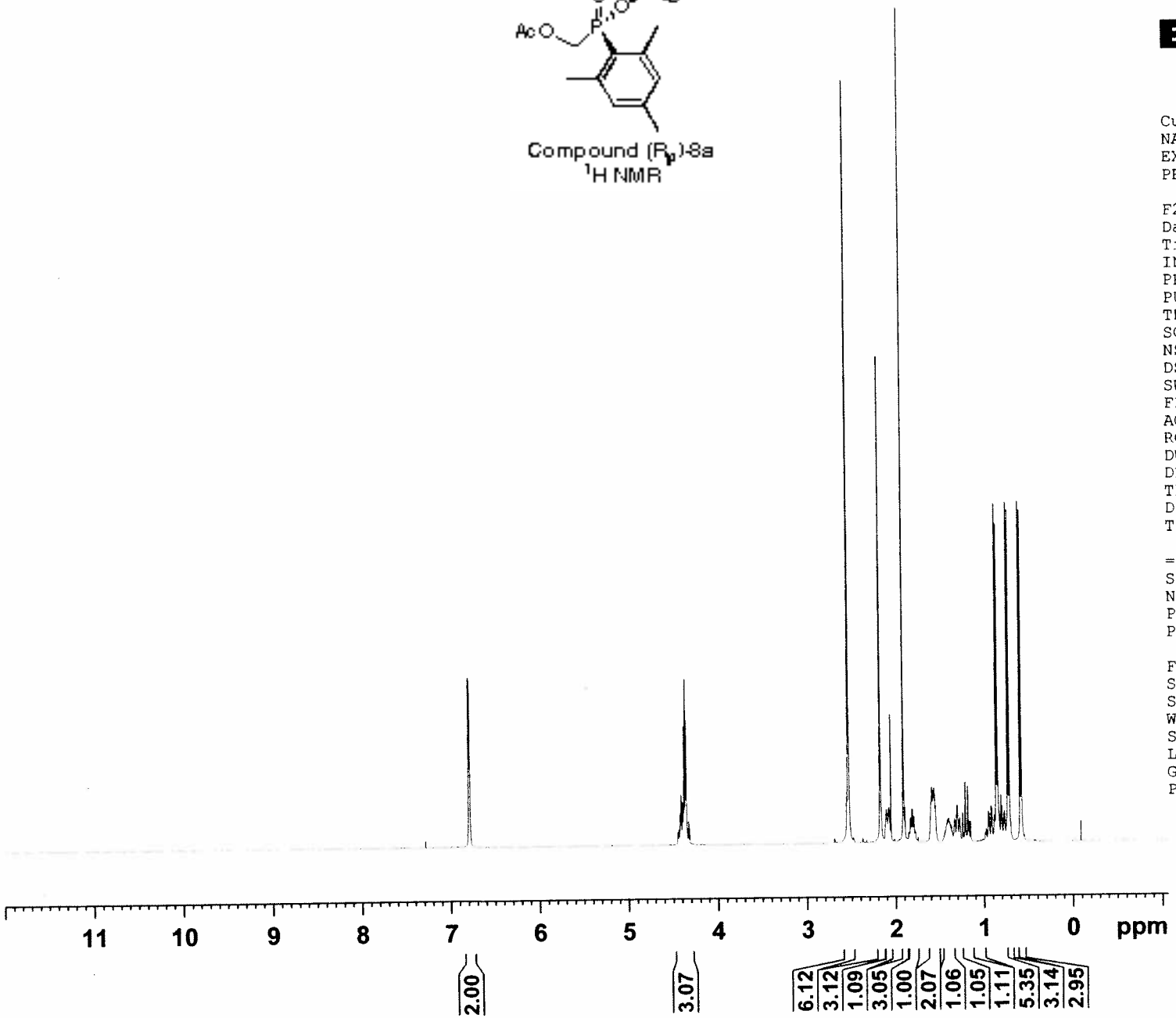
F2 - Acquisition Parameters
Date_           20141121
Time_              9.24
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE                296.1 K
D1           2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1               31P
P1               14.25 usec
PLW1        15.00000000 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



Compound (R_p)-8a
^{1}H NMR

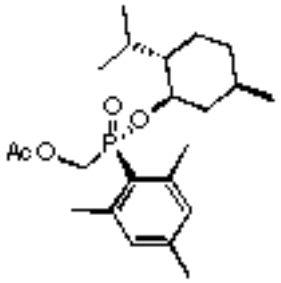


Current Data Parameters
NAME OB 1843 after column
EXPNO 3
PROCNO 1

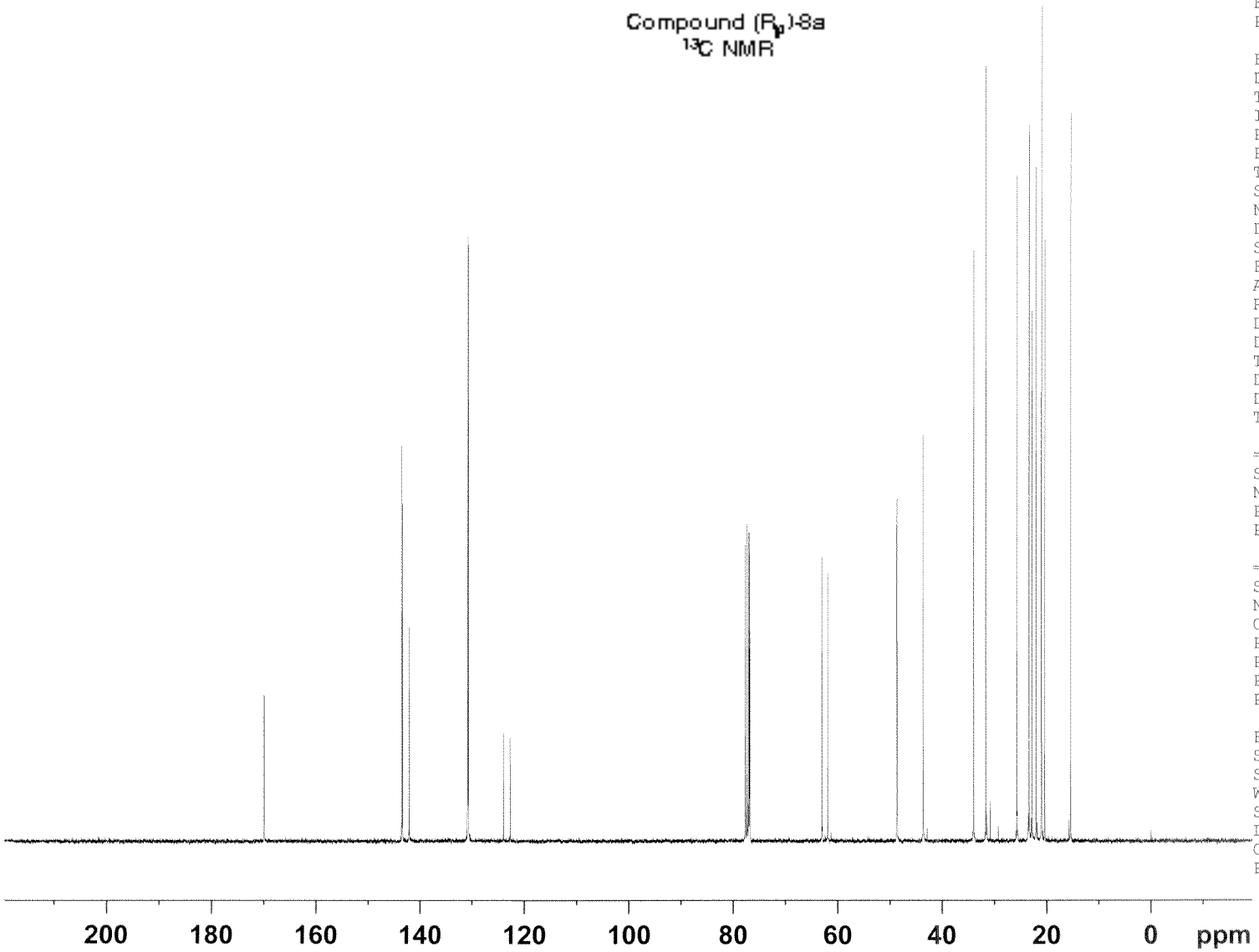
F2 - Acquisition Parameters
Date_ 20141121
Time 9.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 8.79
DW 62.400 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
PLW1 25.00300026 W

F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Compound (R_p)-8a
^{13}C NMR



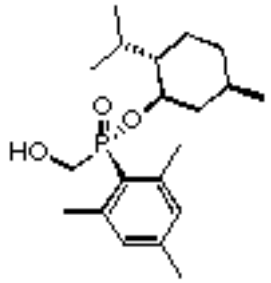

```
Current Data Parameters
NAME      OB 1843 after column
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_           20141121
Time                9.43
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   255
DS                     4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ              1.3631488 sec
RG                 203.57
DW                 20.800 usec
DE                  6.50 usec
TE                 297.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

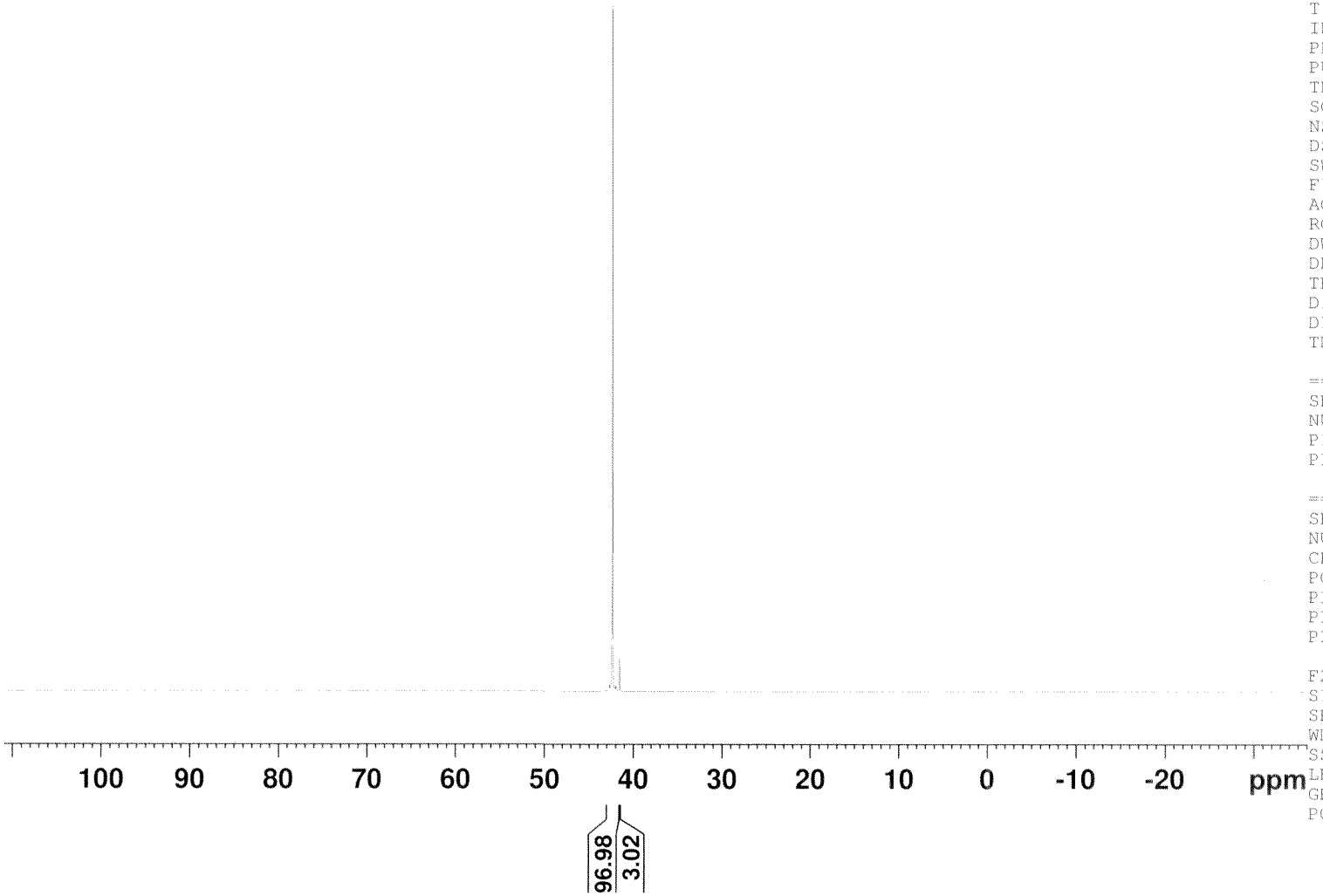
======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                 13C
P1                 10.00 usec
PLW1         45.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



Compound (R_P)-8b
$^{31}P/^{1}H$ NMR decoupled



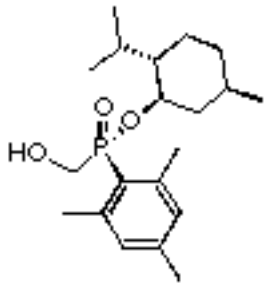

```
Current Data Parameters
NAME      OB 1857 after column
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20141203
Time               17.06
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE                298.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1        15.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



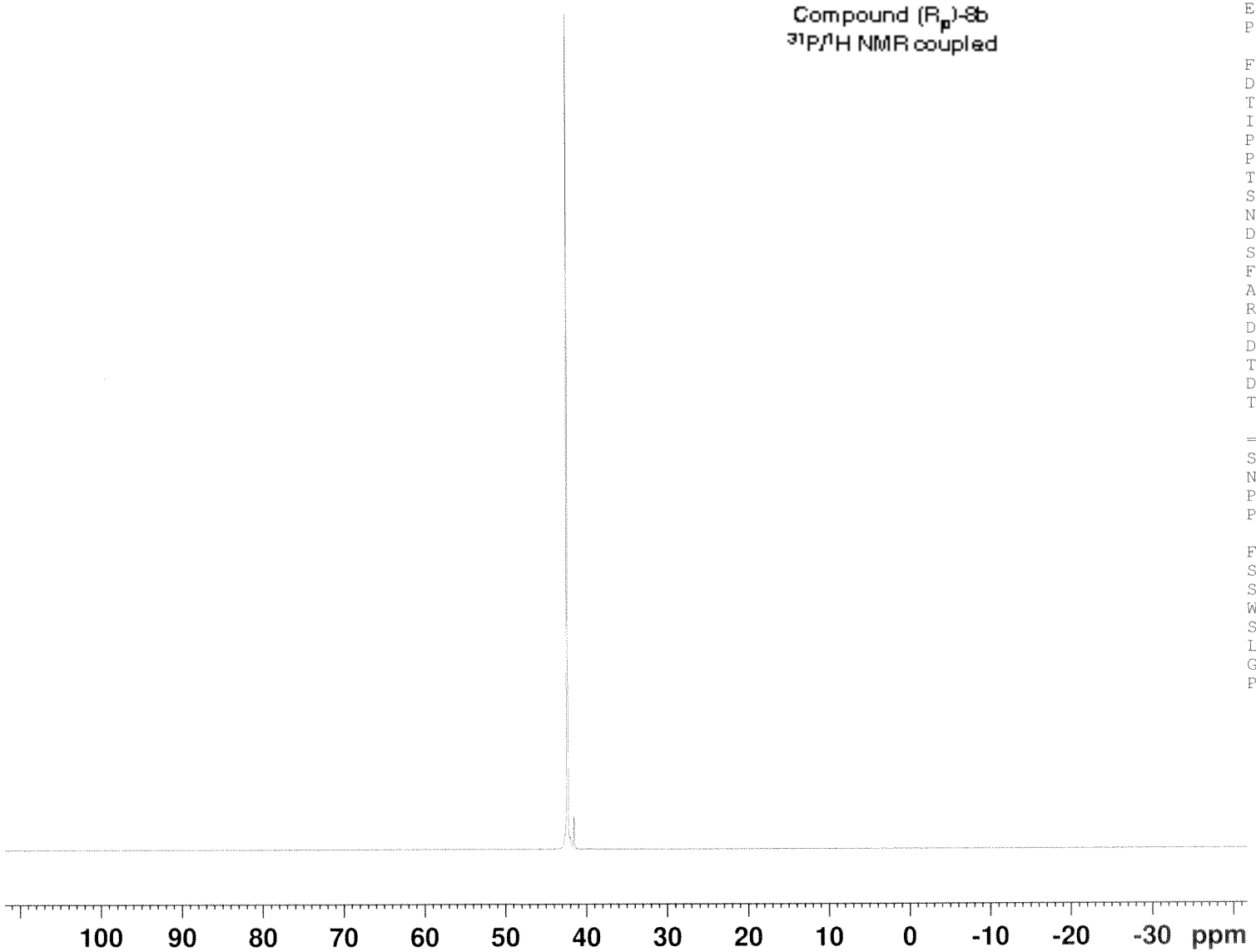
Compound (R_p)-8b
$^{31}P/^{1}H$ NMR coupled

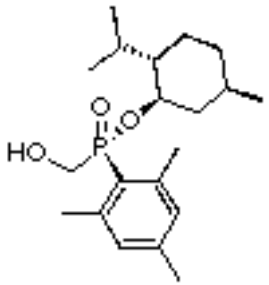
```
Current Data Parameters
NAME      OB 1857 after column
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20141203
Time               17.08
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH            64102.563 Hz
FIDRES          0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                 7.800 usec
DE                 6.50 usec
TE                 297.7 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 32768
SF           161.9755930 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```





Compound (R_p)-8b

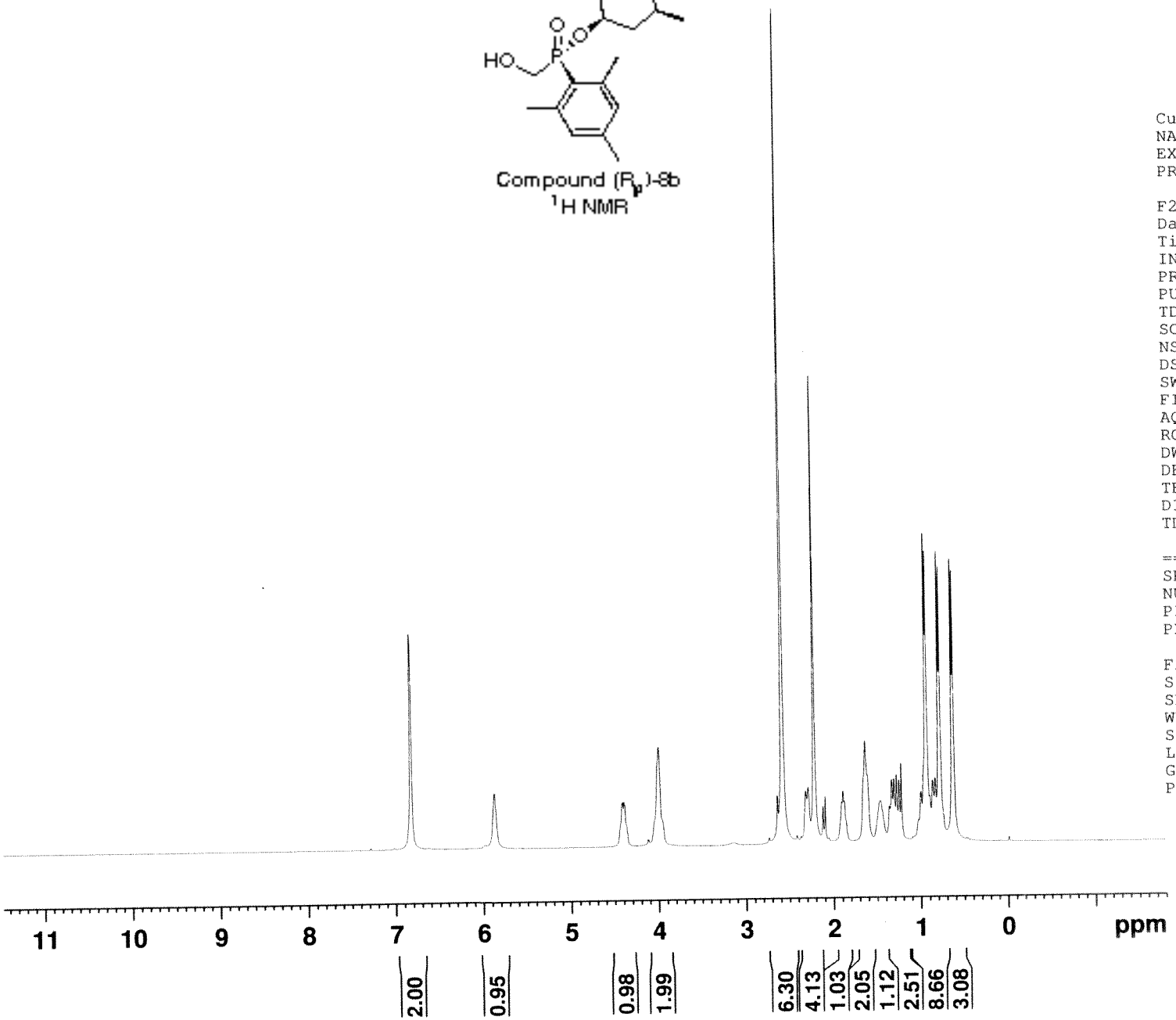
^{1}H NMR

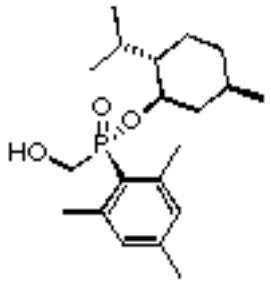
```
Current Data Parameters
NAME      OB 1857 after column
EXPNO                        3
PROCNO                       1

F2 - Acquisition Parameters
Date_              20141203
Time                  17.10
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        7
DS                        2
SWH                8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894465 sec
RG                      6.9
DW                   62.400 usec
DE                    6.50 usec
TE                   297.6 K
D1                1.00000000 sec
TD0                       1

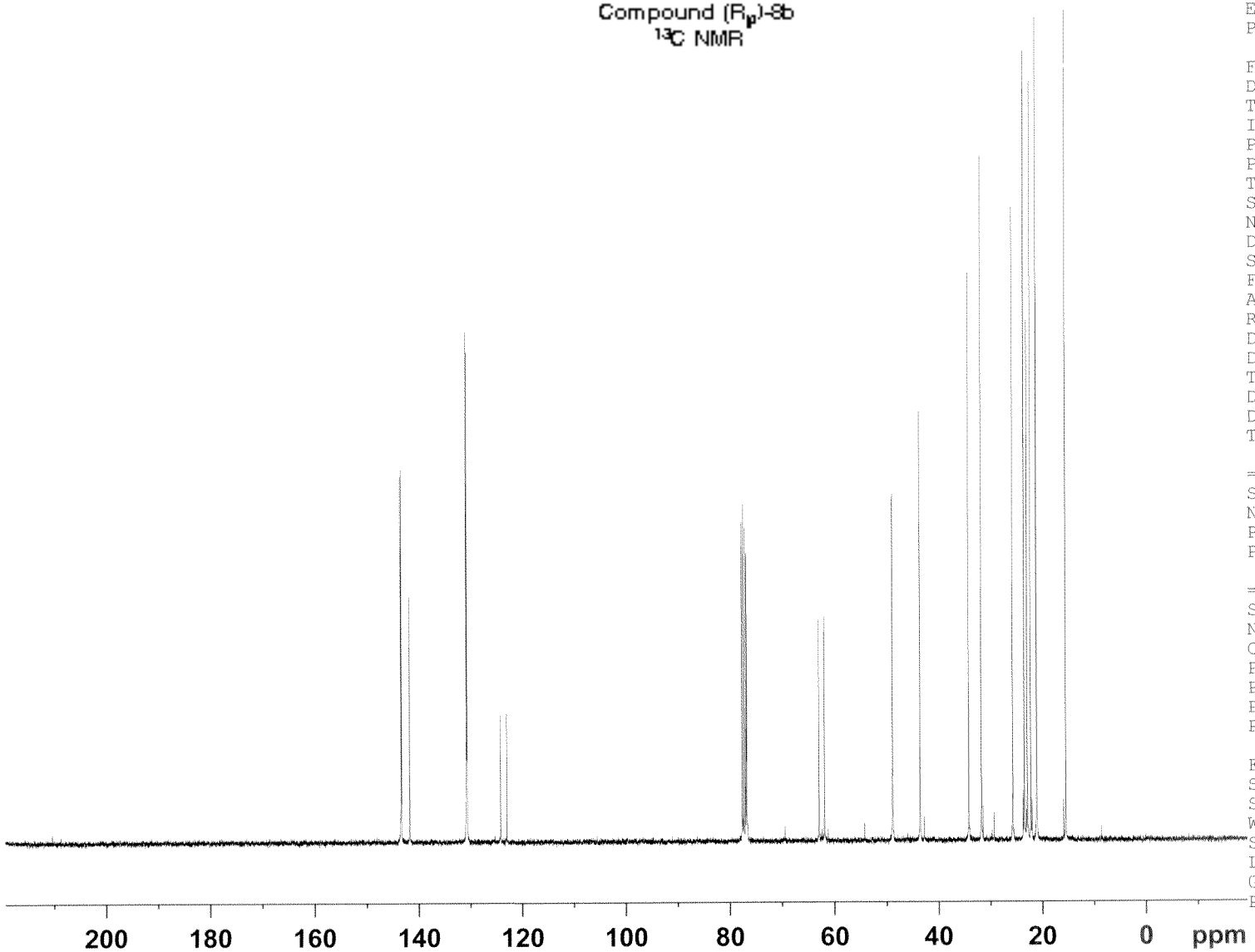
======== CHANNEL f1 ========
SFO1           400.1324710 MHz
NUC1                      1H
P1                    10.00 usec
PLW1           25.00300026 W

F2 - Processing parameters
SI                        65536
SF             400.1300000 MHz
WDW                       EM
SSB       0
LB                     0.30 Hz
GB        0
PC                     1.00
```





Compound (R_p)-8b
^{13}C NMR



BRUKER

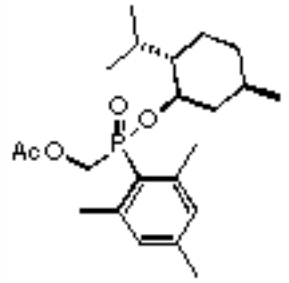
```
Current Data Parameters
NAME        OB 1844 pure
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150306
Time               17.58
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  204
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

========== CHANNEL f1 =========
SFO1         100.6228293 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         45.00000000 W

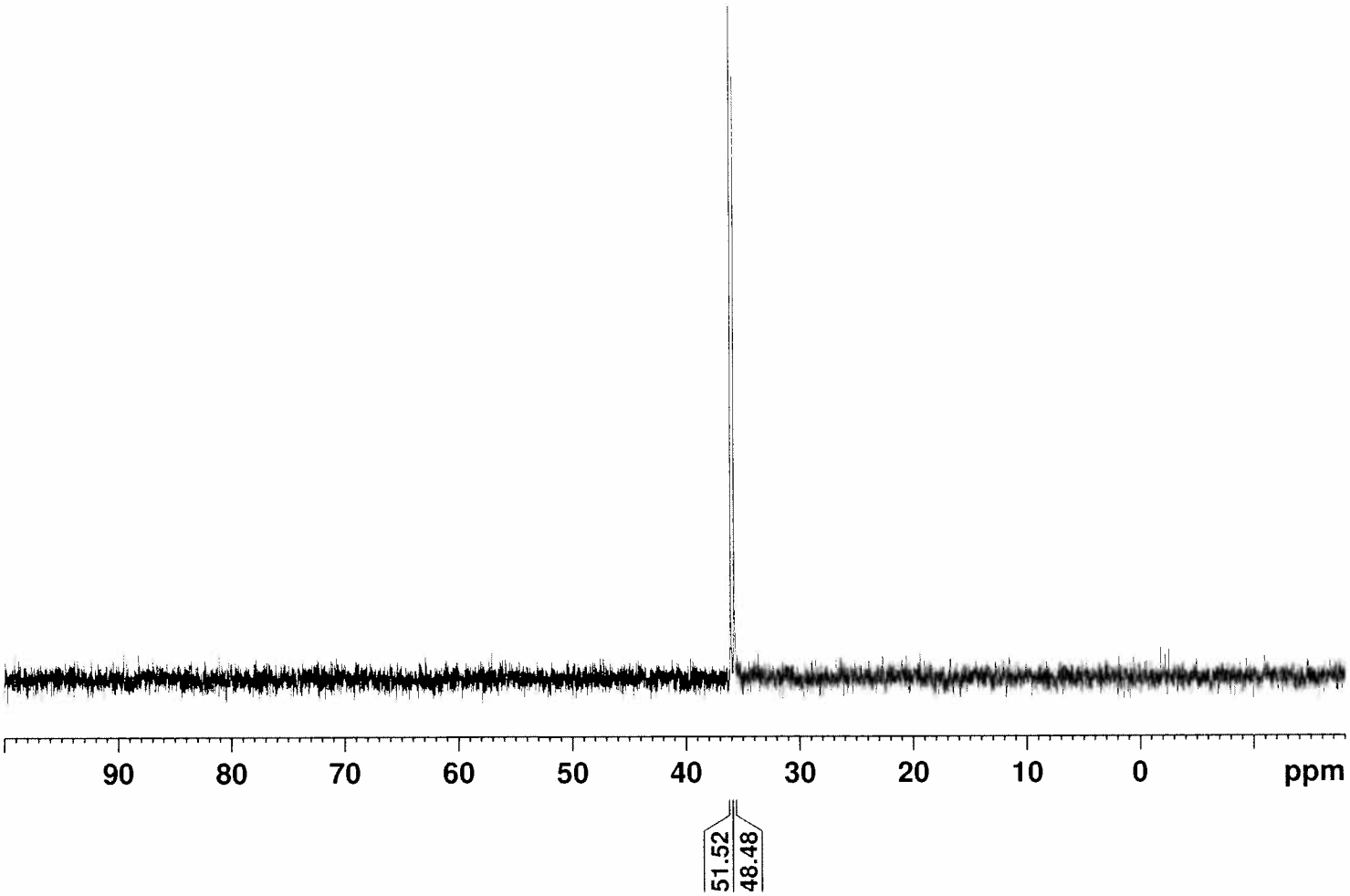
========== CHANNEL f2 =========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



Compound 8a

$^{31}P\{^{1}H\}$ NMR decoupled



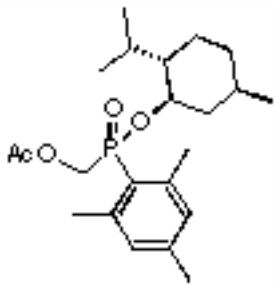

```
Current Data Parameters
NAME                  OB 1415
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20140407
Time                   16.55
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        4
SWH               64102.563 Hz
FIDRES             0.978127 Hz
AQ                 0.5111808 sec
RG                    203.57
DW                     7.800 usec
DE                     6.50 usec
TE                    295.8 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1            161.9674942 MHz
NUC1                    31P
P1                    14.25 usec
PLW1           15.00000000 W

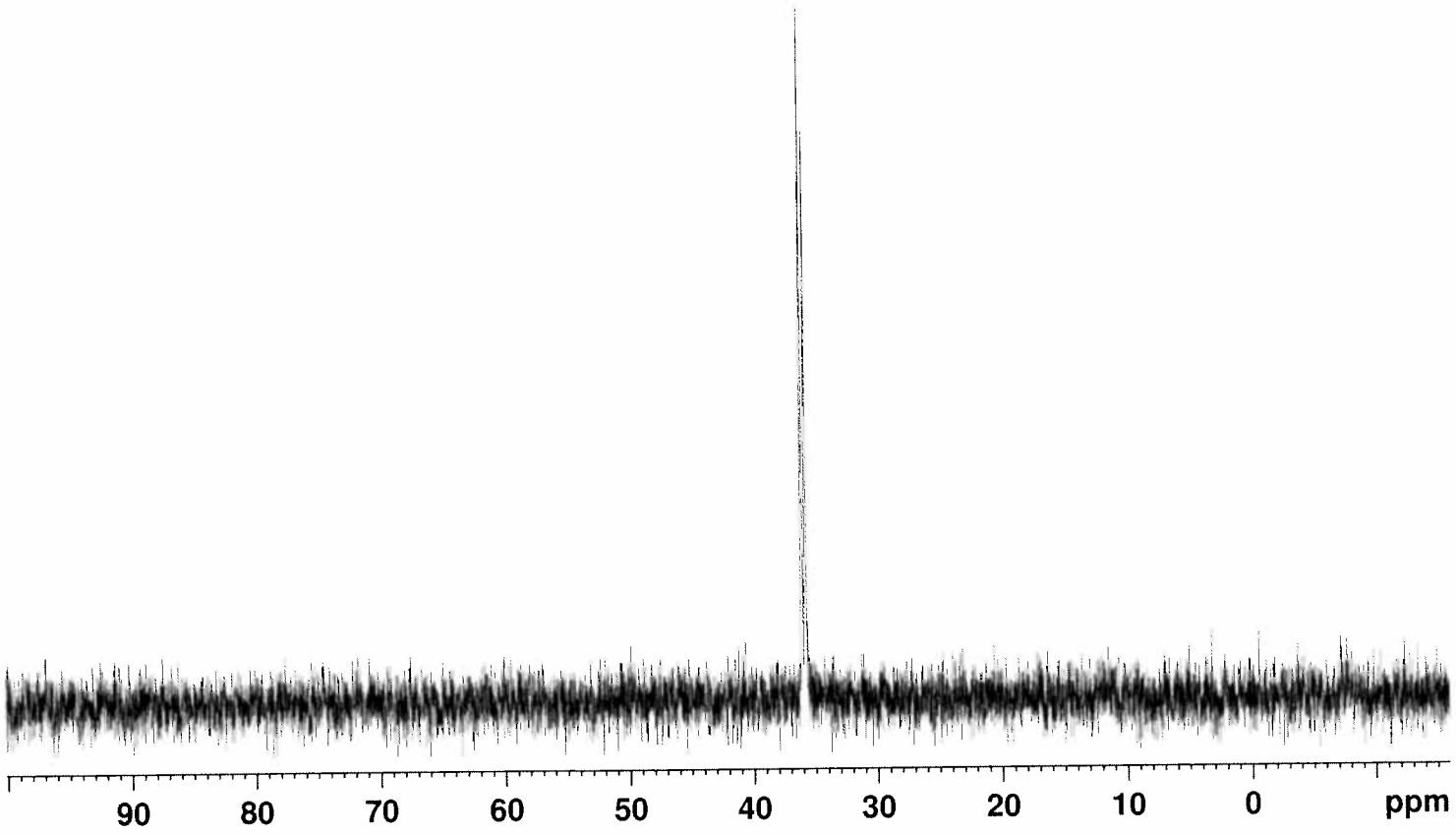
======== CHANNEL f2 ========
SFO2            400.1316005 MHz
NUC2                    1H
CPDPRG[2             waltz16
PCPD2                 90.00 usec
PLW2           10.00000000 W
PLW12           0.31604999 W
PLW13           0.25600001 W

F2 - Processing parameters
SI                      32768
SF              161.9755930 MHz
WDW                        EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.40
```



Compound 8a

$^{31}P\{^{1}H\}$ NMR coupled

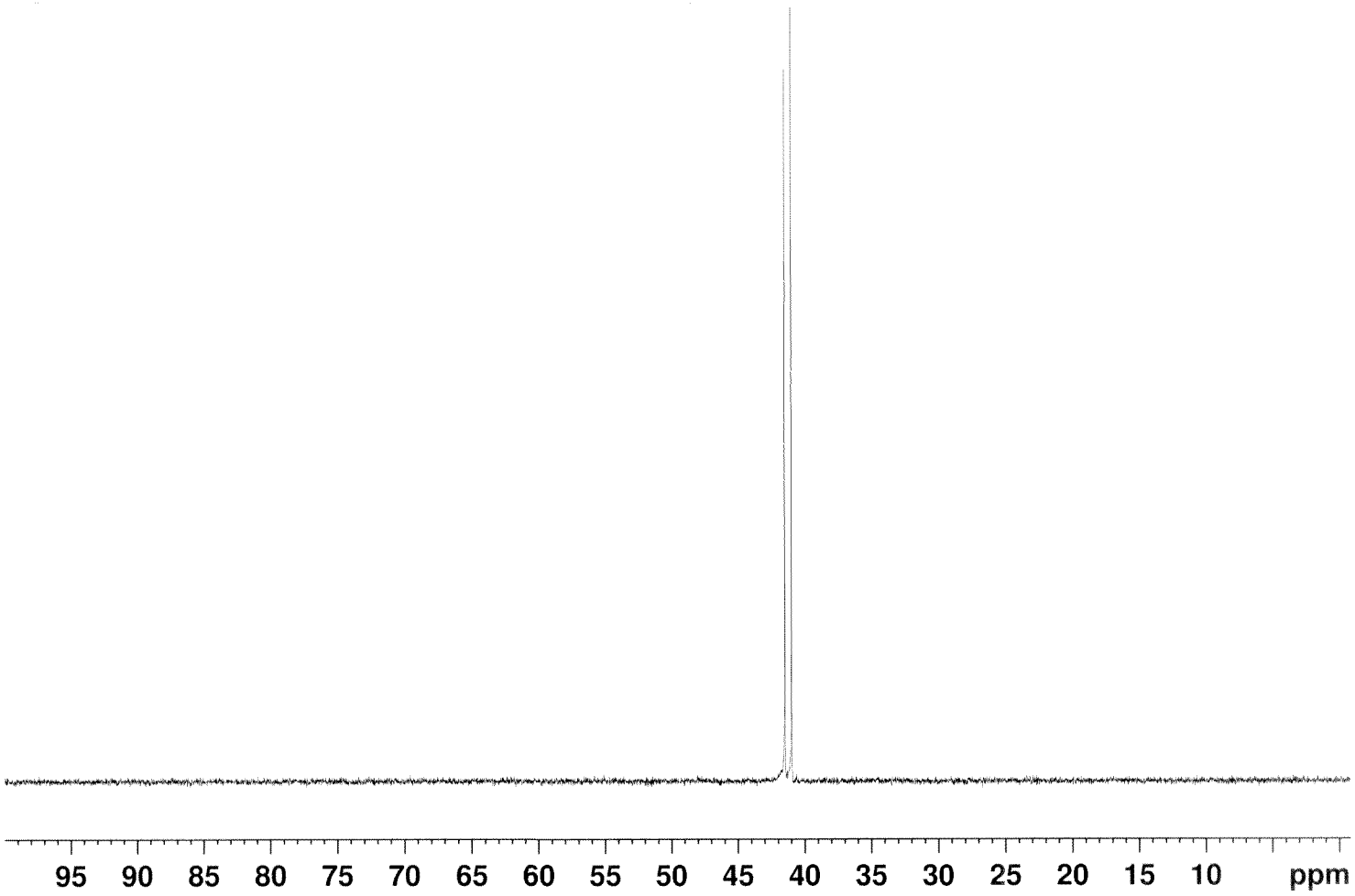


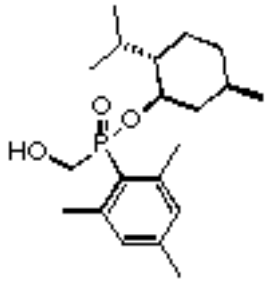

```
Current Data Parameters
NAME                  OB 1415
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20140407
Time                  16.59
INSTRUM               spect
PROBHD     5 mm PABBO BB/
PULPROG               zq30
TD                   65536
SOLVENT              CDCl3
NS                      32
DS                       4
SWH               64102.563 Hz
FIDRES             0.978127 Hz
AQ                0.5111808 sec
RG                   203.57
DW                    7.800 usec
DE                    6.50 usec
TE                   295.7 K
D1                2.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1            161.9674942 MHz
NUC1                    31P
P1                    14.25 usec
PLW1           15.00000000 W

F2 - Processing parameters
SI                      32768
SF              161.9755930 MHz
WDW                        EM
SSB         0
LB                     1.00 Hz
GB          0
PC                     1.40
```





Compound 8b

$^{31}P\{^{1}H\}$ NMR decoupled

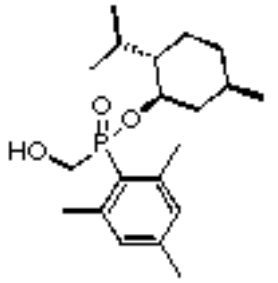
```
Current Data Parameters
NAME        OB 1419 pure
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140411
Time               10.39
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE                296.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1        15.00000000 W

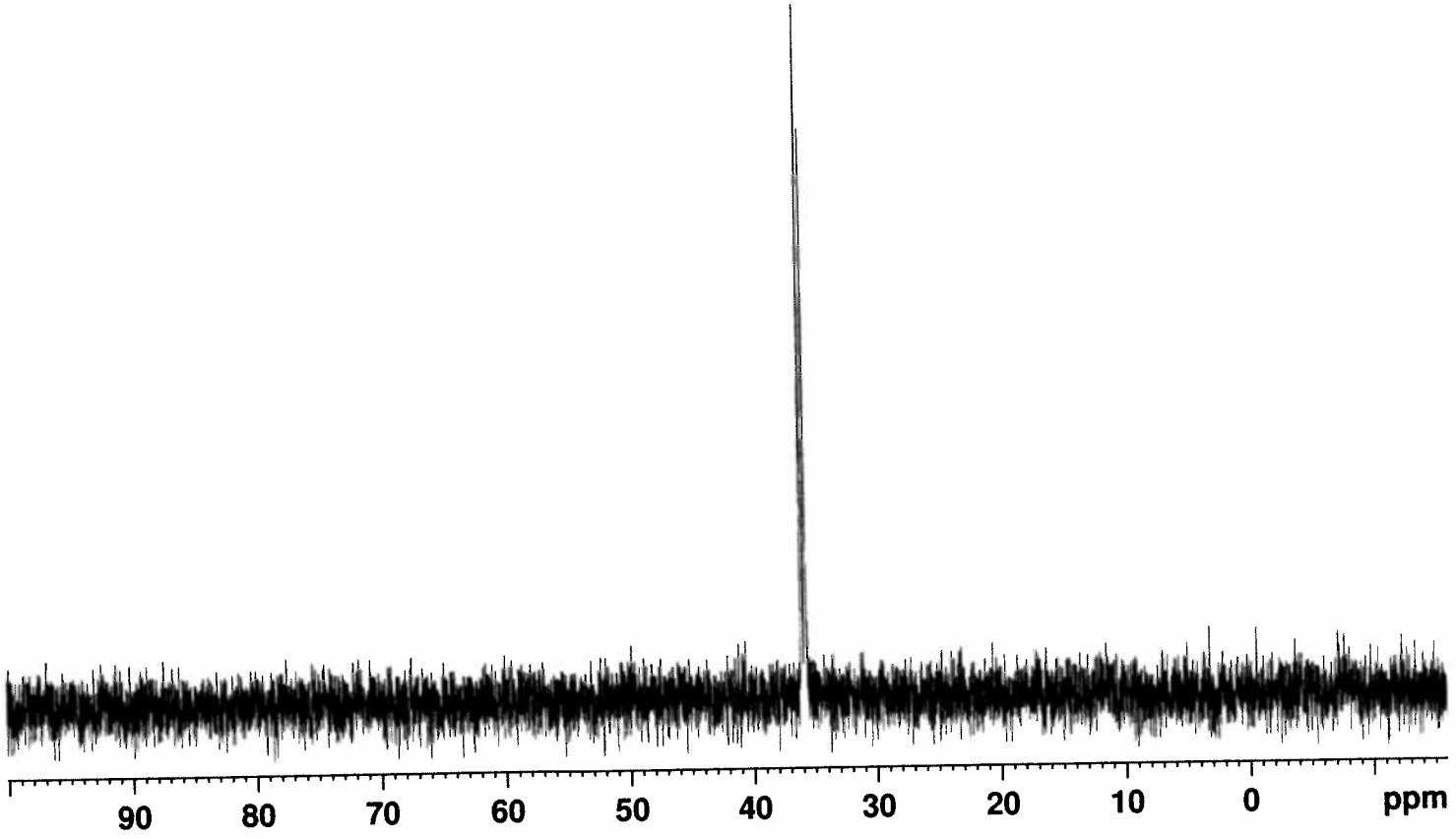
======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                 32768
SF          161.9755930 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



Compound 8b

$^{31}P\{^{1}H\}$ NMR coupled

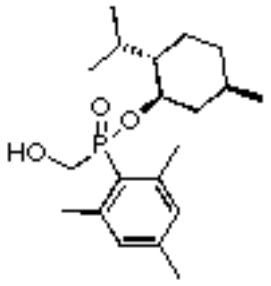



```
Current Data Parameters
NAME                  OB 1415
EXPNO                       2
PROCNO                      1

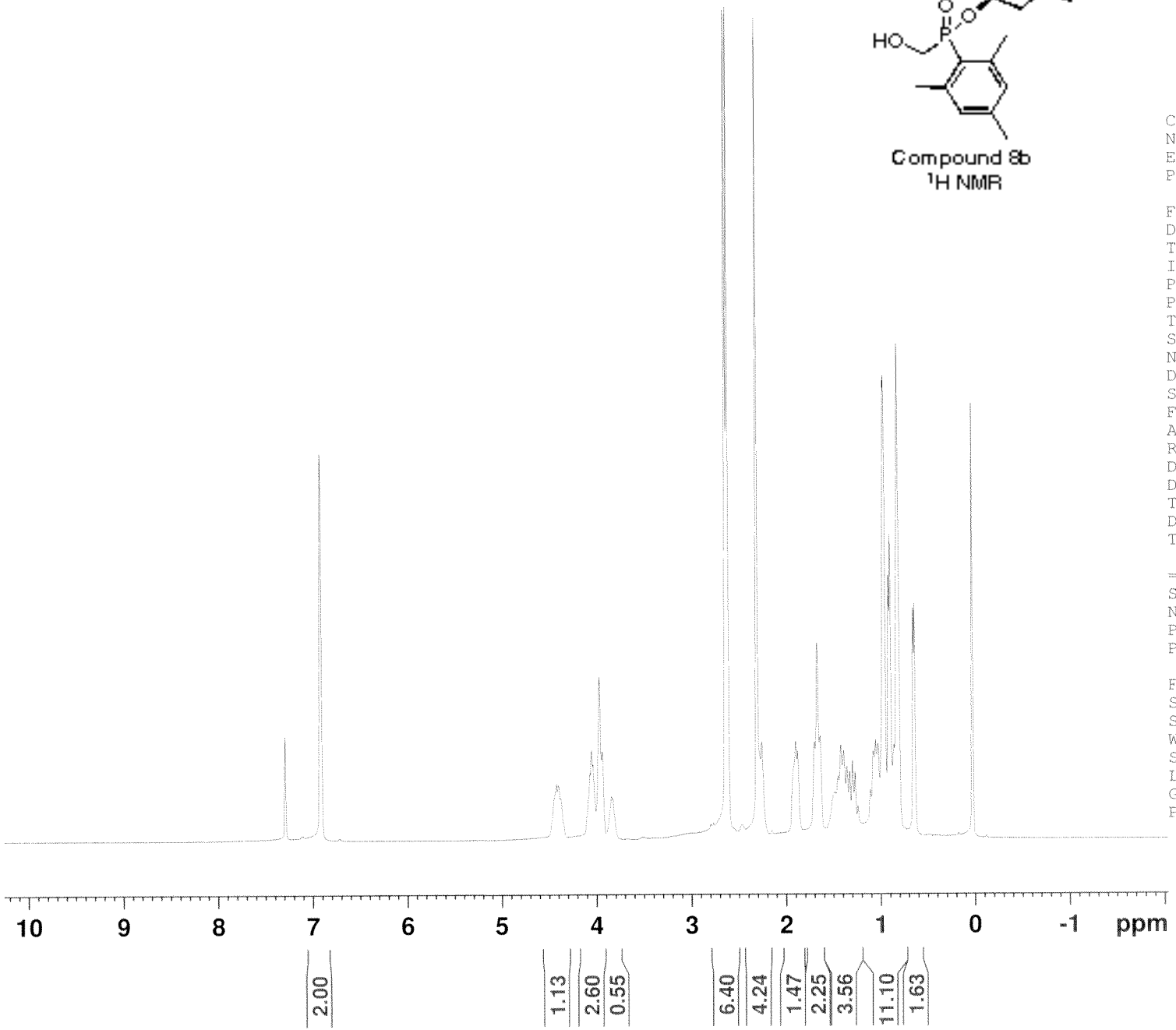
F2 - Acquisition Parameters
Date_              20140407
Time                 16.59
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      4
SWH              64102.563 Hz
FIDRES           0.978127 Hz
AQ               0.5111808 sec
RG                  203.57
DW                  7.800 usec
DE                  6.50 usec
TE                  295.7 K
D1              2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1            161.9674942 MHz
NUC1                   31P
P1                  14.25 usec
PLW1           15.00000000 W

F2 - Processing parameters
SI                     32768
SF              161.9755930 MHz
WDW                    EM
SSB          0
LB                  1.00 Hz
GB           0
PC                  1.40
```



Compound 8b
^{1}H NMR

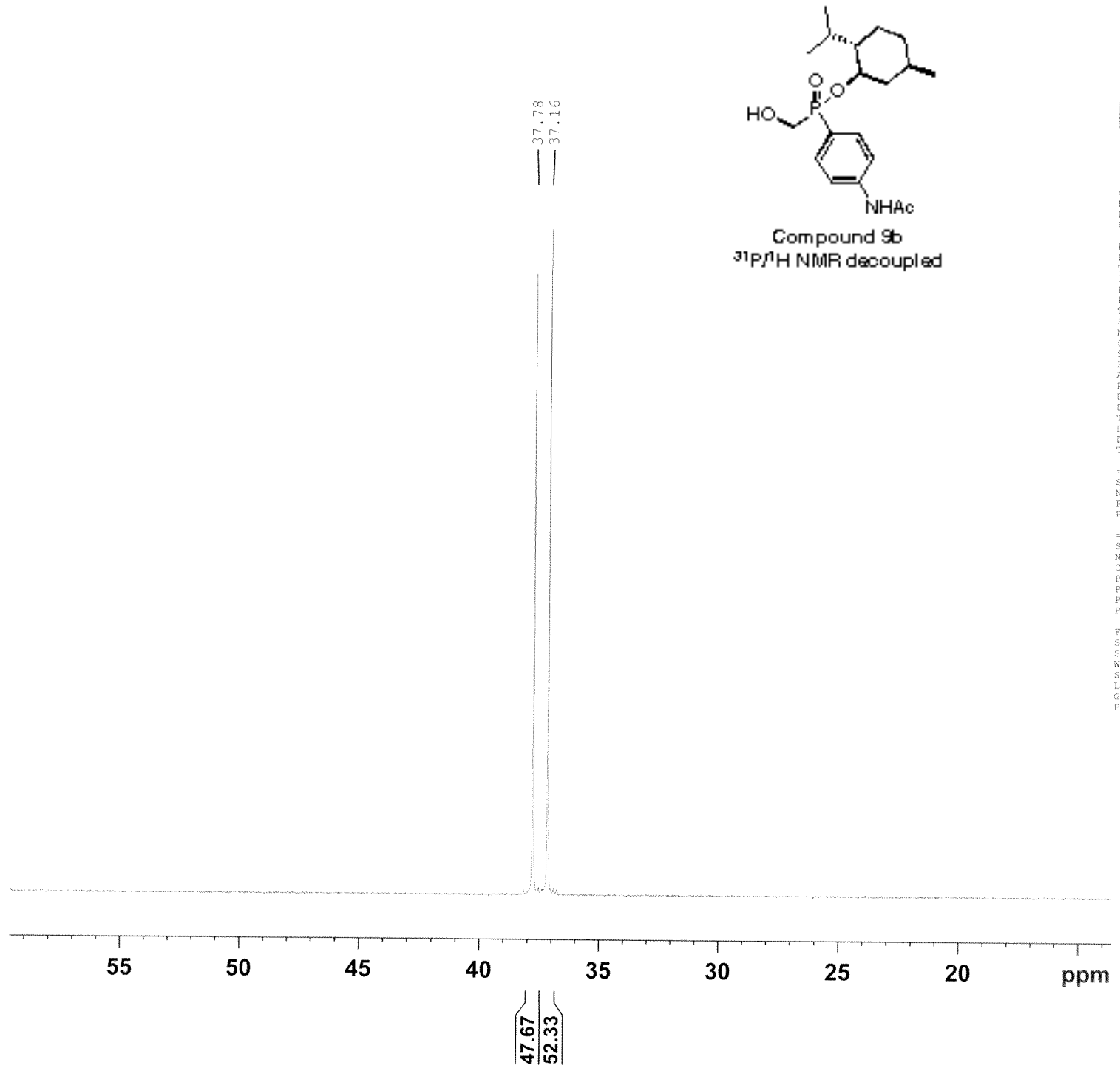



```
Current Data Parameters
NAME        OB 1419 pure
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140411
Time               10.43
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 99.73
DW                62.400 usec
DE                 6.50 usec
TE                295.7 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                  1H
P1                10.00 usec
PLW1          25.00300026 W

F2 - Processing parameters
SI                 65536
SF            400.1300000 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



Compound 9b

$^{31}P\{^{1}H\}$ NMR decoupled

BRUKER

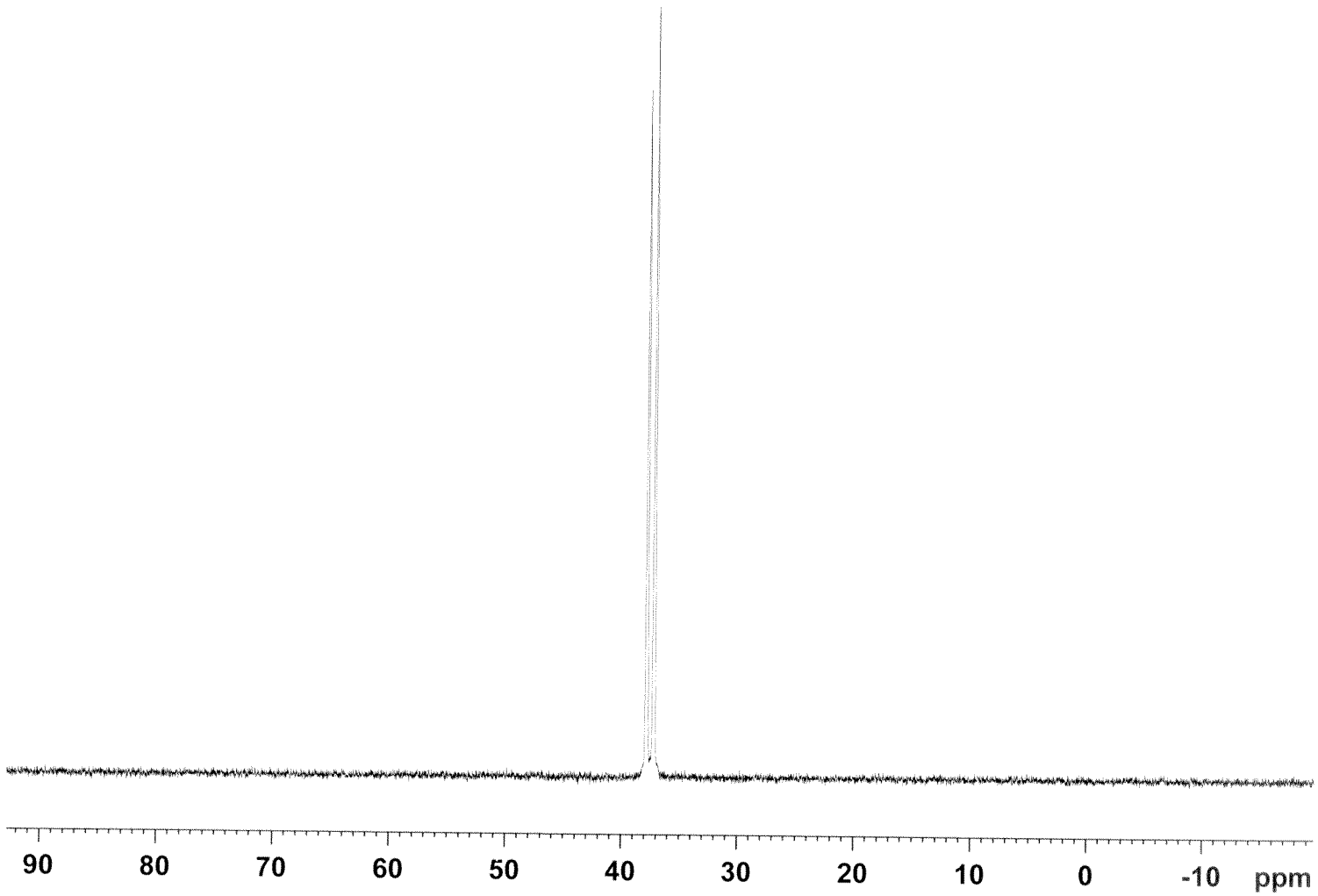
```
Current Data Parameters
NAME      OB 1425 2nd crystallization in CH3CN
EXPNO                 1
PROCNO                1

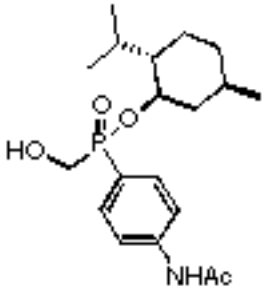
F2 - Acquisition Parameters
Date_          20151111
Time              11.48
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   4
SWH          64102.563 Hz
FIDRES        0.978127 Hz
AQ           0.5111808 sec
RG              203.57
DW               7.800 usec
DE               6.50 usec
TE              293.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1       161.9674942 MHz
NUC1              31P
P1              14.25 usec
PLW1       15.00000000 W

======== CHANNEL f2 ========
SFO2       400.1316005 MHz
NUC2              1H
CPDPRG[2       waltz16
PCPD2           90.00 usec
PLW2       10.00000000 W
PLW12       0.31604999 W
PLW13       0.25600001 W

F2 - Processing parameters
SI               32768
SF         161.9755930 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```





Compound 9b

$^{31}P\{^{1}H\}$ NMR coupled

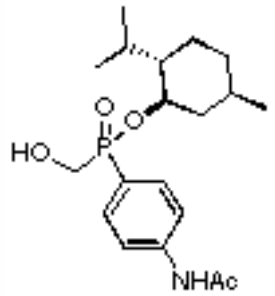
BRUKER

```
Current Data Parameters
NAME      OB 1425 2nd crystallization in CH3CN
EXPNO                 2
PROCNO                1

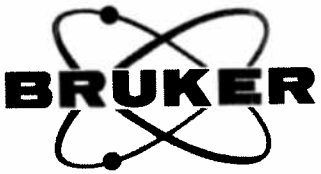
F2 - Acquisition Parameters
Date_          20151111
Time              11.51
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  32
DS                   4
SWH          64102.563 Hz
FIDRES        0.978127 Hz
AQ           0.5111808 sec
RG              203.57
DW               7.800 usec
DE               6.50 usec
TE              293.5 K
D1           2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1       161.9674942 MHz
NUC1              31P
P1              14.25 usec
PLW1       15.00000000 W

F2 - Processing parameters
SI               32768
SF         161.9755930 MHz
WDW                EM
SSB       0
LB               1.00 Hz
GB        0
PC               1.40
```



Compound 9b
^{1}H NMR

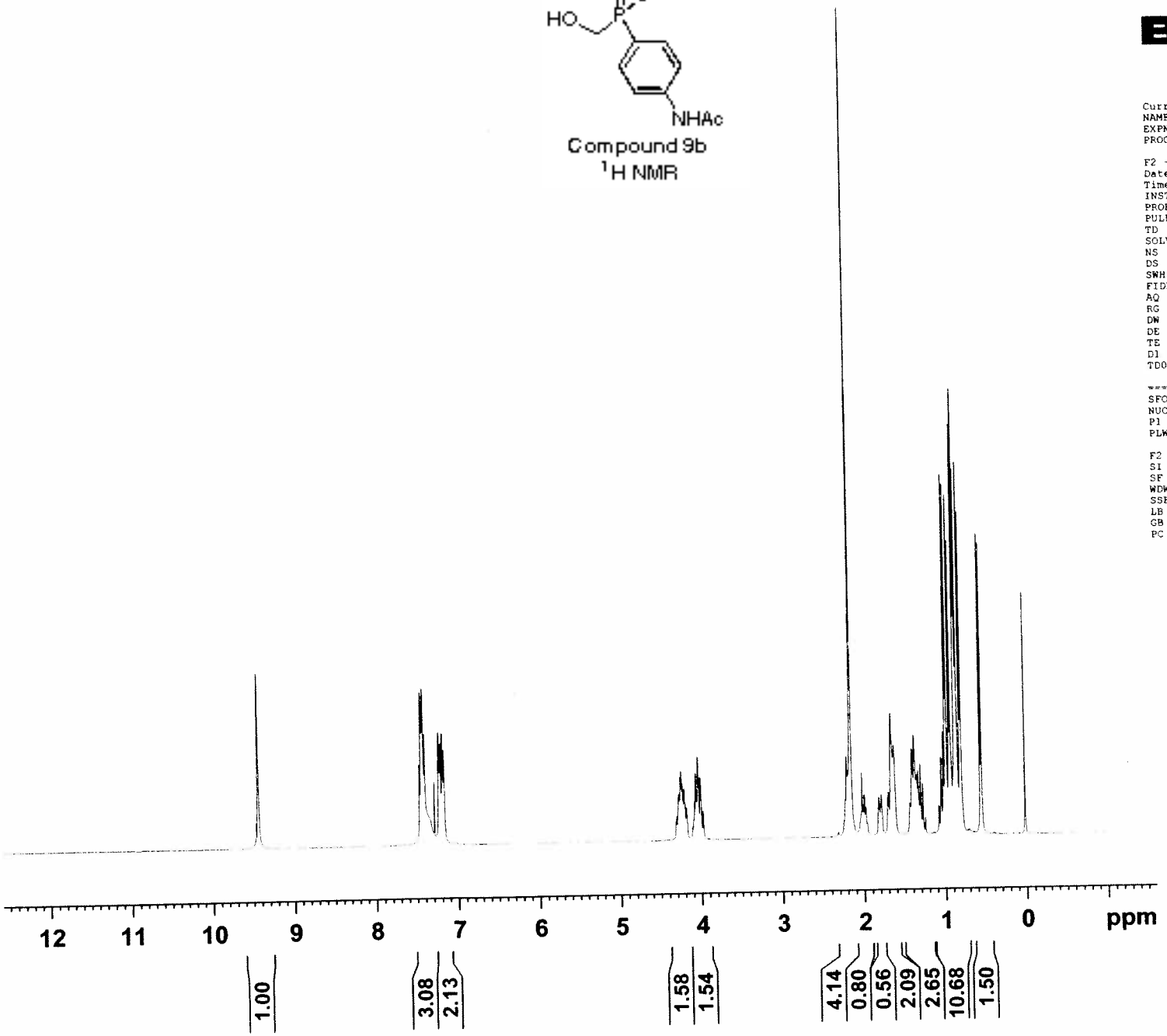



```
Current Data Parameters
NAME      OB 1425 2nd crystallization in CH3CN
EXPNO                3
PROCNO               1

F2 - Acquisition Parameters
Date_         20151111
Time              11.52
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   9
DS                   2
SWH           8012.820 Hz
FIDRES           0.122266 Hz
AQ            4.0894465 sec
RG                32.38
DW               62.400 usec
DE                6.50 usec
TE               293.5 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1                1H
P1                10.00 usec
PLW1         25.00300026 W

F2 - Processing parameters
SI                65536
SF          400.1300000 MHz
WDW                 EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

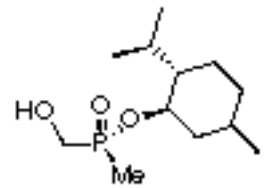


OB 320

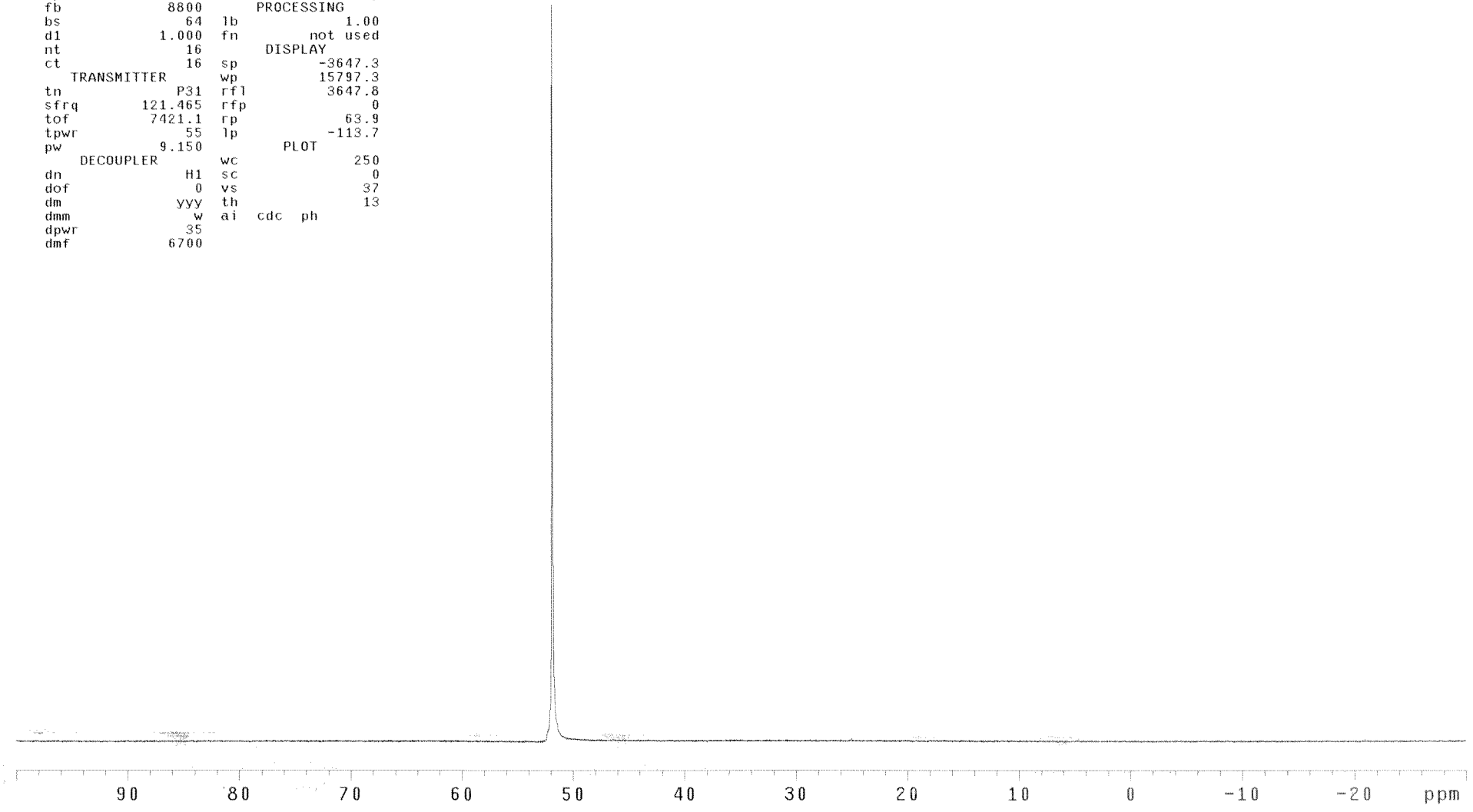
pad=10 run with findz0 before acquisitio
n

exp1 Phosphorus

SAMPLE		SPECIAL	
date	Apr 12 2012	temp	not used
solvent	cdcl3	gain	14
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2012.04.10/s_2012~		pw90	18.300
0412_30/data/cdcl3~		alfa	10.000
_03.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	63.9
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	37
dm	yyy	th	13
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



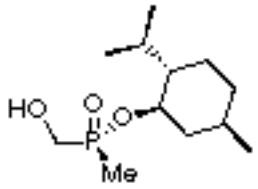
Compound (R_p)-10
$^{31}P\{^1H\}$ NMR decoupled



OB 320

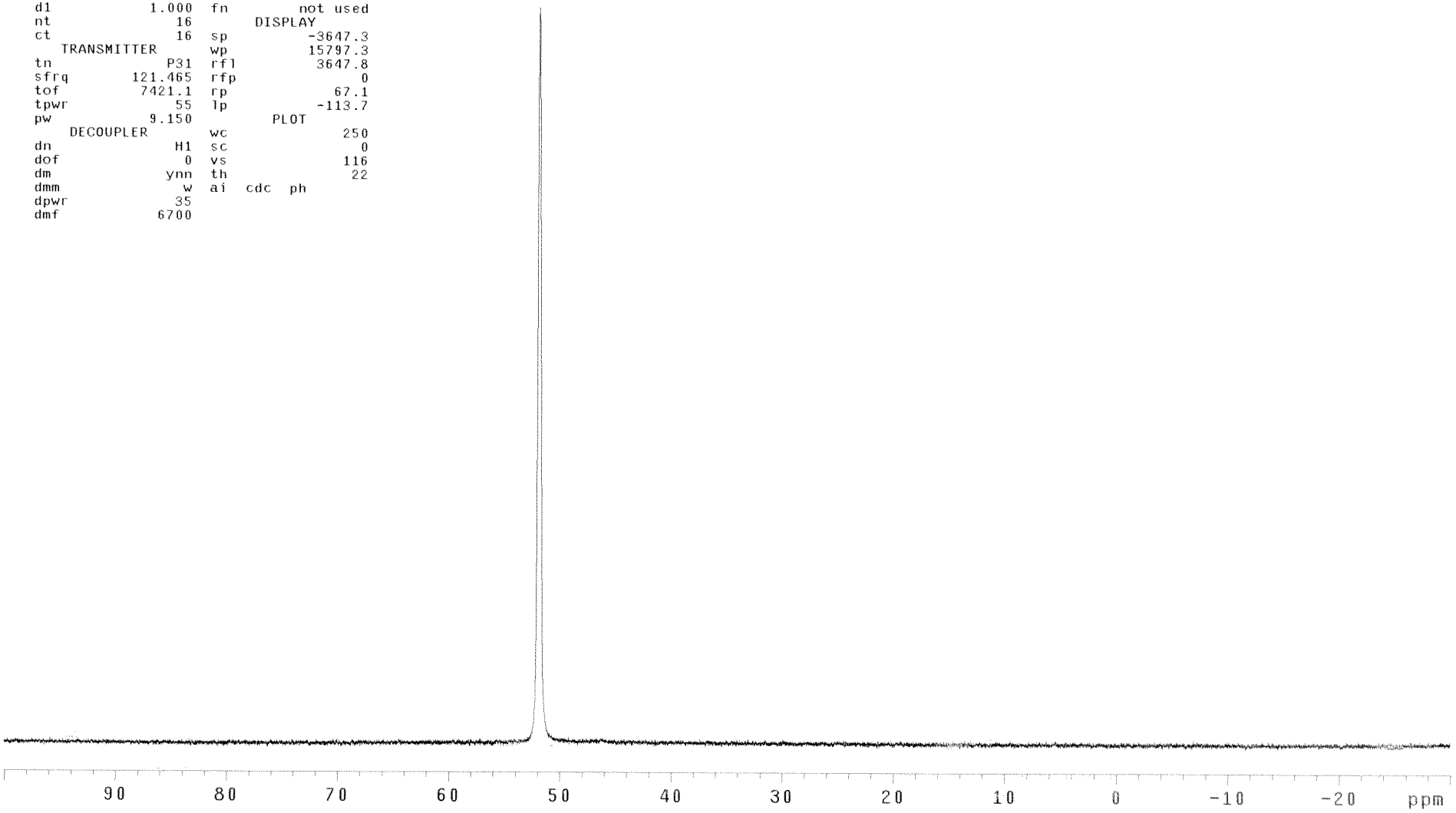
exp1 Phosphorus

SAMPLE		SPECIAL	
date	Apr 12 2012	temp	not used
solvent	cdcl3	gain	14
file	/home/TCUuser~	spin	20
	/vnmrsys/data/auto~	hst	0.008
	_2012.04.10/s_2012~	pw90	18.300
	0412_30/data/cdcl3~	alfa	10.000
	_06.fid	FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	67.1
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	116
dm	ynn	th	22
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



Compound (R_p)-10

$^{31}P\{^1H\}$ NMR coupled



OB 320

exp1 Proton

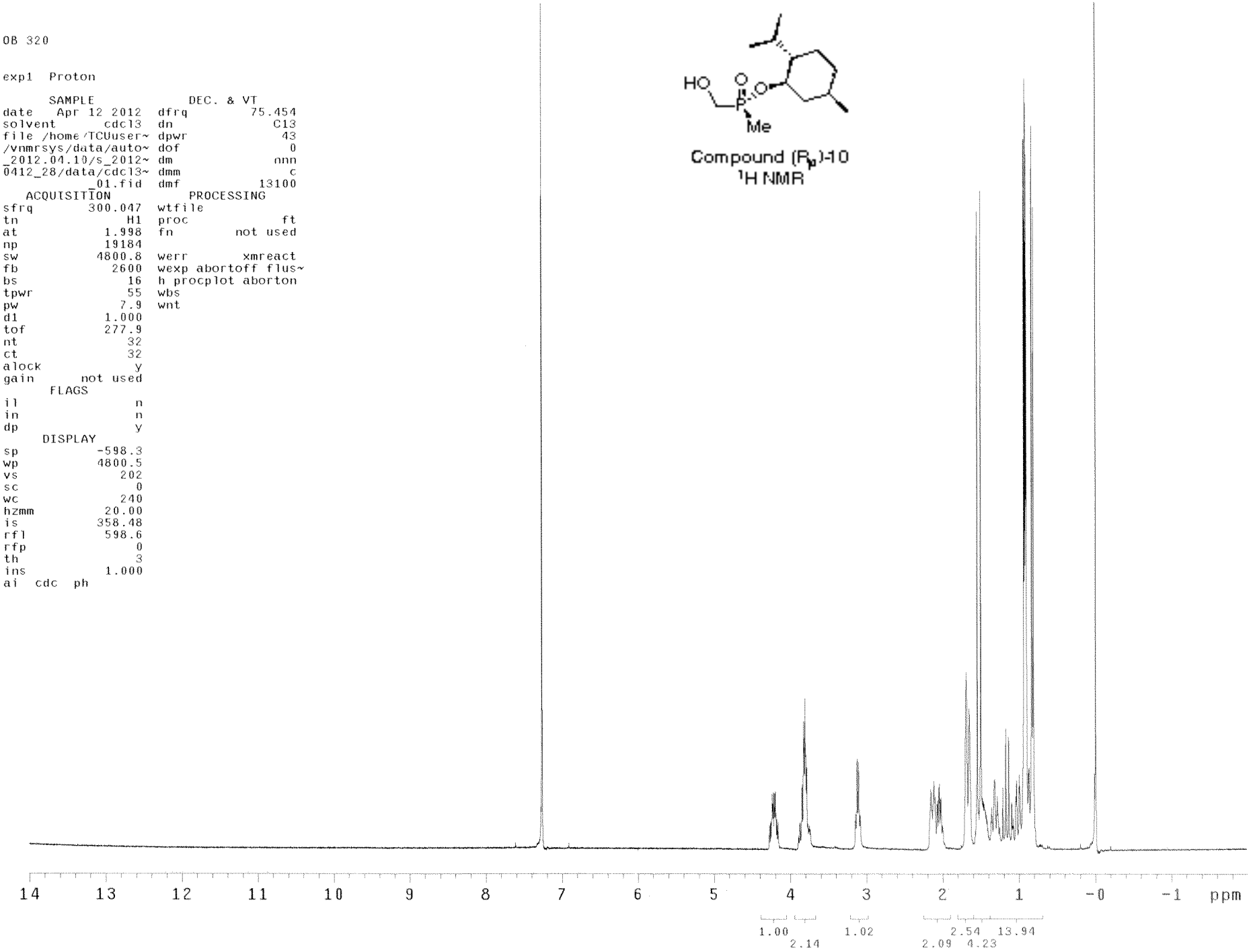
SAMPLE		DEC. & VT	
date	Apr 12 2012	dfrq	75.454
solvent	cdcl3	dn	C13
file	/home/TCUuser~	dpwr	43
	/vnmrsys/data/auto~	dof	0
	_2012.04.10/s_2012~	dm	nnn
	0412_28/data/cdcl3~	dmm	c
	_01.fid	dmf	13100
ACQUISITION		**PROCESSING**	
sfrq	300.047	wtfile	
tn	H1	proc	ft
at	1.998	fn	not used
np	19184		
sw	4800.8	werr	xmreact
fb	2600	wexp	abortoff flus~
bs	16	h procplot	aborton
tpwr	55	wbs	
pw	7.9	wnt	
d1	1.000		
tof	277.9		
nt	32		
ct	32		
alock	y		
gain	not used		

FLAGS	
il	n
in	n
dp	y

DISPLAY	
sp	-598.3
wp	4800.5
vs	202
sc	0
wc	240
hzmm	20.00
is	358.48
rfl	598.6
rfp	0
th	3
ins	1.000

ai cdc ph

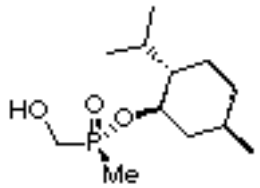
Compound (R_p)-10
^{1}H NMR



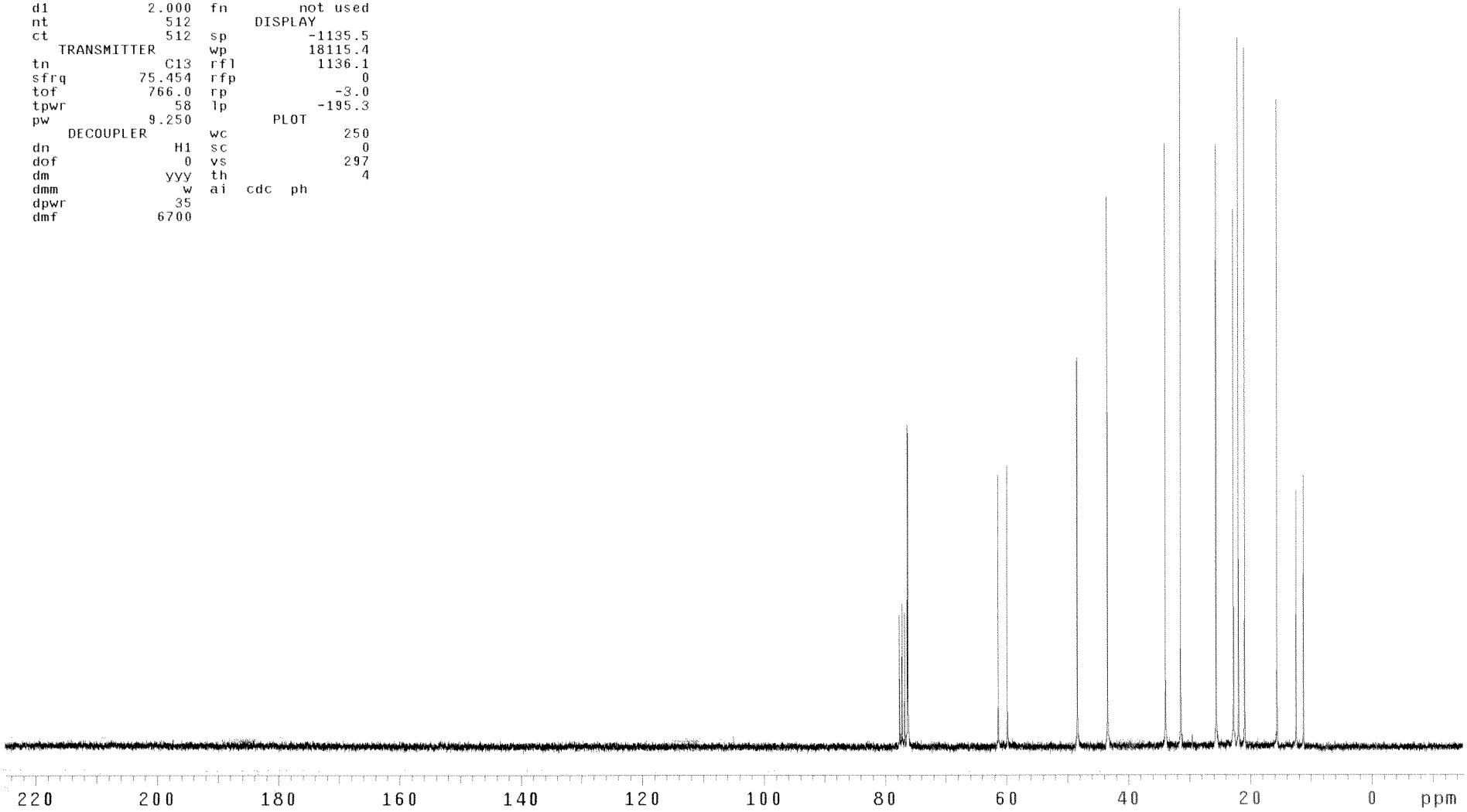
OB 320

exp1 Carbon

SAMPLE		SPECIAL	
date	Apr 12 2012	temp	not used
solvent	cdcl3	gain	20
file	/home/CUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2012.04.10/s_2012~		pw90	18.500
0412_30/data/cdcl3~		alfa	10.000
_07.fid		FLAGS	
ACQUISITION		il	n
sw	18115.9	in	n
at	1.301	dp	y
np	47120	hs	nn
fb	10000	PROCESSING	
bs	64	lb	0.50
d1	2.000	fn	not used
nt	512	DISPLAY	
ct	512	sp	-1135.5
TRANSMITTER		wp	18115.4
tn	C13	rfl	1136.1
sfrq	75.454	rfp	0
tof	766.0	rp	-3.0
tpwr	58	lp	-195.3
pw	9.250	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	297
dm	yyy	th	4
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



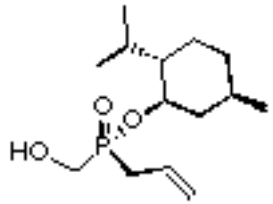
Compound (R_p)-10
^{13}C NMR



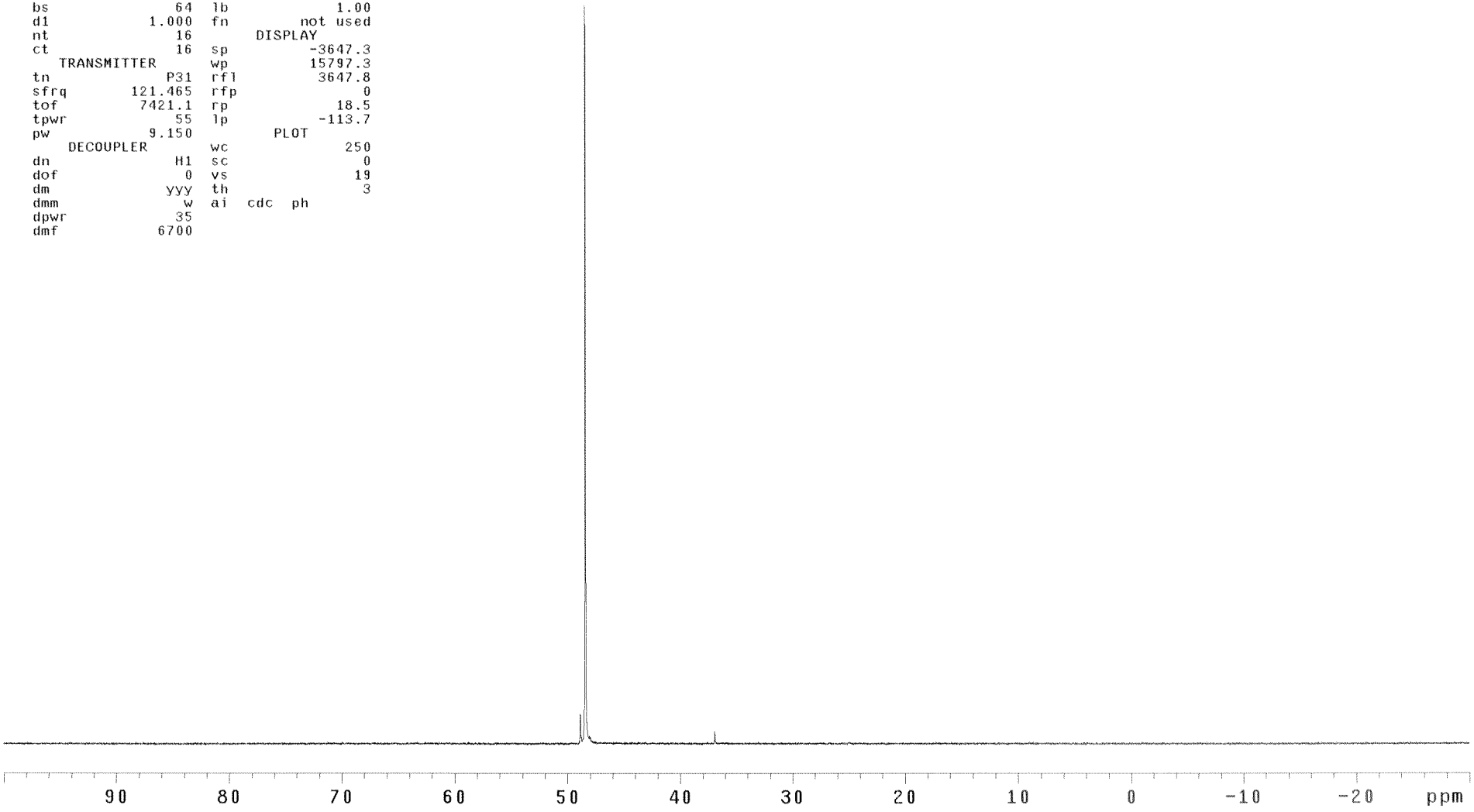
OB 877f2
pad=10 run with findz0 before acquisitio
n

exp1 Phosphorus

SAMPLE		SPECIAL	
date	Dec 19 2012	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2012.12.14/s_2012~		pw90	18.300
1219_28/data/cdcl3~		alfa	10.000
_02.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	18.5
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	19
dm	yyy	th	3
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



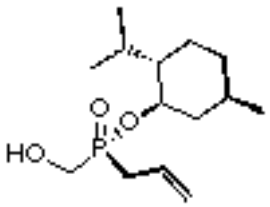
Compound (R_p)-11
$^{31}P\{^1H\}$ NMR decoupled



OB 877f2

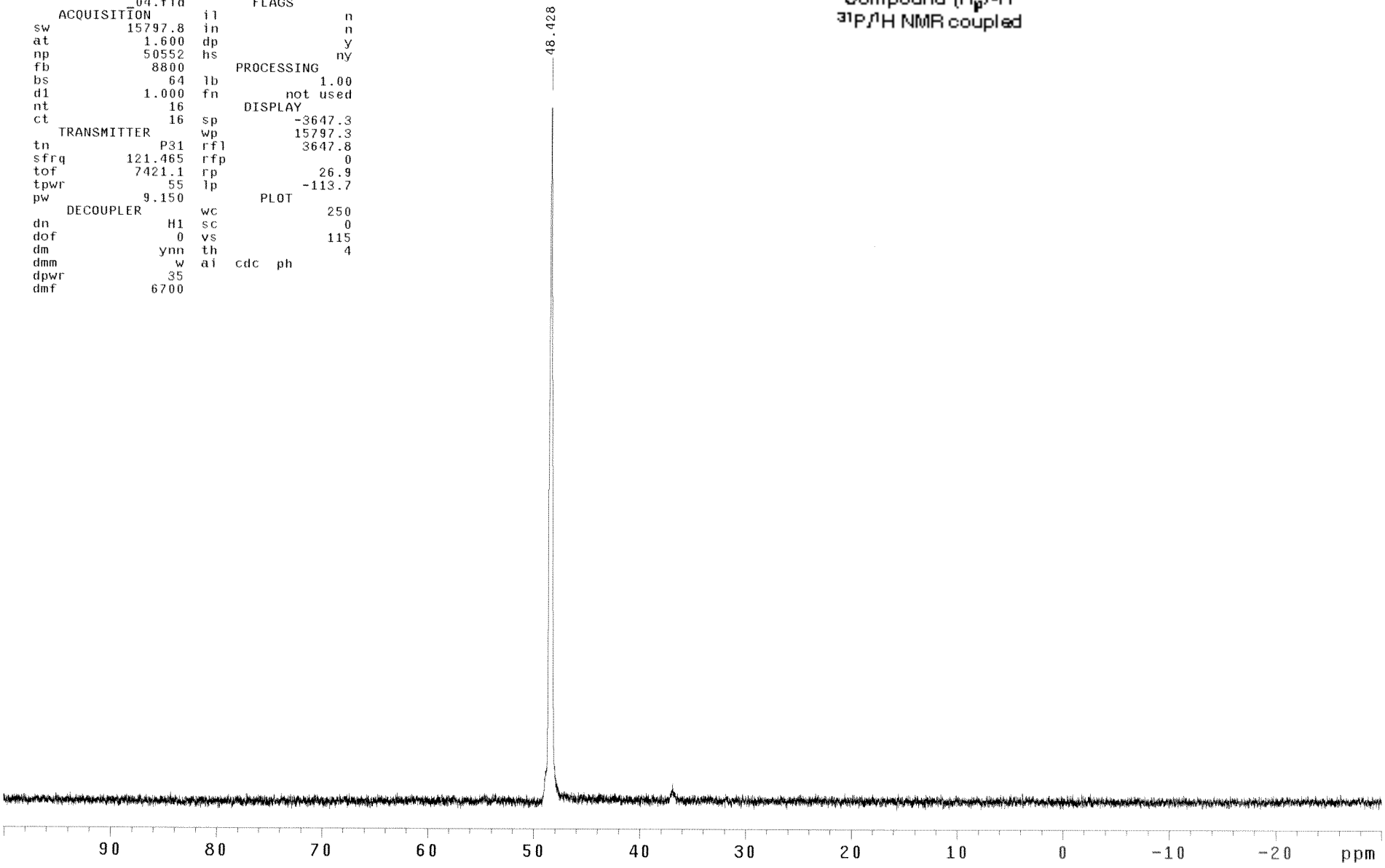
exp1 Phosphorus

SAMPLE		SPECIAL	
date	Dec 19 2012	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
	/vnmrsys/data/auto~	hst	0.008
	_2012.12.14/s_2012~	pw90	18.300
	1219_28/data/cdcl3~	alfa	10.000
	04.fid	FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	26.9
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	115
dm	ynn	th	4
dmm	w	ai cdc ph	
dpwr	35		
dmf	6700		



Compound (R_p)-11

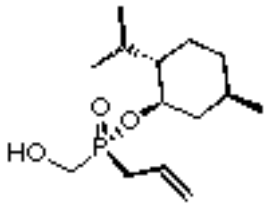
$^{31}P\{^1H\}$ NMR coupled



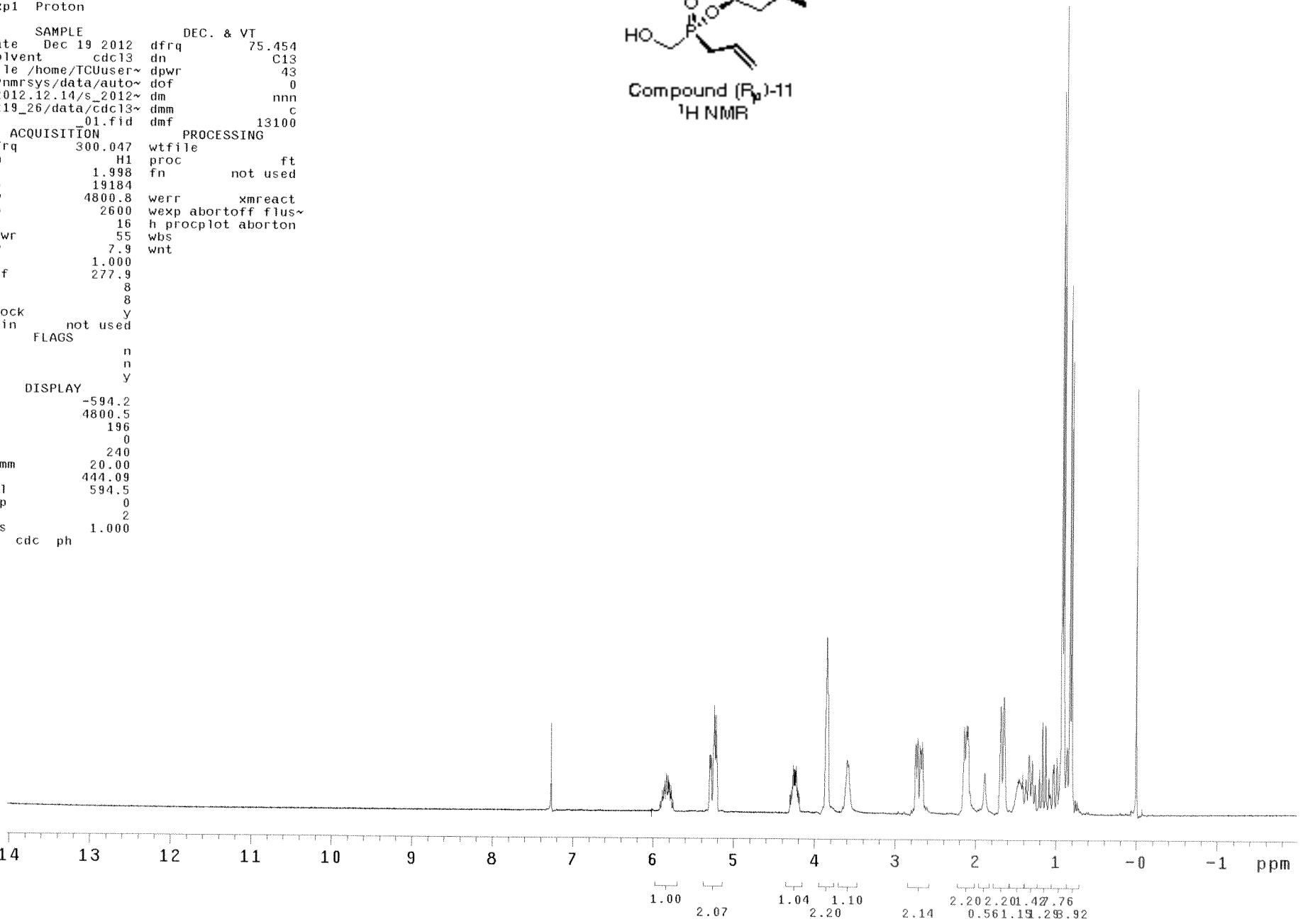
OB 877f1
pad=10 run with findz0 before acquisitio
n

exp1 Proton

SAMPLE		DEC. & VT	
date	Dec 19 2012	dfrq	75.454
solvent	cdcl3	dn	C13
file	/home/TCUuser~	dpwr	43
	/vnmrsys/data/auto~	dof	0
	_2012.12.14/s_2012~	dm	nnn
	1219_26/data/cdcl3~	dmm	c
	01.fid	dmf	13100
ACQUISITION		**PROCESSING**	
sfrq	300.047	wtfile	
tn	H1	proc	ft
at	1.998	fn	not used
np	19184		
sw	4800.8	werr	xmreact
fb	2600	wexp	abortoff flus~
bs	16	h procplot	aborton
tpwr	55	wbs	
pw	7.9	wnt	
d1	1.000		
tof	277.9		
nt	8		
ct	8		
alock	y		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-594.2		
wp	4800.5		
vs	196		
sc	0		
wc	240		
hzmm	20.00		
is	444.09		
rfl	594.5		
rfp	0		
th	2		
ins	1.000		
ai cdc ph			



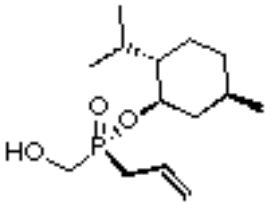
Compound (R_p)-11
^{1}H NMR



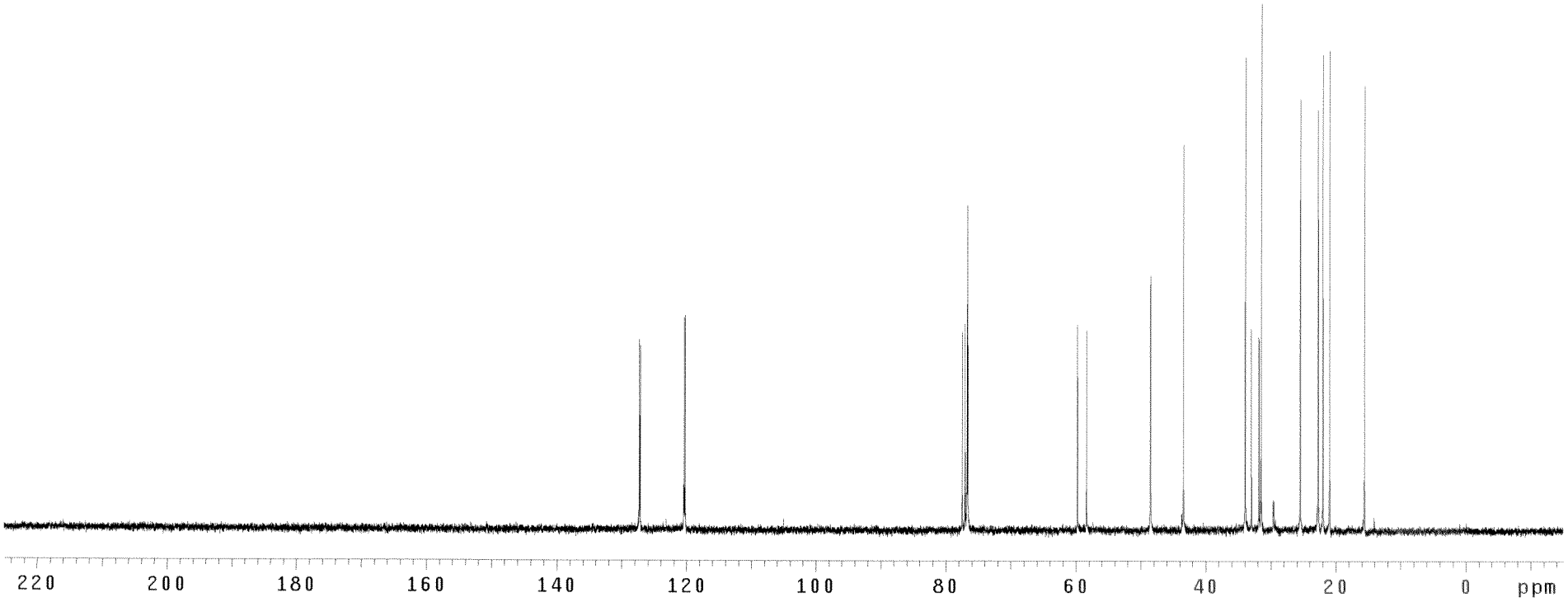
OB 877f2

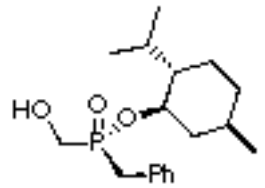
exp1 Carbon

SAMPLE		SPECIAL	
date	Dec 19 2012	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2012.12.14/s_2012~		pw90	18.500
1219_28/data/cdcl3~		alfa	10.000
_05.fid		FLAGS	
ACQUISITION		il	n
sw	18115.9	in	n
at	1.301	dp	y
np	47120	hs	nn
fb	10000	PROCESSING	
bs	64	lb	0.50
d1	2.000	fn	not used
nt	1800	DISPLAY	
ct	1800	sp	-1135.5
TRANSMITTER		wp	18115.4
tn	C13	rfl	1136.1
sfrq	75.454	rfp	0
tof	766.0	rp	-2.0
tpwr	58	lp	-210.4
pw	9.250	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	513
dm	yyy	th	8
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		

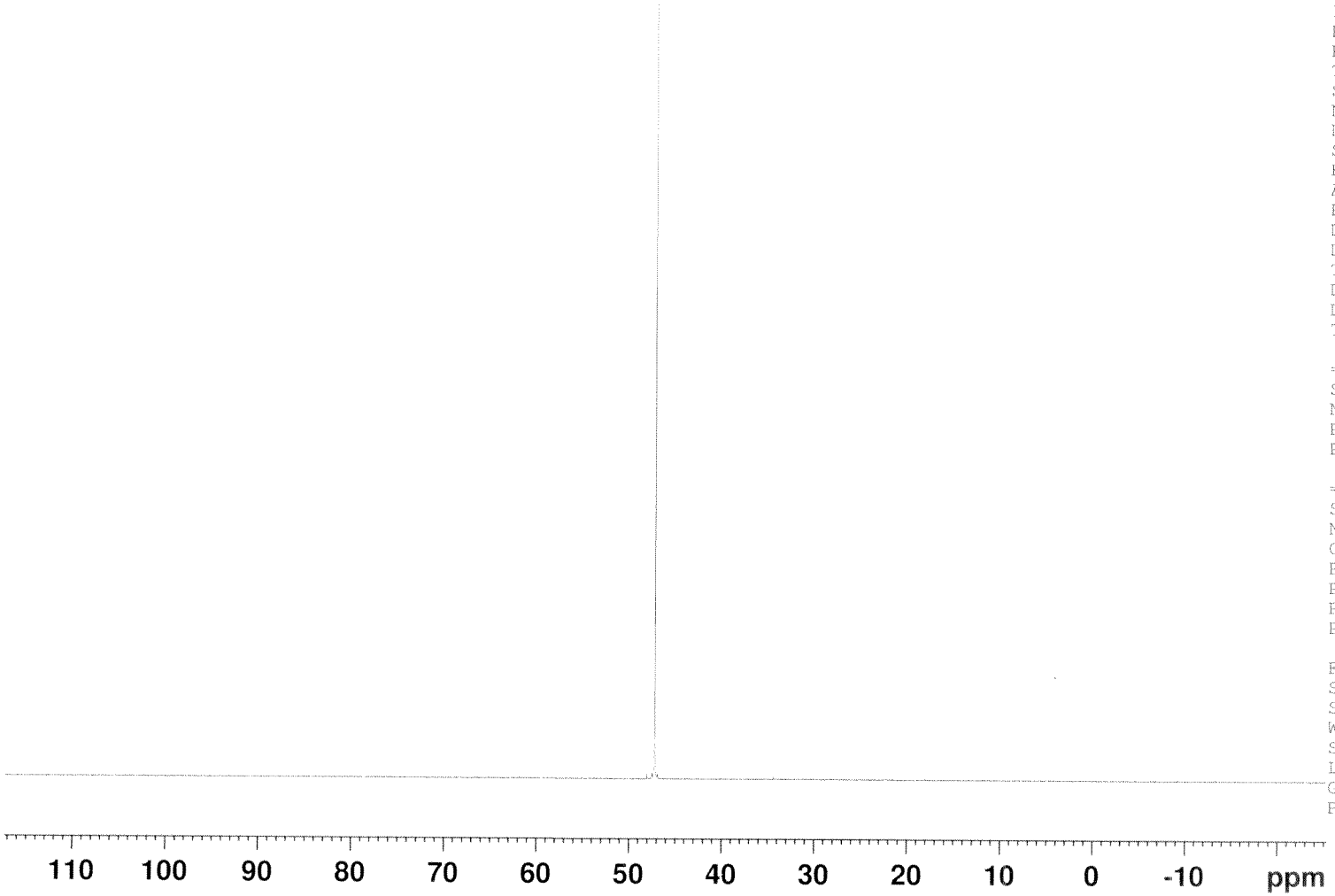


Compound (R_p)-11
^{13}C NMR





Compound (R_p)-12
$^{31}P\{^1H\}$ NMR decoupled

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Current Data Parameters
NAME      OB 1614 after column
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20140805
Time                9.06
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE                295.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1         15.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                32768
SF           161.9755930 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

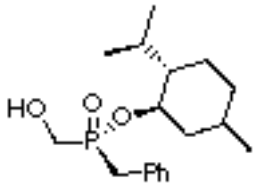

Compound (R_p)-12
$^{31}P\{^1H\}$ NMR coupled

```
Current Data Parameters
NAME      OB 1614 after column
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20140805
Time                9.08
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH            64102.563 Hz
FIDRES          0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                7.800 usec
DE                6.50 usec
TE                295.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         161.9674942 MHz
NUC1                  31P
P1                 14.25 usec
PLW1        15.00000000 W

F2 - Processing parameters
SI                 32768
SF           161.9755930 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
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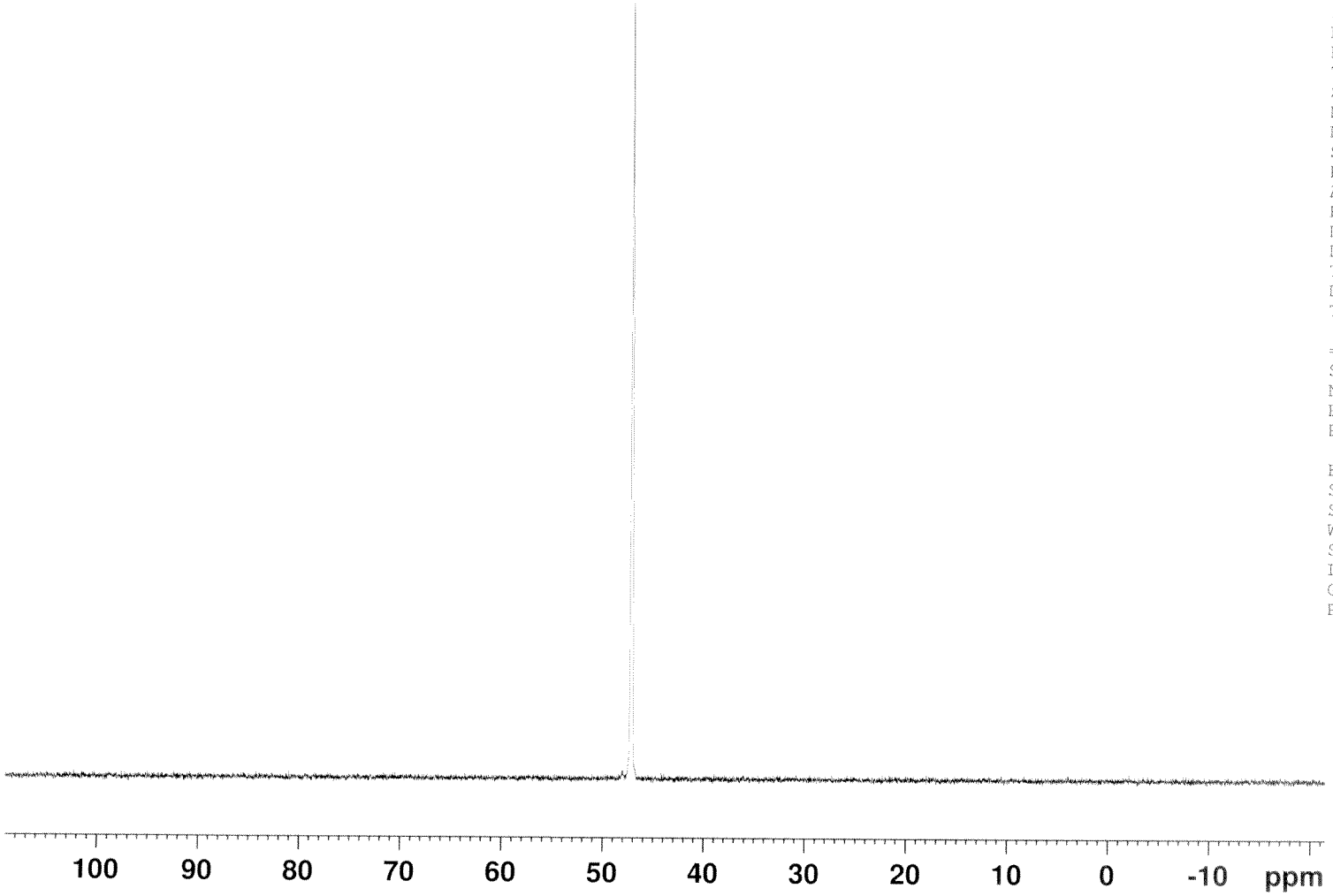
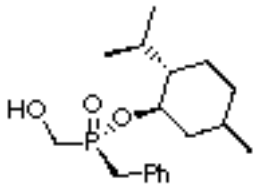
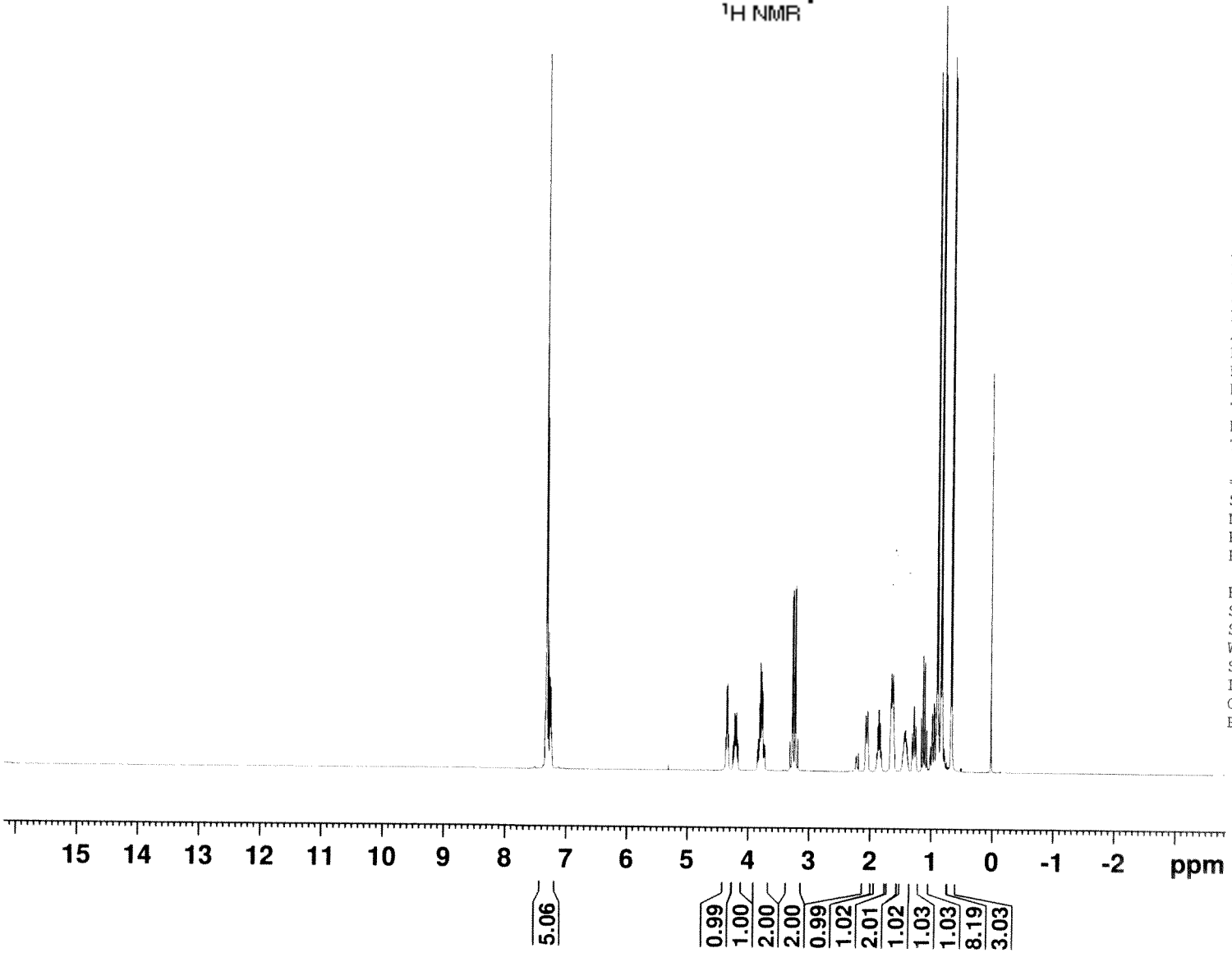




Compound (R_p)-12
^{1}H NMR

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Current Data Parameters
NAME      OB 1614 after column
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_             20140805
Time                 9.11
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  32.38
DW                 62.400 usec
DE                  6.50 usec
TE                 294.9 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                    1H
P1                  10.00 usec
PLW1          25.00300026 W

F2 - Processing parameters
SI                   65536
SF            400.1300000 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                   1.00
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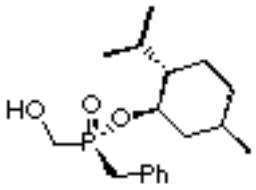
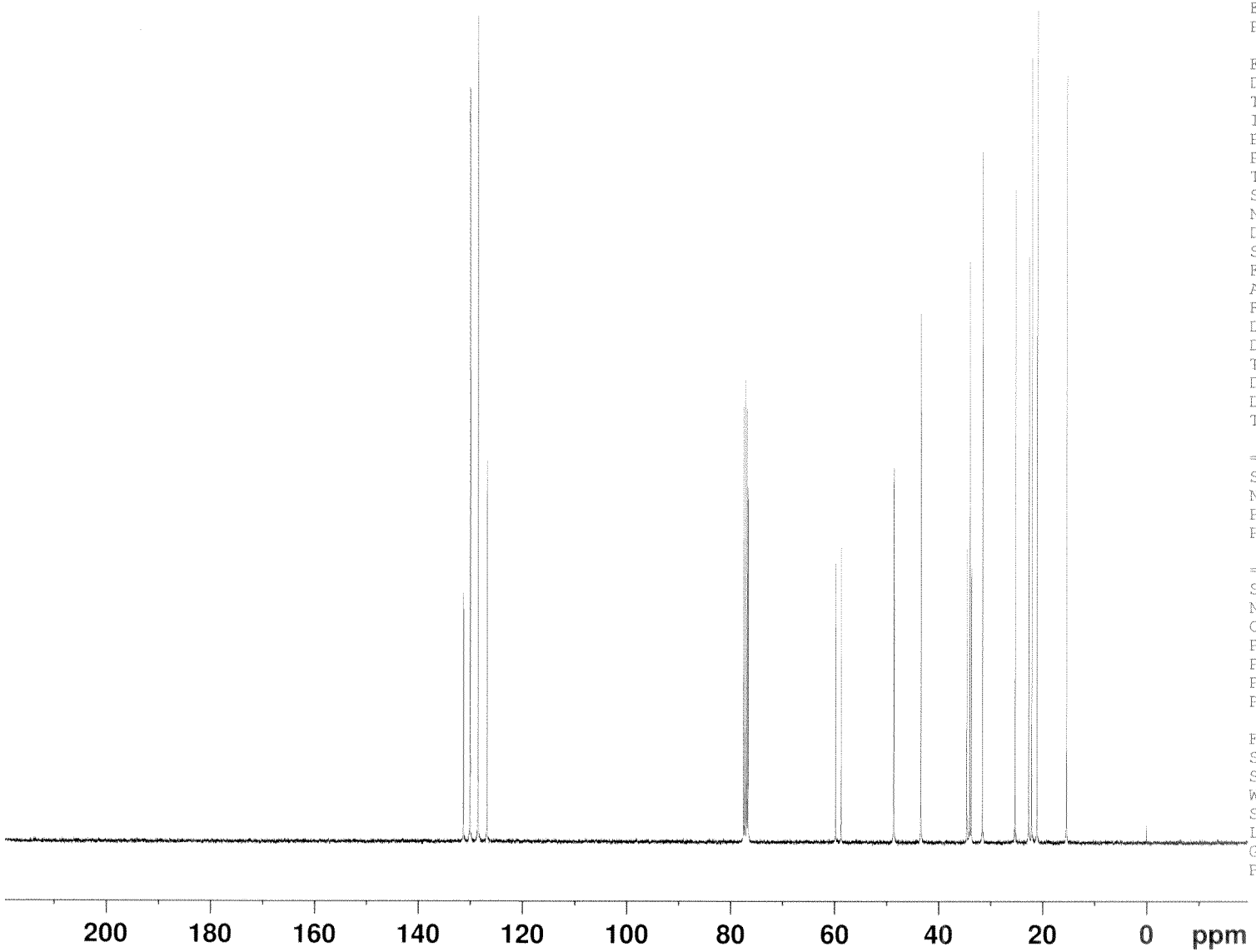


Compound (R_p)-12
^{13}C NMR

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Current Data Parameters
NAME      OB 1614 after column
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_           20140805
Time               19.59
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  456
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                295.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1         45.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                32768
SF          100.6127685 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
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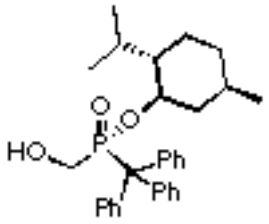
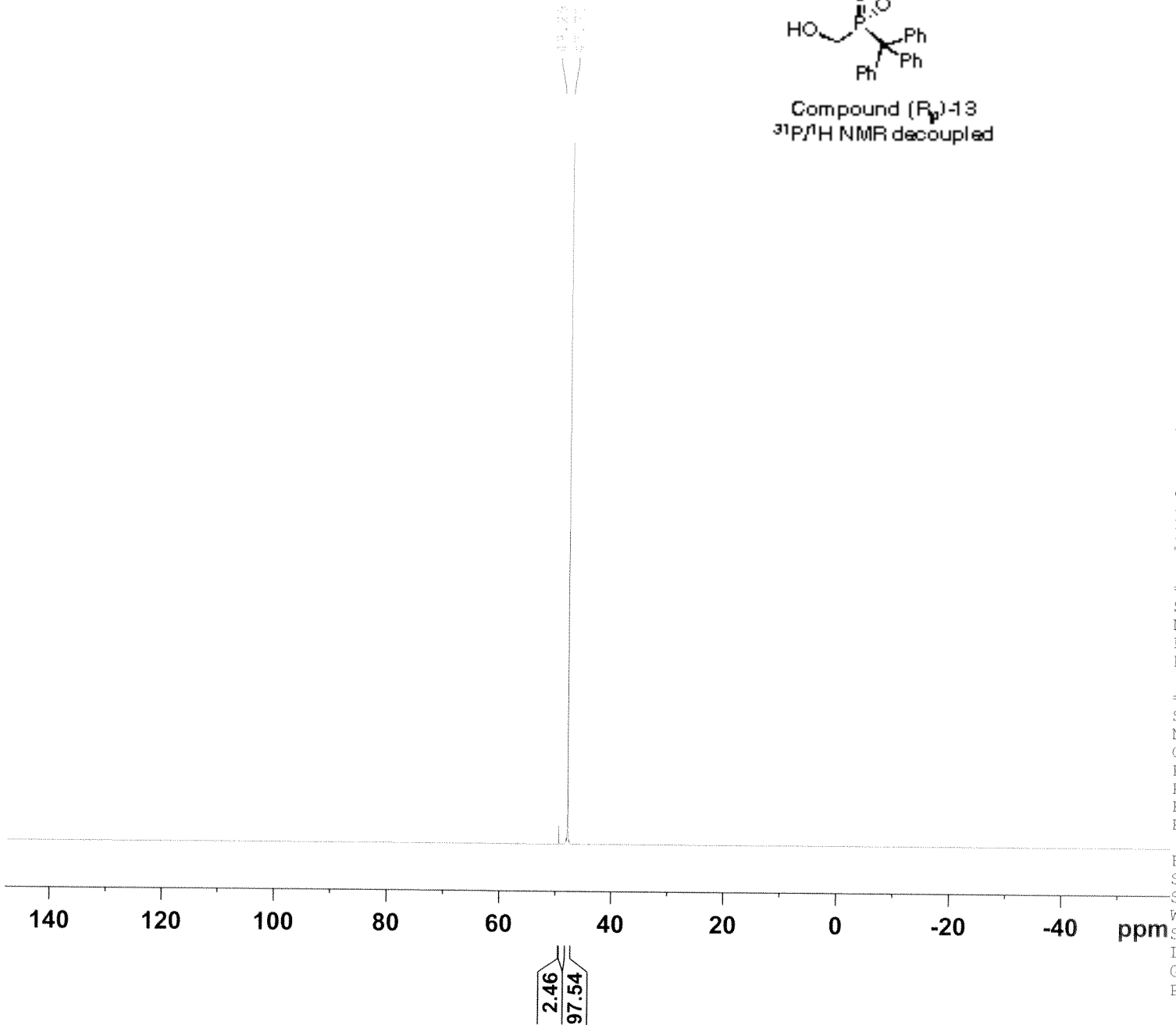


Compound (R_p)-13
$^{31}P\{^1H\}$ NMR decoupled



BRUKER

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Current Data Parameters
NAME                  OB 1840f2
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20141120
Time                      17.48
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                           16
DS                            4
SWH                   64102.563 Hz
FIDRES                0.978127 Hz
AQ                    0.5111808 sec
RG                       203.57
DW                        7.800 usec
DE                        6.50 usec
TE                       296.2 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

========== CHANNEL f1 ==========
SFO1                161.9674942 MHz
NUC1                        31P
P1                        14.25 usec
PLW1                15.00000000 W

========== CHANNEL f2 ==========
SFO2                400.1316005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     90.00 usec
PLW2                10.00000000 W
PLW12                0.31604999 W
PLW13                0.25600001 W

F2 - Processing parameters
SI                        32768
SF                  161.9755930 MHz
WDW                          EM
SSB                   0
LB                        1.00 Hz
GB                    0
PC                        1.40
```
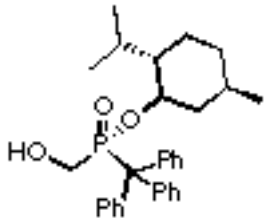
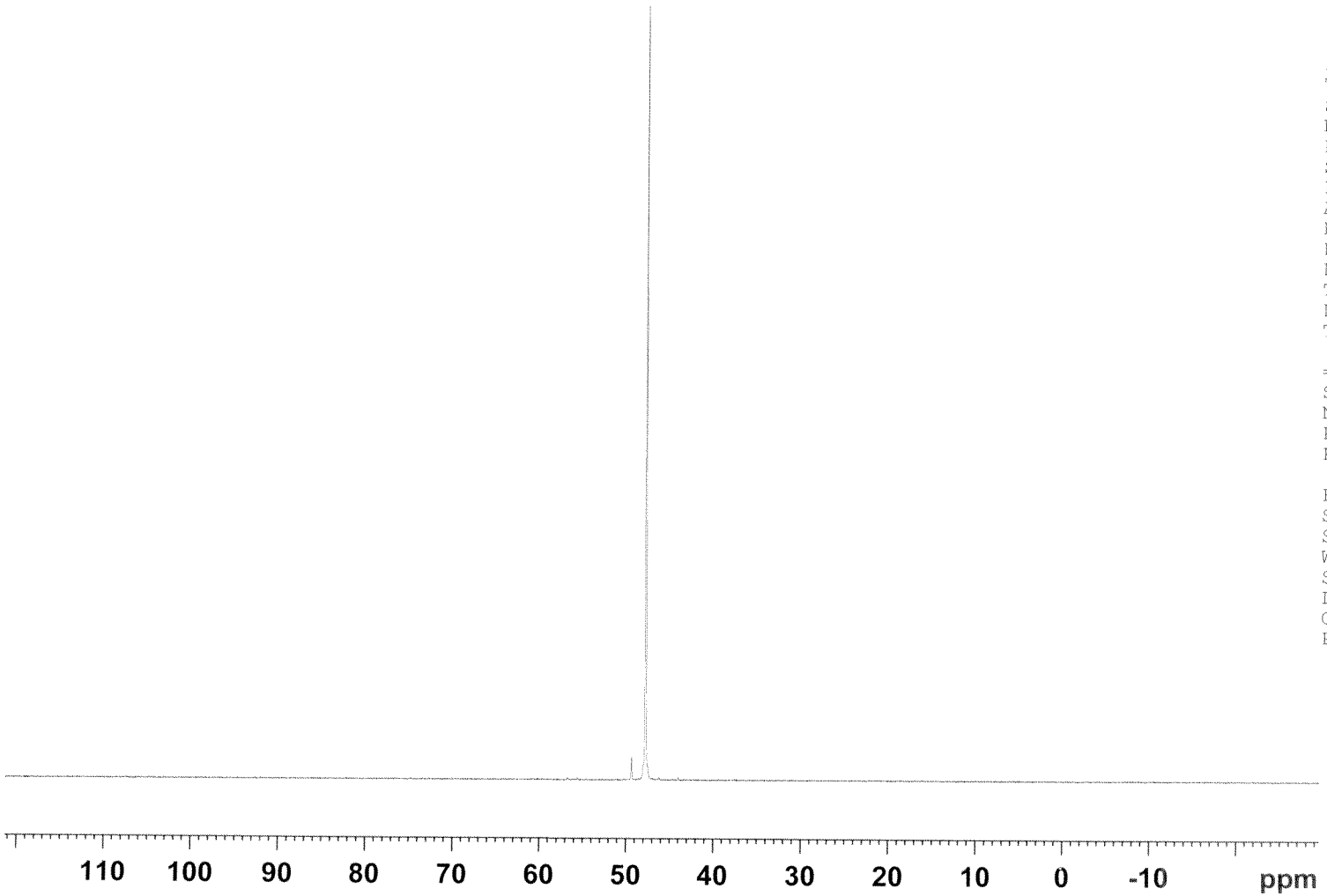
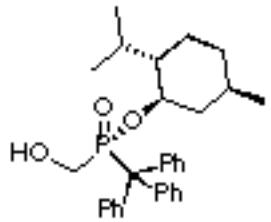



Compound (R_p)-13
$^{31}P\{^1H\}$ NMR coupled

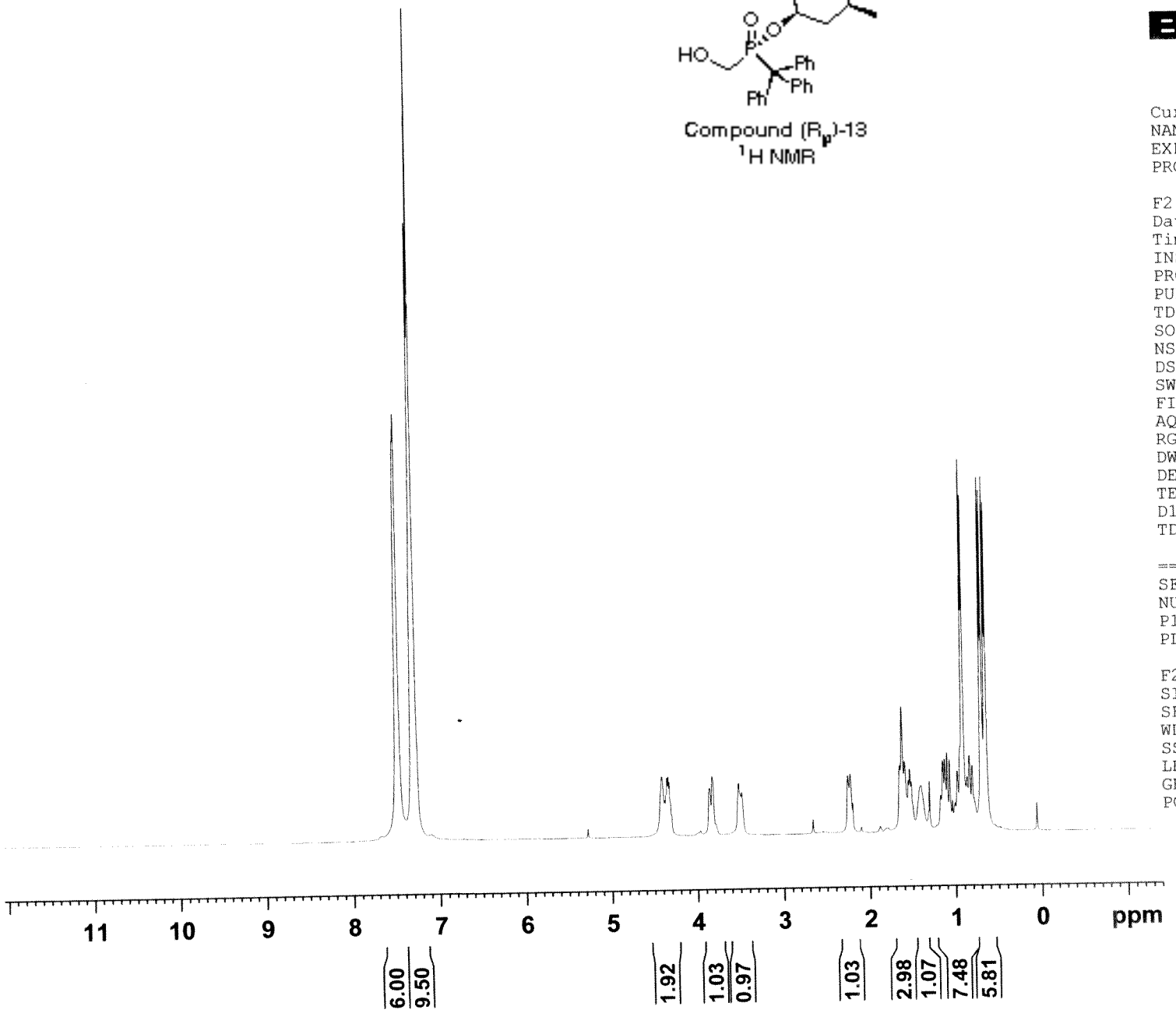
BRUKER

```
Current Data Parameters
NAME                  OB 1840f2
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20141120
Time                      17.50
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                           32
DS                            4
SWH                   64102.563 Hz
FIDRES                 0.978127 Hz
AQ                     0.5111808 sec
RG                        203.57
DW                         7.800 usec
DE                         6.50 usec
TE                        295.6 K
D1                    2.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                161.9674942 MHz
NUC1                        31P
P1                        14.25 usec
PLW1                15.00000000 W

F2 - Processing parameters
SI                        32768
SF                  161.9755930 MHz
WDW                          EM
SSB               0
LB                        1.00 Hz
GB                0
PC                        1.40
```





Compound (R_p)-13
^{1}H NMR



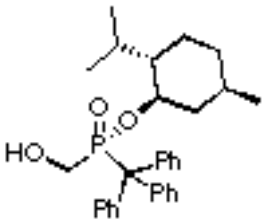
BRUKER

Current Data Parameters
NAME OB 1840f2
EXPNO 3
PROCNO 1

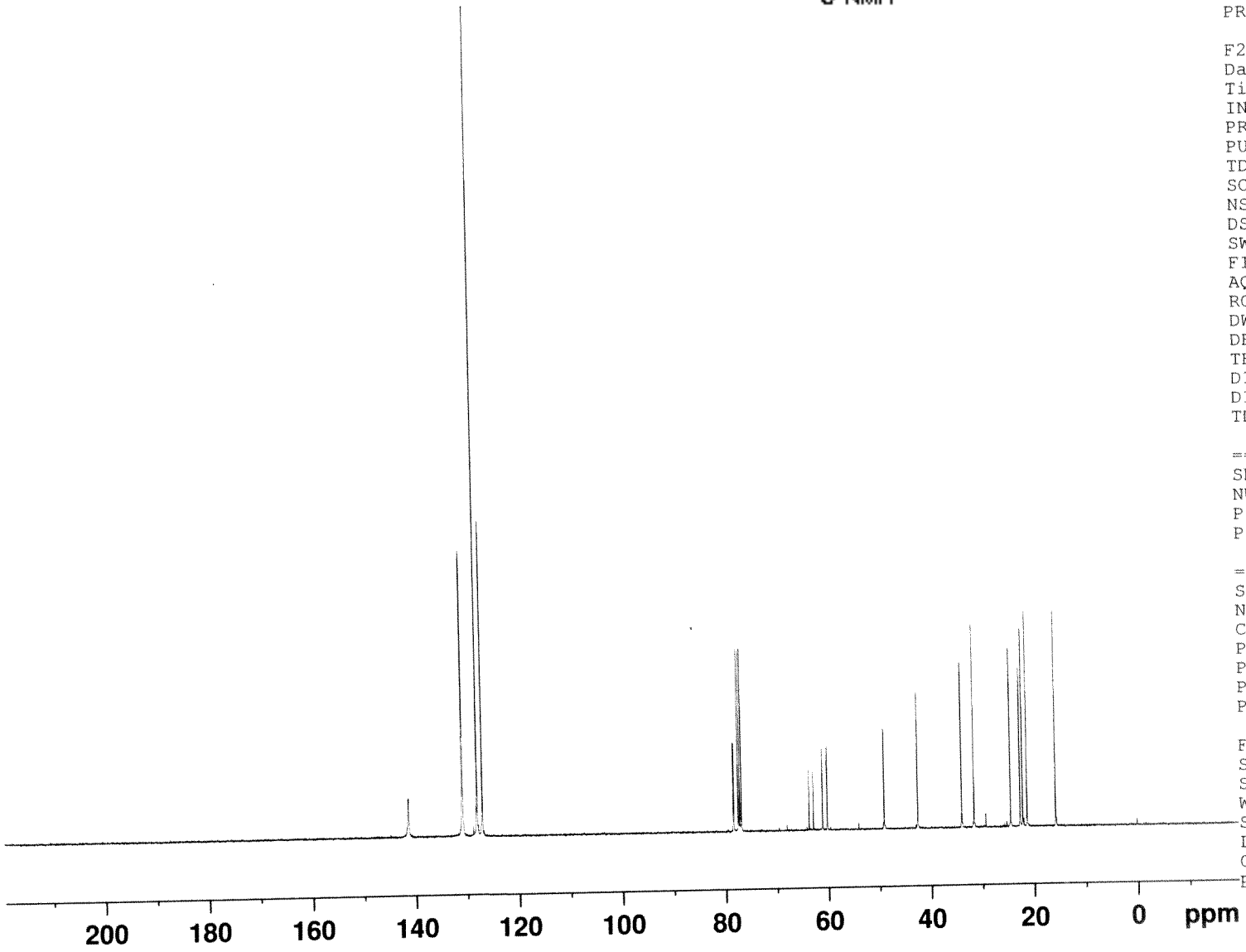
F2 - Acquisition Parameters
Date_ 20141120
Time 17.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 13.94
DW 62.400 usec
DE 6.50 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
PLW1 25.00300026 W

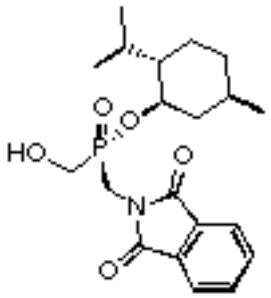
F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



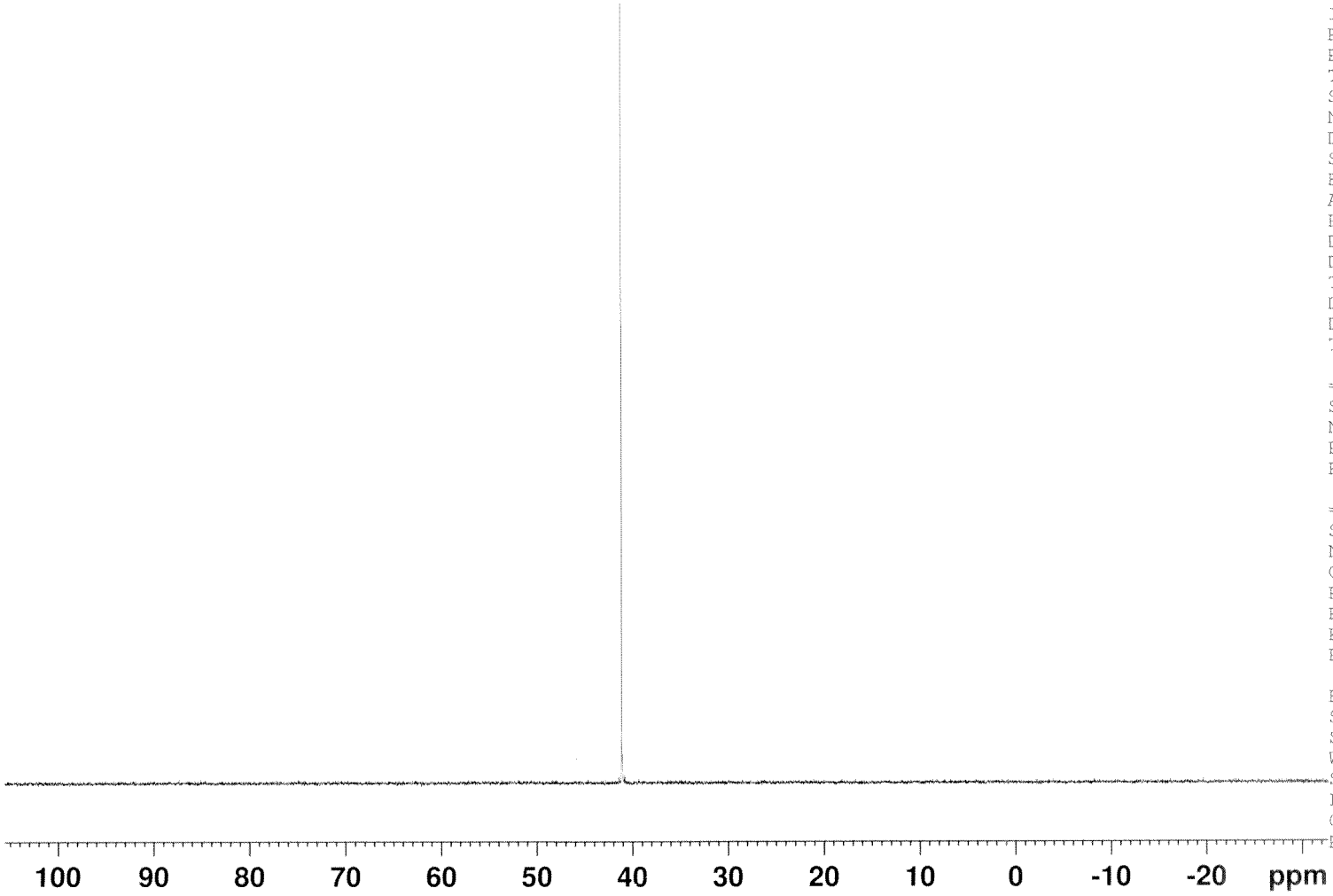
Compound (R_p)-13
^{13}C NMR



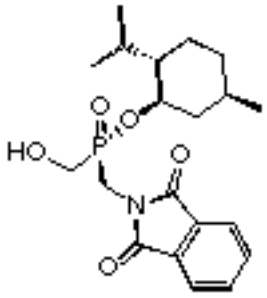

```
Current Data Parameters
NAME                  OB 1840f2
EXPNO                         4
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20141120
Time                      17.44
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDC13
NS                         682
DS                           4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631488 sec
RG                     203.57
DW                     20.800 usec
DE                      6.50 usec
TE                     296.9 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1           100.6228293 MHz
NUC1                     13C
P1                     10.00 usec
PLW1           45.00000000 W

======== CHANNEL f2 ========
SFO2           400.1316005 MHz
NUC2                     1H
CPDPRG[2             waltz16
PCPD2                  90.00 usec
PLW2           10.00000000 W
PLW12           0.31604999 W
PLW13           0.25600001 W

F2 - Processing parameters
SI                       32768
SF             100.6127685 MHz
WDW                         EM
SSB                  0
LB                     1.00 Hz
GB                   0
PC                     1.40
```



Compound (R_p)-14
$^{31}P\{^1H\}$ NMR decoupled

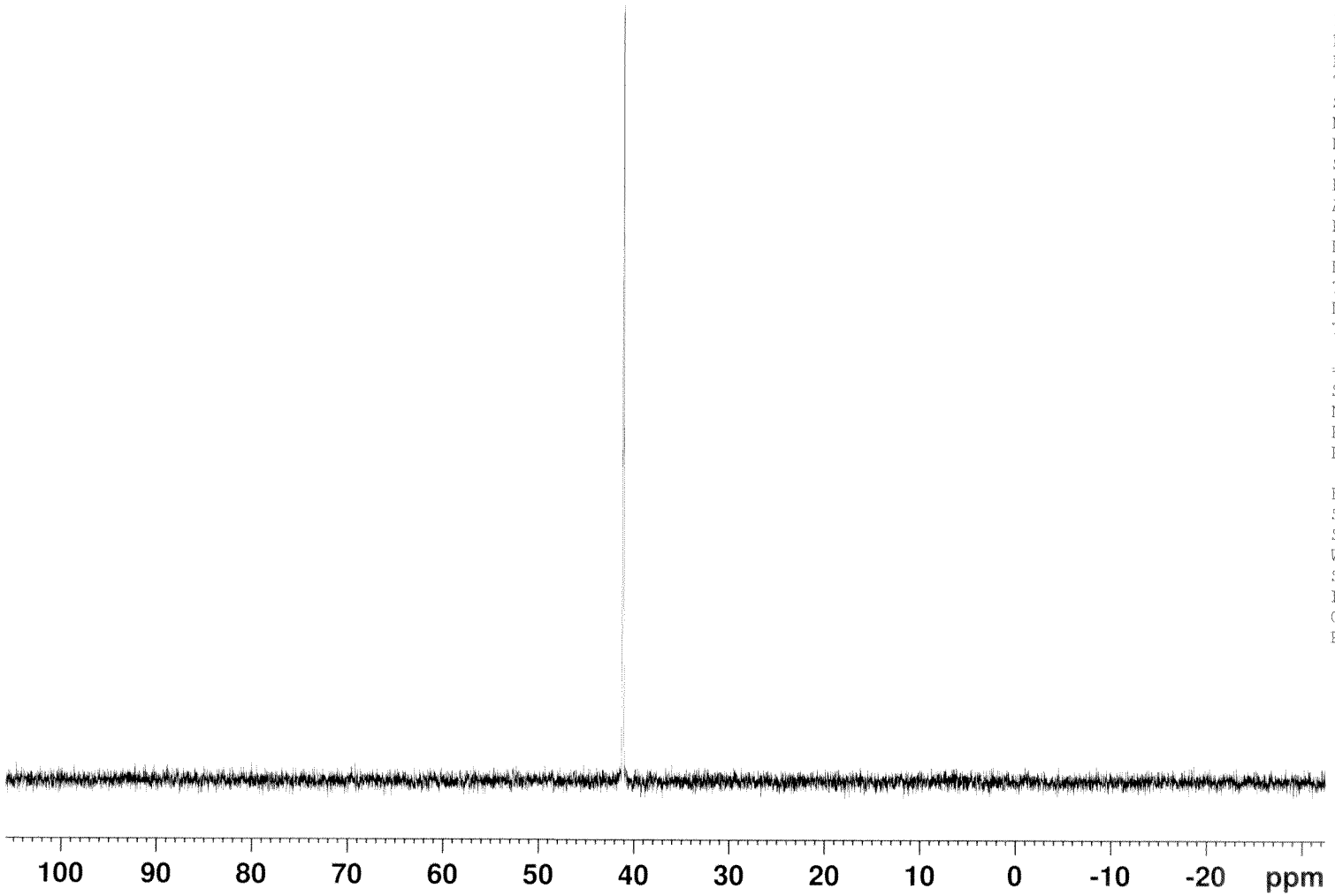


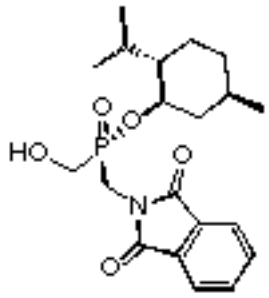

```
Current Data Parameters
NAME                  OB 1052
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20130510
Time                   10.02
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        4
SWH               64102.563 Hz
FIDRES             0.978127 Hz
AQ                0.5111808 sec
RG                   203.57
DW                    7.800 usec
DE                    6.50 usec
TE                   297.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1           161.9674942 MHz
NUC1                    31P
P1                    14.25 usec
PLW1           15.00000000 W

======== CHANNEL f2 ========
SFO2           400.1316005 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2                 90.00 usec
PLW2           10.00000000 W
PLW12           0.31604999 W
PLW13           0.25600001 W

F2 - Processing parameters
SI                      32768
SF             161.9755930 MHz
WDW                        EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.40
```



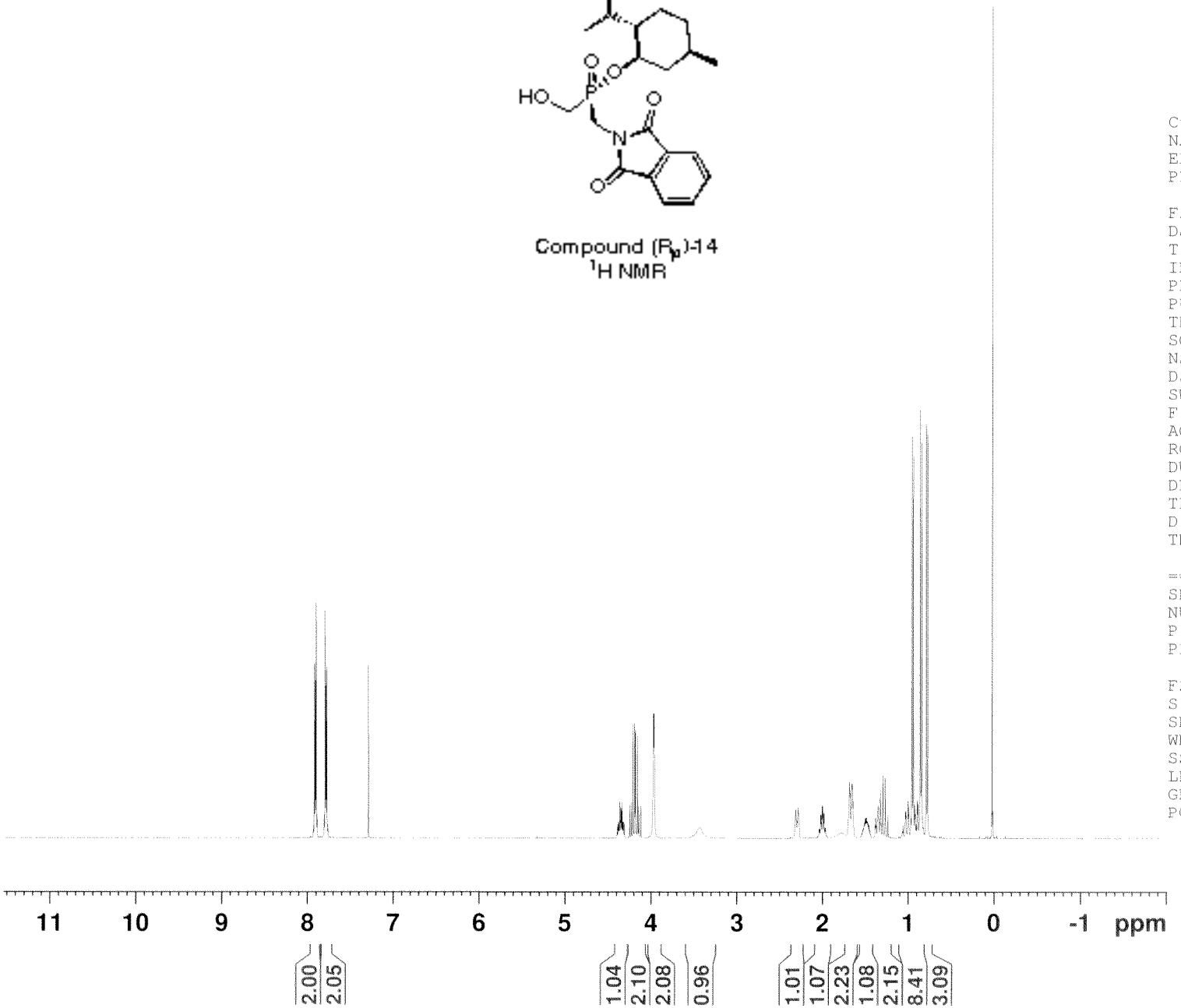
Compound (R_p)-14
$^{31}P\{^1H\}$ NMR coupled

```
Current Data Parameters
NAME                  OB 1052
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_               20130510
Time                   10.08
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        32
DS                         4
SWH               64102.563 Hz
FIDRES             0.978127 Hz
AQ                 0.5111808 sec
RG                    203.57
DW                     7.800 usec
DE                     6.50 usec
TE                     296.3 K
D1                2.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1            161.9674942 MHz
NUC1                    31P
P1                    14.25 usec
PLW1           15.00000000 W

F2 - Processing parameters
SI                     32768
SF              161.9755930 MHz
WDW                       EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.40
```

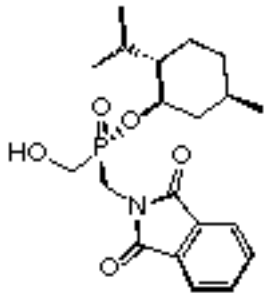




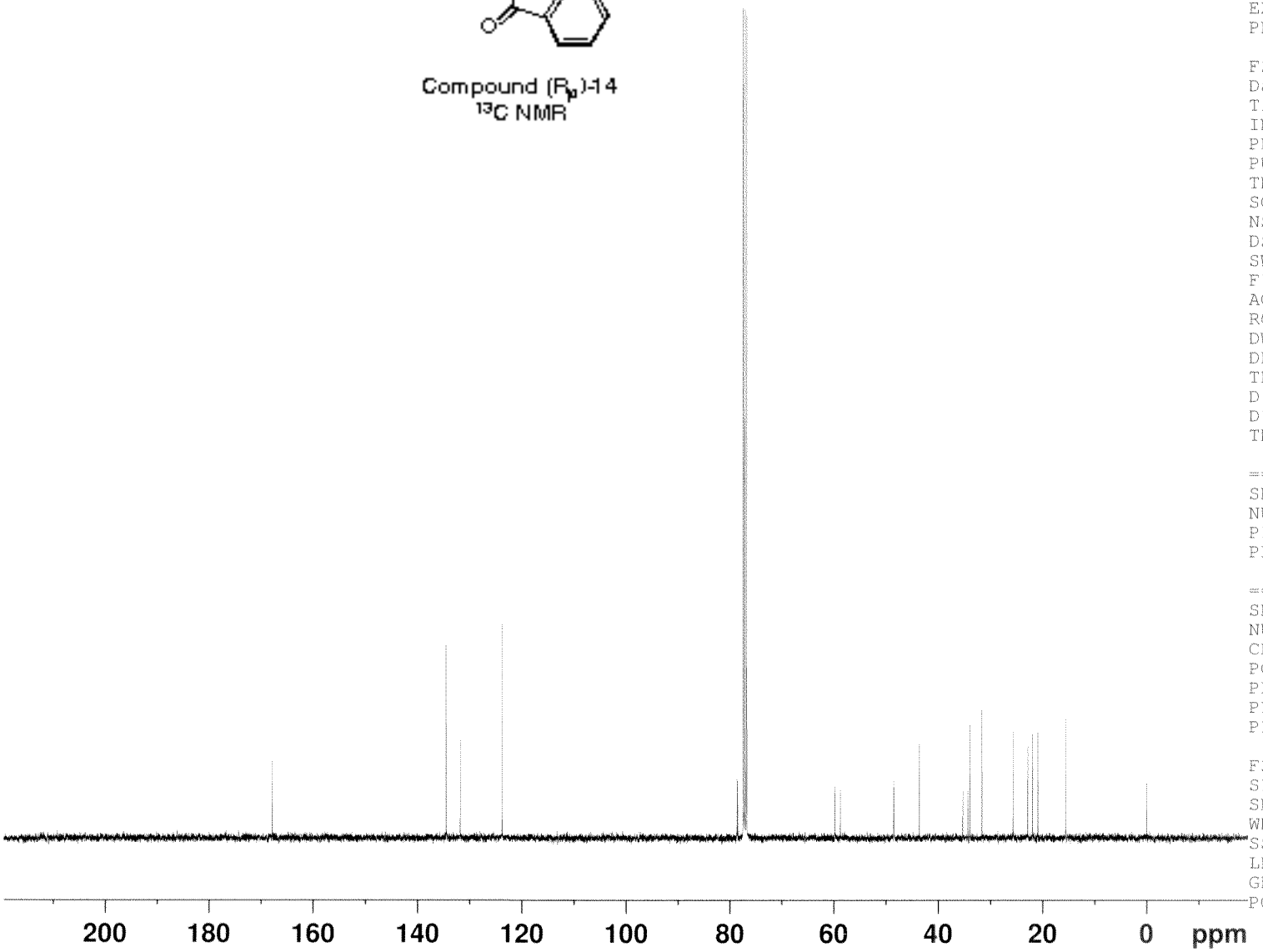
Compound (R_p)-14
^{1}H NMR



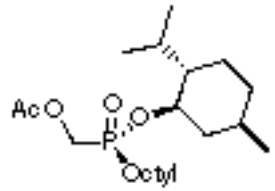

```
Current Data Parameters
NAME                  OB 1052
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameters
Date_               20130510
Time                   10.15
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                        16
DS                         2
SWH                 8012.820 Hz
FIDRES              0.122266 Hz
AQ                  4.0894465 sec
RG                      128.8
DW                     62.400 usec
DE                      6.50 usec
TE                     296.1 K
D1                1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1              400.1324710 MHz
NUC1                       1H
P1                     10.00 usec
PLW1             25.00300026 W

F2 - Processing parameters
SI                     65536
SF                400.1300000 MHz
WDW                       EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```



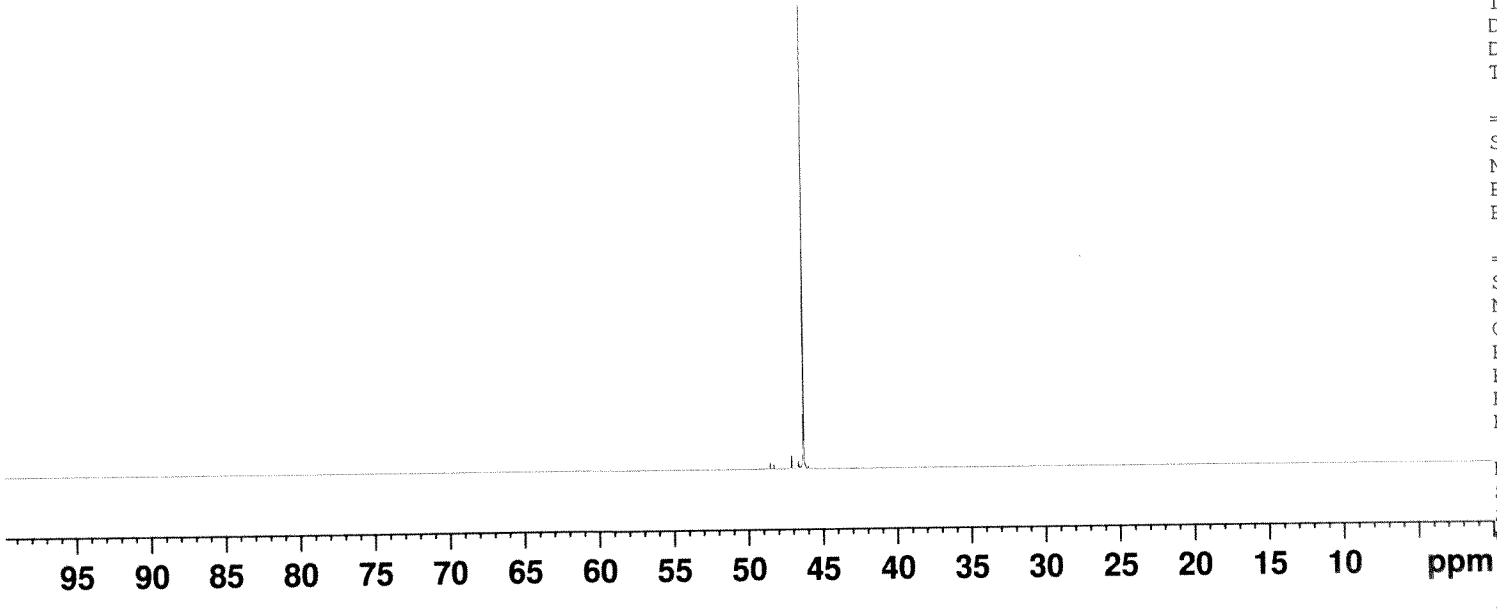
Compound (R_a)-14
^{13}C NMR



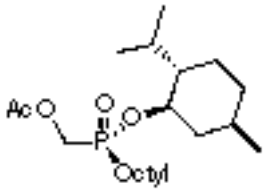

```
Current Data Parameters
NAME                  OB 1052
EXPNO                       4
PROCNO                      1

F2 - Acquisition Parameters
Date_               20130510
Time                   10.58
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      639
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631488 sec
RG                   203.57
DW                   20.800 usec
DE                    6.50 usec
TE                   297.6 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           100.6228293 MHz
NUC1                    13C
P1                   10.00 usec
PLW1           45.00000000 W

======== CHANNEL f2 ========
SFO2           400.1316005 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2           10.00000000 W
PLW12           0.31604999 W
PLW13           0.25600001 W

F2 - Processing parameters
SI                    32768
SF             100.6127690 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```



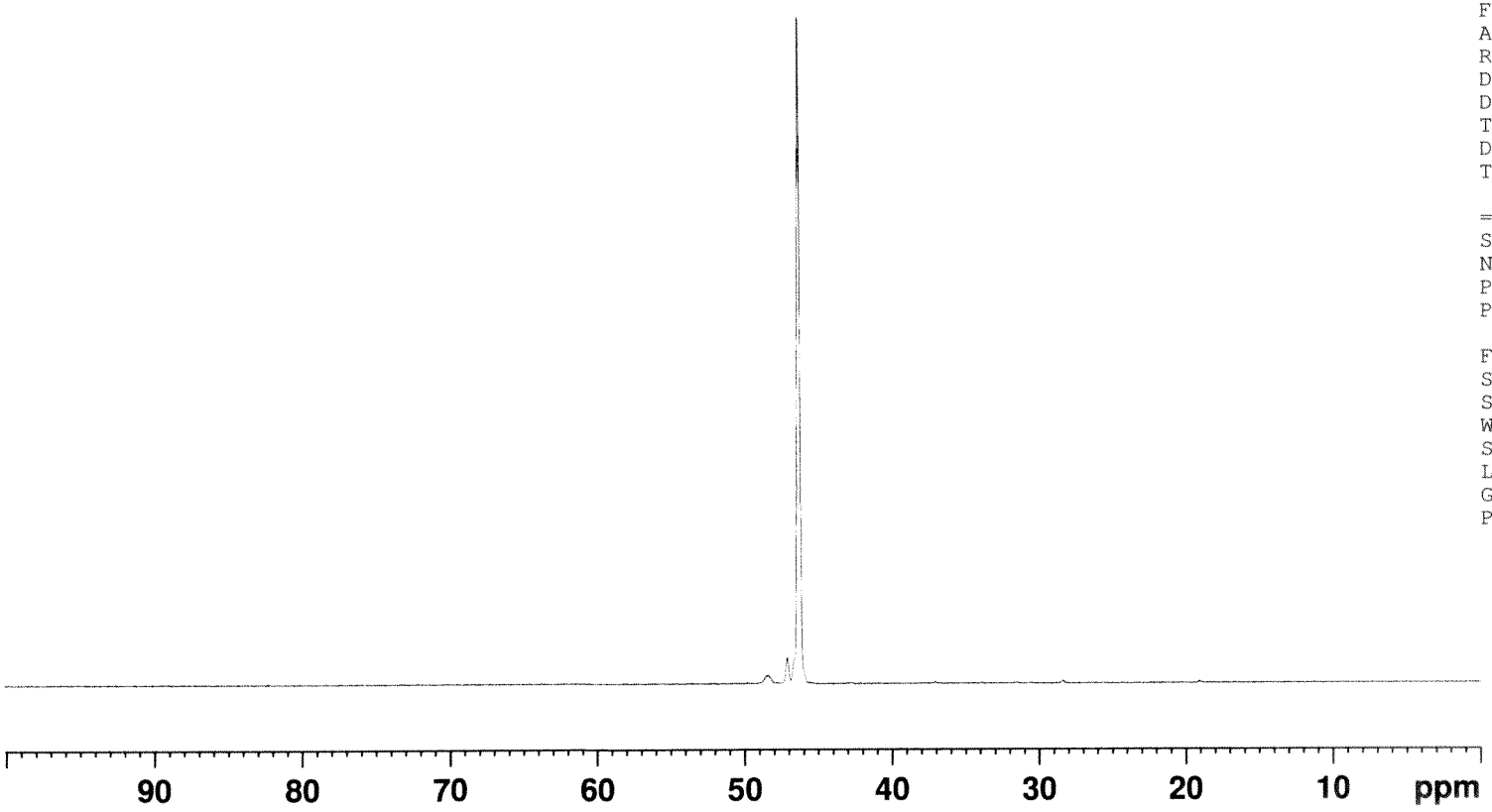
Compound (R_p)-15
$^{31}P\{^1H\}$ NMR decoupled



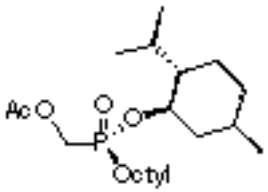

```
Current Data Parameters
NAME                  OB 1172
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20130717
Time                  20.05
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       4
SWH               64102.563 Hz
FIDRES             0.978127 Hz
AQ                 0.5111808 sec
RG                    203.57
DW                   7.800 usec
DE                   6.50 usec
TE                   294.6 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             161.9674942 MHz
NUC1                     31P
P1                     14.25 usec
PLW1             15.00000000 W

======== CHANNEL f2 ========
SFO2             400.1316005 MHz
NUC2                     1H
CPDPRG[2              waltz16
PCPD2                  90.00 usec
PLW2             10.00000000 W
PLW12             0.31604999 W
PLW13             0.25600001 W

F2 - Processing parameters
SI                       32768
SF               161.9755930 MHz
WDW                         EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```



Compound (R_p)-15
$^{31}P\{^1H\}$ NMR coupled

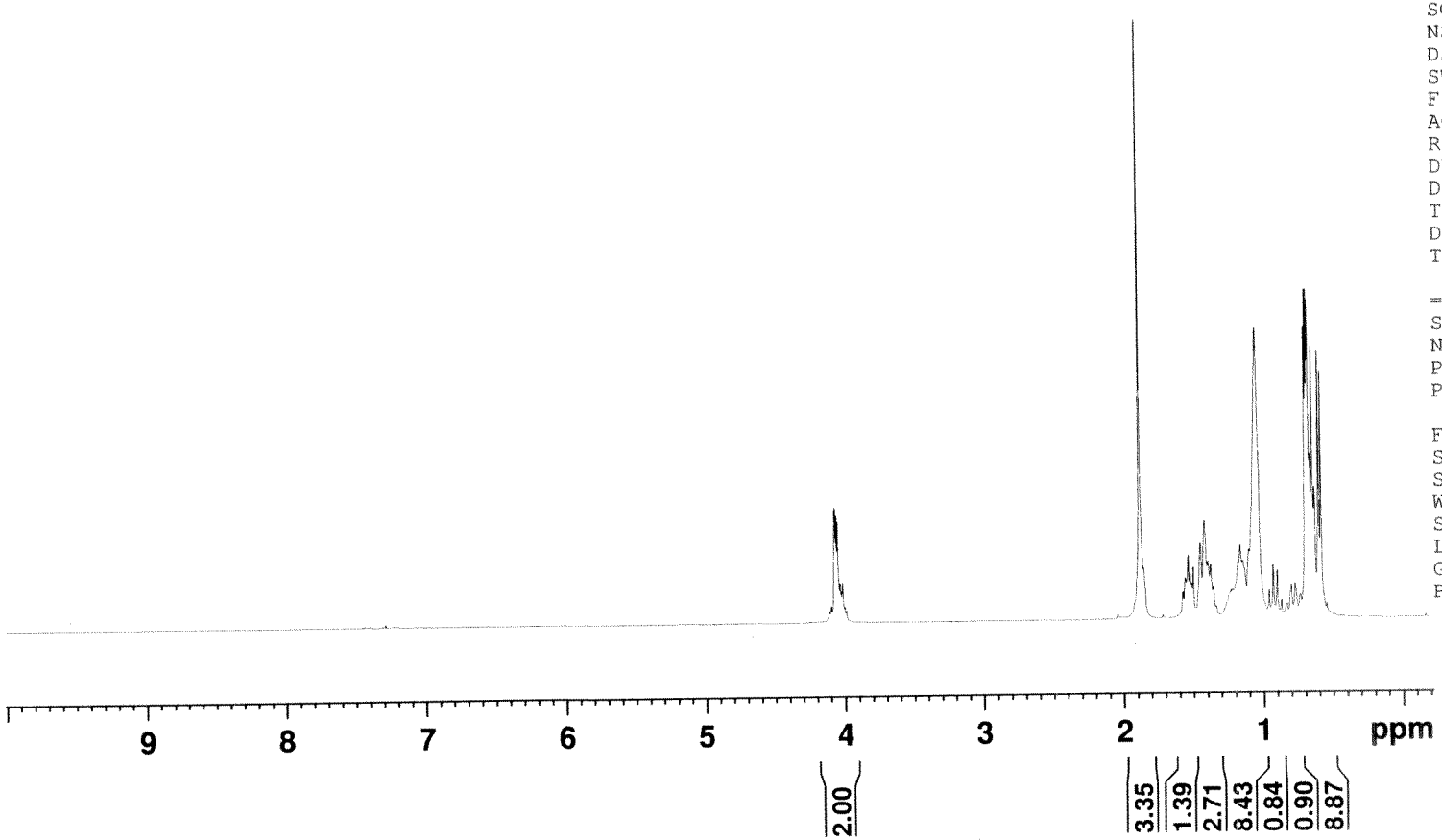


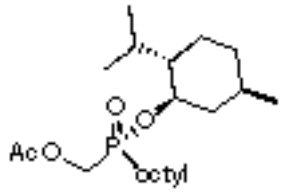

```
Current Data Parameters
NAME                  OB 1172
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20130717
Time                  20.11
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       32
DS                        4
SWH               64102.563 Hz
FIDRES             0.978127 Hz
AQ                0.5111808 sec
RG                   203.57
DW                    7.800 usec
DE                    6.50 usec
TE                   293.9 K
D1              2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           161.9674942 MHz
NUC1                   31P
P1                   14.25 usec
PLW1           15.00000000 W

F2 - Processing parameters
SI                    32768
SF             161.9755930 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```



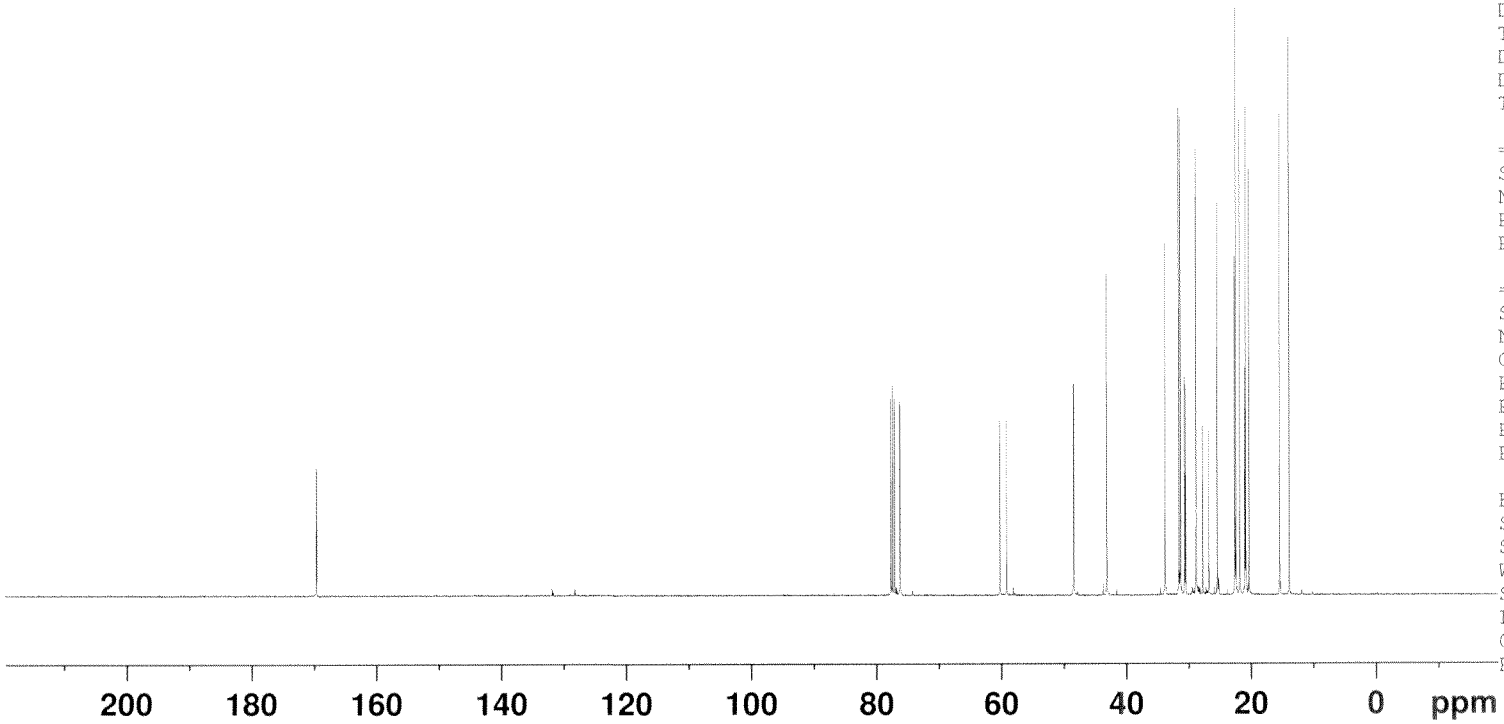
Compound (R_p)-15
^{1}H NMR

```
Current Data Parameters
NAME                  OB 1172
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameters
Date_              20130717
Time                  20.14
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894465 sec
RG                     6.28
DW                   62.400 usec
DE                    6.50 usec
TE                   293.9 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           400.1324710 MHz
NUC1                     1H
P1                   10.00 usec
PLW1           25.00300026 W

F2 - Processing parameters
SI                    65536
SF             400.1300000 MHz
WDW                      EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

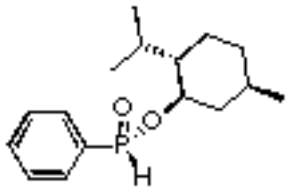




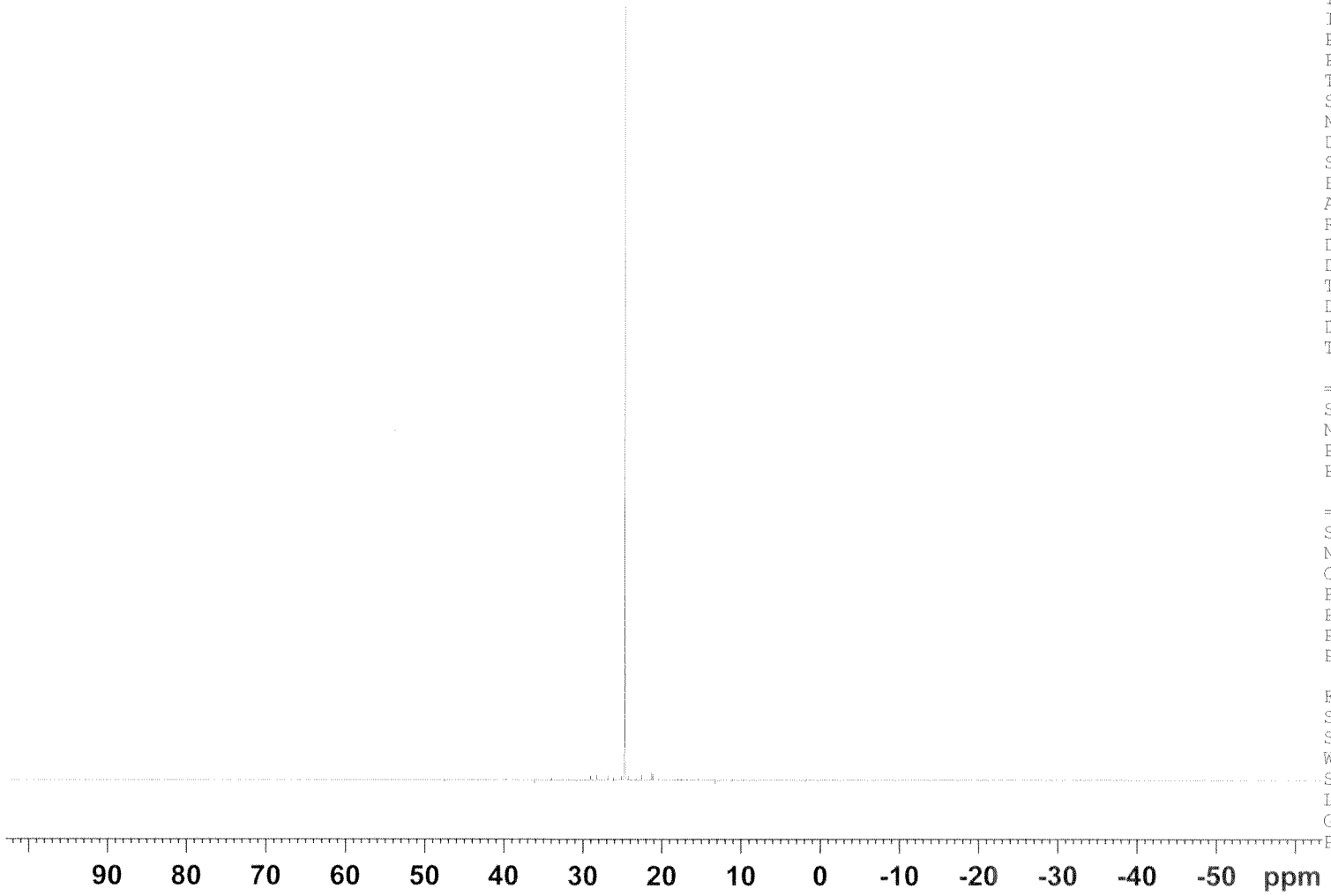
Compound (R_p)-15
^{13}C NMR



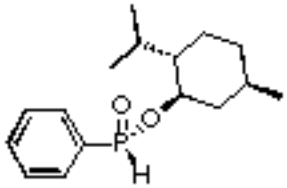

```
Current Data Parameters
NAME                  OB 1172
EXPNO                       4
PROCNO                      1

F2 - Acquisition Parameters
Date_               20130717
Time                   20.59
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        750
DS                          4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631488 sec
RG                     203.57
DW                     20.800 usec
DE                      6.50 usec
TE                     294.7 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

========== CHANNEL f1 ========
SFO1             100.6228293 MHz
NUC1                      13C
P1                     10.00 usec
PLW1            45.00000000 W

========== CHANNEL f2 ========
SFO2             400.1316005 MHz
NUC2                      1H
CPDPRG[2              waltz16
PCPD2                  90.00 usec
PLW2            10.00000000 W
PLW12            0.31604999 W
PLW13            0.25600001 W

F2 - Processing parameters
SI                        32768
SF               100.6127690 MHz
WDW                          EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```



Compound (R_p)-1
$^{31}P\{^1H\}$ NMR decoupled



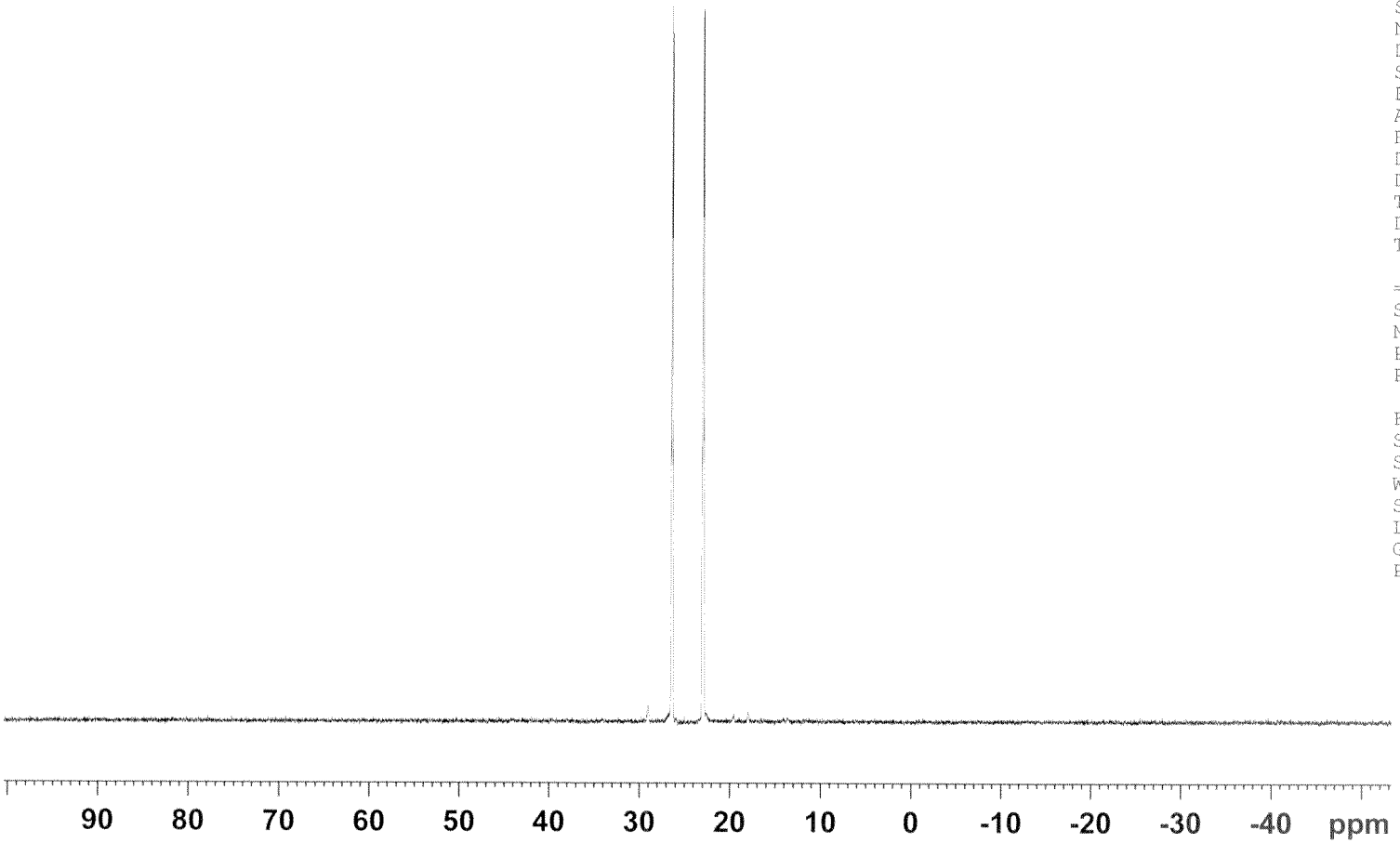

```
Current Data Parameters
NAME                  OB 2018
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20150429
Time                  17.03
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        4
SWH              64102.563 Hz
FIDRES            0.978127 Hz
AQ               0.5111808 sec
RG                   203.57
DW                    7.800 usec
DE                    6.50 usec
TE                   295.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                       1

========== CHANNEL f1 ==========
SFO1           161.9674942 MHz
NUC1                   31P
P1                   14.25 usec
PLW1           15.00000000 W

========== CHANNEL f2 ==========
SFO2           400.1316005 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2           10.00000000 W
PLW12           0.31604999 W
PLW13           0.25600001 W

F2 - Processing parameters
SI                    32768
SF             161.9755930 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

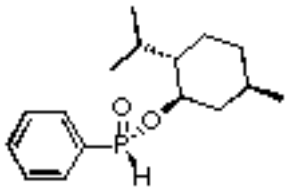


Compound (R_p)-1
$^{31}P\{^1H\}$ NMR coupled

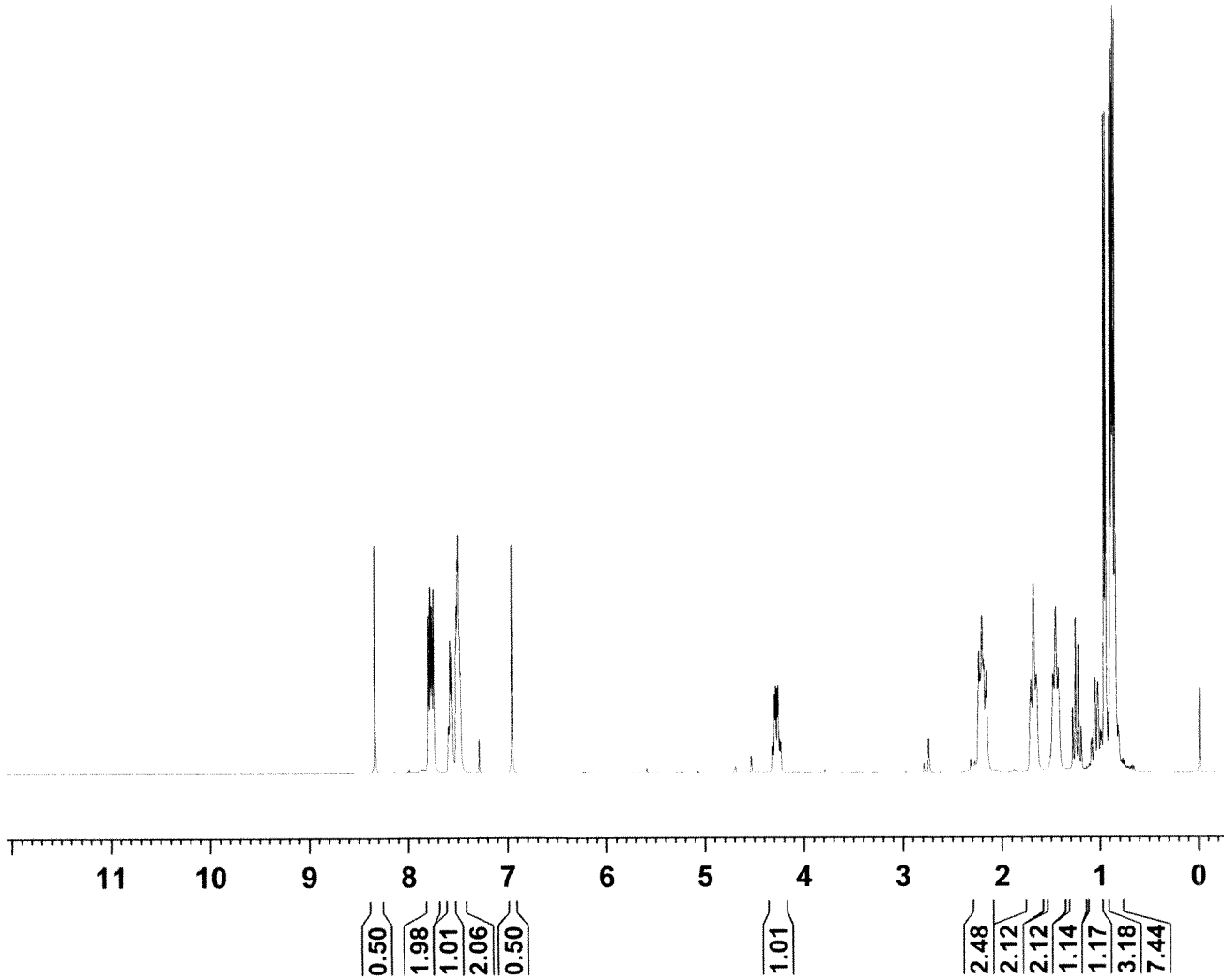
BRUKER

```
Current Data Parameters
NAME                  OB 2018
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20150429
Time                  17.05
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       32
DS                        4
SWH              64102.563 Hz
FIDRES            0.978127 Hz
AQ                0.5111808 sec
RG                   203.57
DW                   7.800 usec
DE                   6.50 usec
TE                   295.4 K
D1               2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           161.9674942 MHz
NUC1                   31P
P1                   14.25 usec
PLW1           15.00000000 W

F2 - Processing parameters
SI                    32768
SF             161.9755930 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```





Compound (R_p)-1
^{1}H NMR

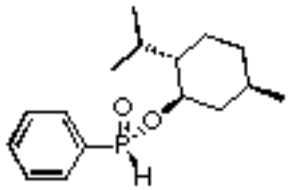


Current Data Parameters
NAME OB 2018
EXPNO 3
PROCNO 1

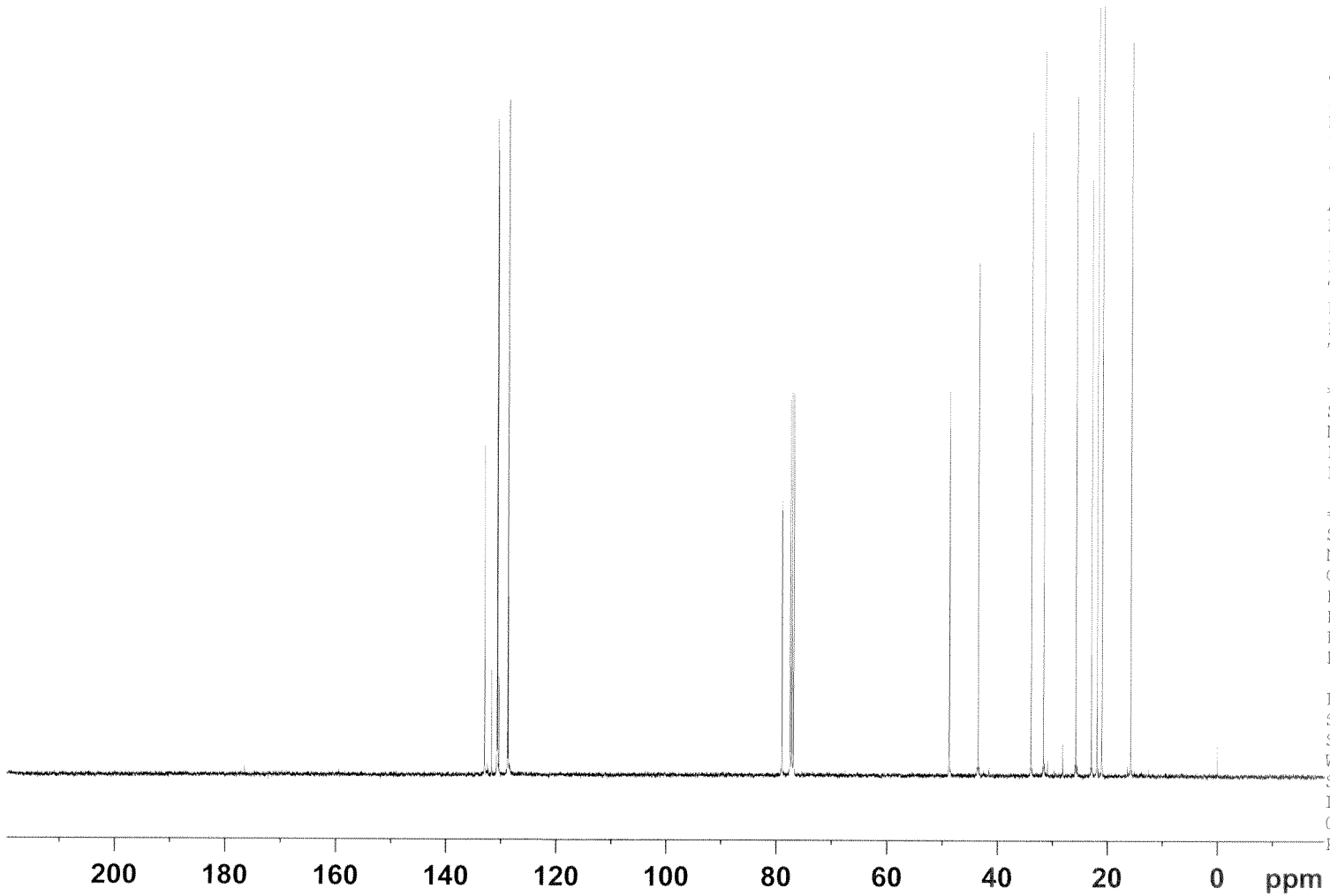
F2 - Acquisition Parameters
Date_ 20150429
Time 17.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 10
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 32.38
DW 62.400 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
PLW1 25.00300026 W

F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.60 Hz
GB 0
PC 1.00



Compound (R_p)-1
^{13}C NMR

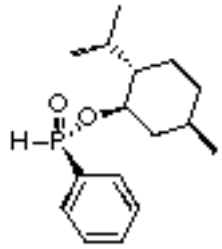



```
Current Data Parameters
NAME                  OB 2018
EXPNO                       4
PROCNO                      1

F2 - Acquisition Parameters
Date_              20150429
Time                  18.03
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      942
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631488 sec
RG                   203.57
DW                  20.800 usec
DE                   6.50 usec
TE                  295.2 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           100.6228293 MHz
NUC1                    13C
P1                   10.00 usec
PLW1           45.00000000 W

======== CHANNEL f2 ========
SFO2           400.1316005 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                90.00 usec
PLW2           10.00000000 W
PLW12           0.31604999 W
PLW13           0.25600001 W

F2 - Processing parameters
SI                    32768
SF             100.6127685 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```

08085

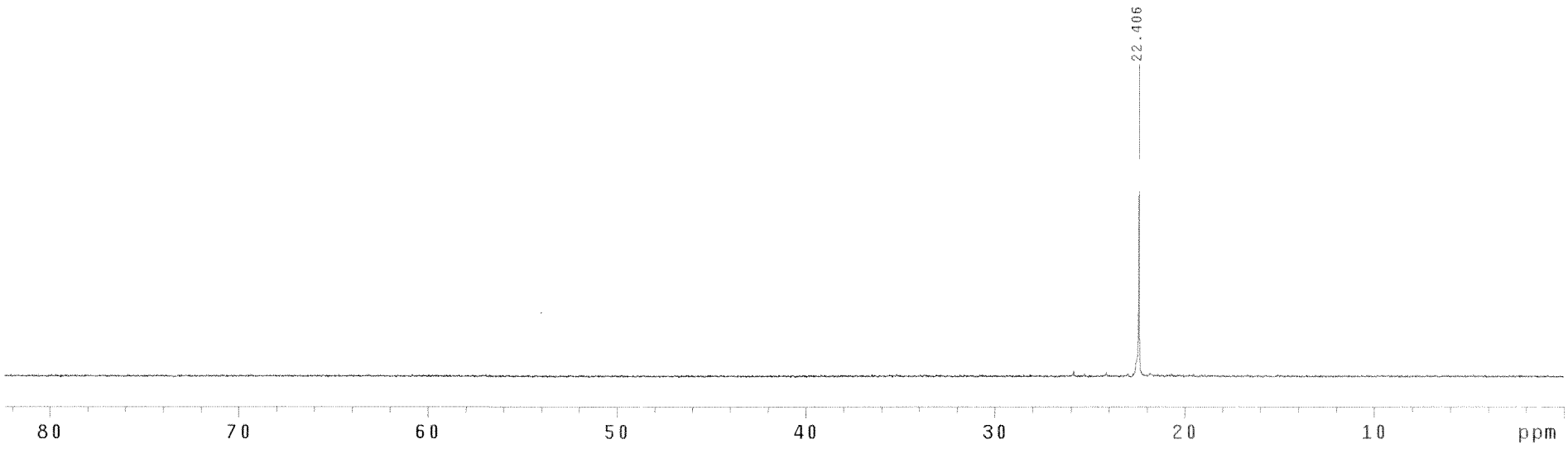
~~exp1 s2pul~~

SAMPLE		SPECIAL	
date	Oct 24 2011	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	16	in	n
ss	4	dp	y
d1	1.000	hs	nn
nt	64	PROCESSING	
ct	32	lb	1.00
TRANSMITTER		fn	not used
tn	P31	DISPLAY	
sfrq	121.471	sp	-0.2
tof	10598.6	wp	10910.0
tpwr	55	rfl	2437.5
pw	7.117	rfp	0
DECOUPLER		rp	-140.4
dn	H1	lp	-288.8
dof	0	PLOT	
dm	yyy	wc	250
dmm	w	sc	0
dpwr	35	vs	5
dmf	6700	th	4
		ai	no ph



Compound (S_p)-1

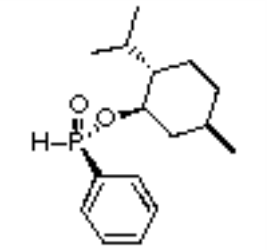
$^{31}P\{^1H\}$ NMR decoupled



08085

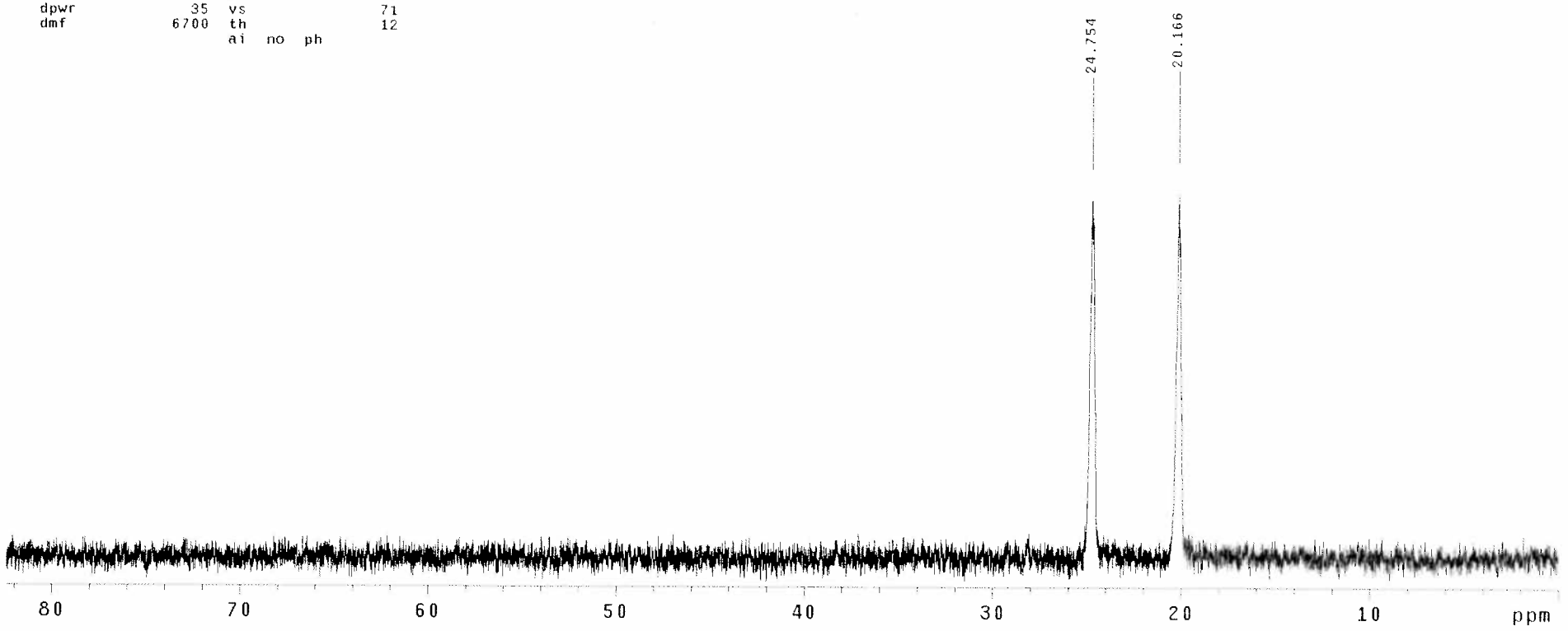
~~exp1 s2pul~~

SAMPLE		SPECIAL	
date	Oct 24 2011	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	16	in	n
ss	4	dp	y
dl	1.000	hs	nn
nt	64	**PROCESSING**	
ct	32	lb	1.00
TRANSMITTER		fn	not used
tn	P31	**DISPLAY**	
sfrq	121.471	sp	-0.2
tof	10598.6	wp	10010.0
tpwr	55	rfl	2437.5
pw	7.117	rfp	0
DECOUPLER		rp	-135.0
dn	H1	lp	-326.4
dof	0	**PLOT**	
dm	ynn	wc	250
dmm	w	sc	0
dpwr	35	vs	71
dmf	6700	th	12
		ai no ph	



Compound (S_p)-1

$^{31}P\{^{1}H\}$ NMR coupled

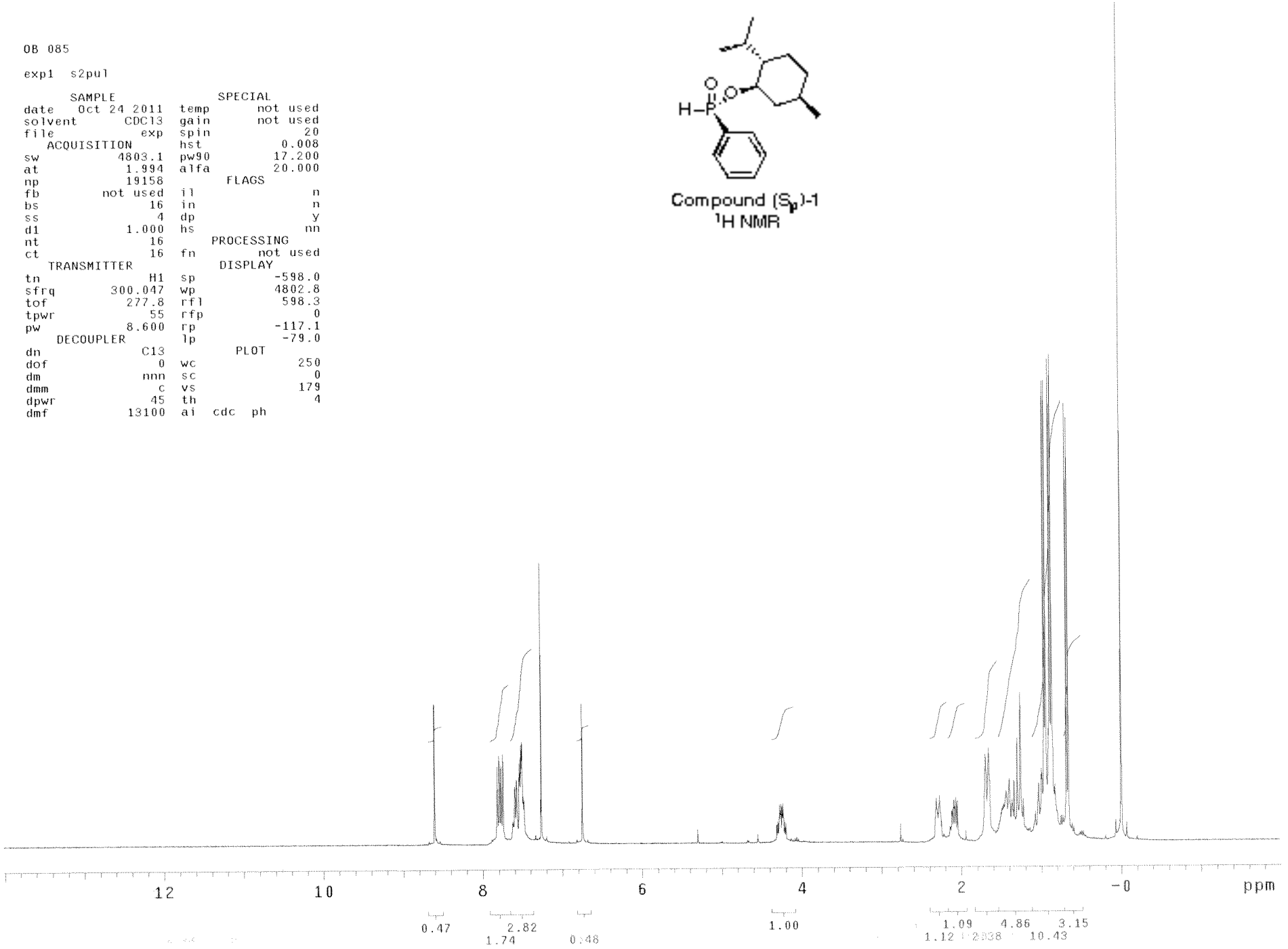


OB 085

exp1 s2pul

SAMPLE		SPECIAL	
date	Oct 24 2011	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	16	PROCESSING	
ct	16	fn	not used
TRANSMITTER		DISPLAY	
tn	H1	sp	-598.0
sfrq	300.047	wp	4802.8
tof	277.8	rfl	598.3
tpwr	55	rfp	0
pw	8.600	rp	-117.1
DECOUPLER		lp	-79.0
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	179
dpwr	45	th	4
dmf	13100	ai cdc ph	

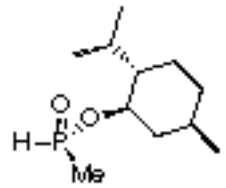
Compound (S_p)-1
^{1}H NMR



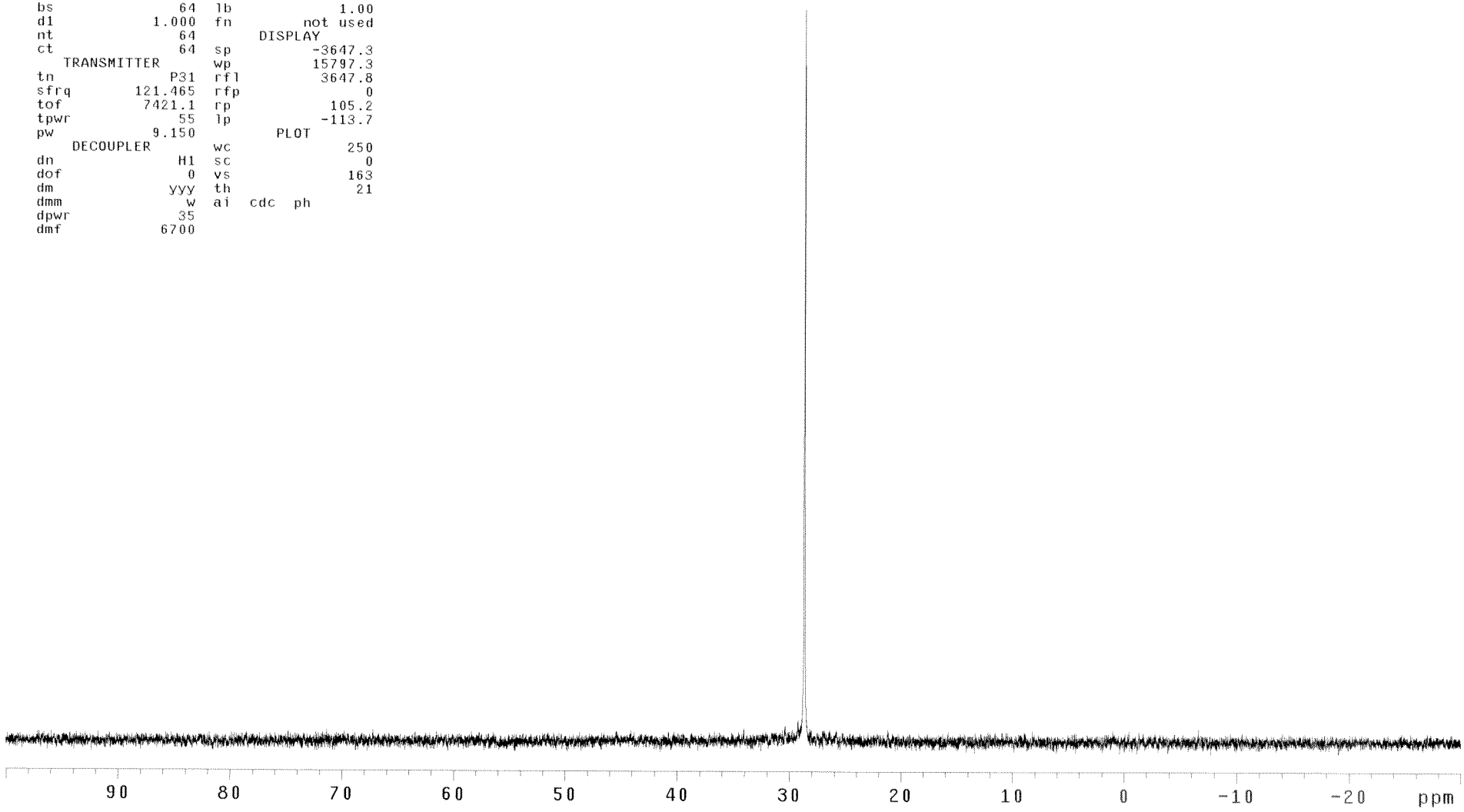
OB 953
pad=10 run with findz0 before acquisitio
n

exp10 Phosphorus

SAMPLE		SPECIAL	
date	Feb 14 2013	temp	not used
solvent	cdcl3	gain	25
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2013.02.14/s_2013~		pw90	18.300
0214_12/data/cdcl3~		alfa	10.000
01.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	64	DISPLAY	
ct	64	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	105.2
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	163
dm	yyy	th	21
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



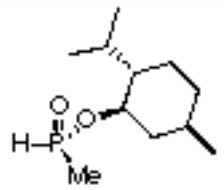
Compound (S_p)-16
$^{31}P\{^1H\}$ NMR decoupled



OB 953

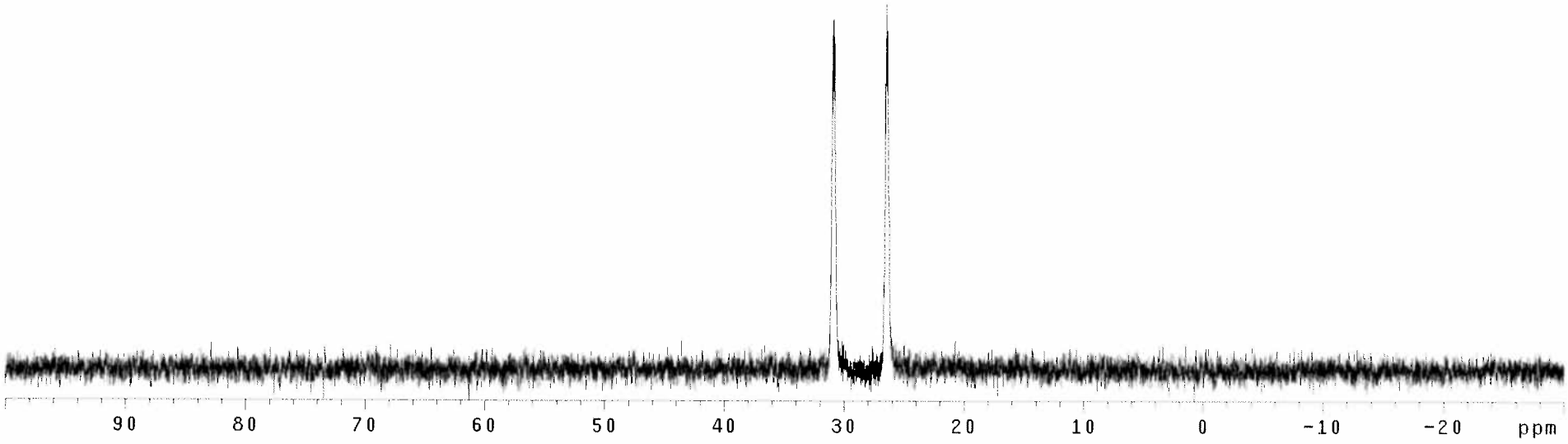
exp10 Phosphorus

SAMPLE		SPECIAL	
date	Feb 14 2013	temp	not used
solvent	cdcl3	gain	25
file	/home/TCUuser~	spin	20
	/vnmrsys/data/auto~	hst	0.008
	_2013.02.14/s_2013~	pw90	18.300
	0214_12/data/cdcl3~	alfa	10.000
	_02.fid	FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	64	DISPLAY	
ct	64	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	113.7
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	357
dm	ynn	th	12
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		

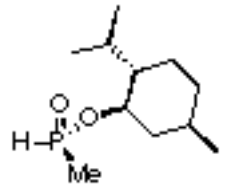


Compound (S_p)-16

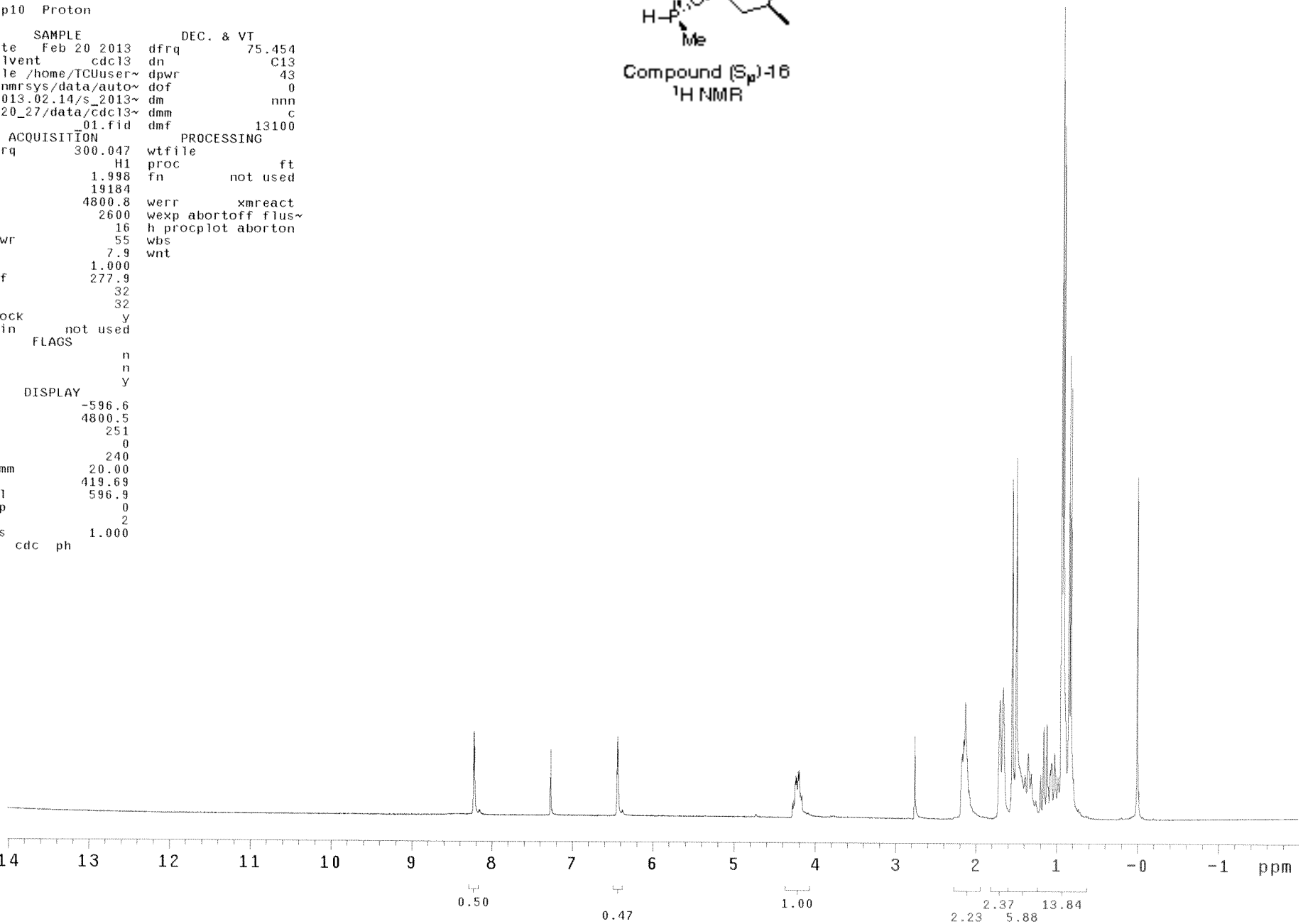
$^{31}P\{^{1}H\}$ NMR coupled



```
OB 970
pad=10 run with findz0 before acquisitio
n

exp10  Proton

      SAMPLE
date   Feb 20 2013
solvent      cdcl3
file /home/TCUuser~
/vnmrsys/data/auto~
_2013.02.14/s_2013~
0220_27/data/cdcl3~
            01.fid
   ACQUISITION
sfrq       300.047
tn              H1
at             1.998
np             19184
sw            4800.8
fb              2600
bs                16
tpwr              55
pw               7.9
d1             1.000
tof            277.9
nt                32
ct                32
alock              y
gain       not used
      FLAGS
il                 n
in                 n
dp                 y
     DISPLAY
sp            -596.6
wp            4800.5
vs               251
sc                 0
wc               240
hzmm           20.00
is            419.69
rfl            596.9
rfp                0
th                 2
ins            1.000
ai  cdc  ph

      DEC. & VT
dfrq         75.454
dn              C13
dpwr             43
dof               0
dm              nnn
dmm               c
dmf           13100
      PROCESSING
wtfile
proc              ft
fn         not used

werr        xmreact
wexp abortoff flus~
h procplot aborton
wbs
wnt
```



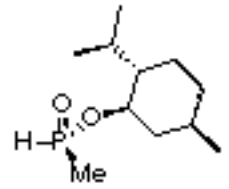
Compound (S_p)-16
^{1}H NMR



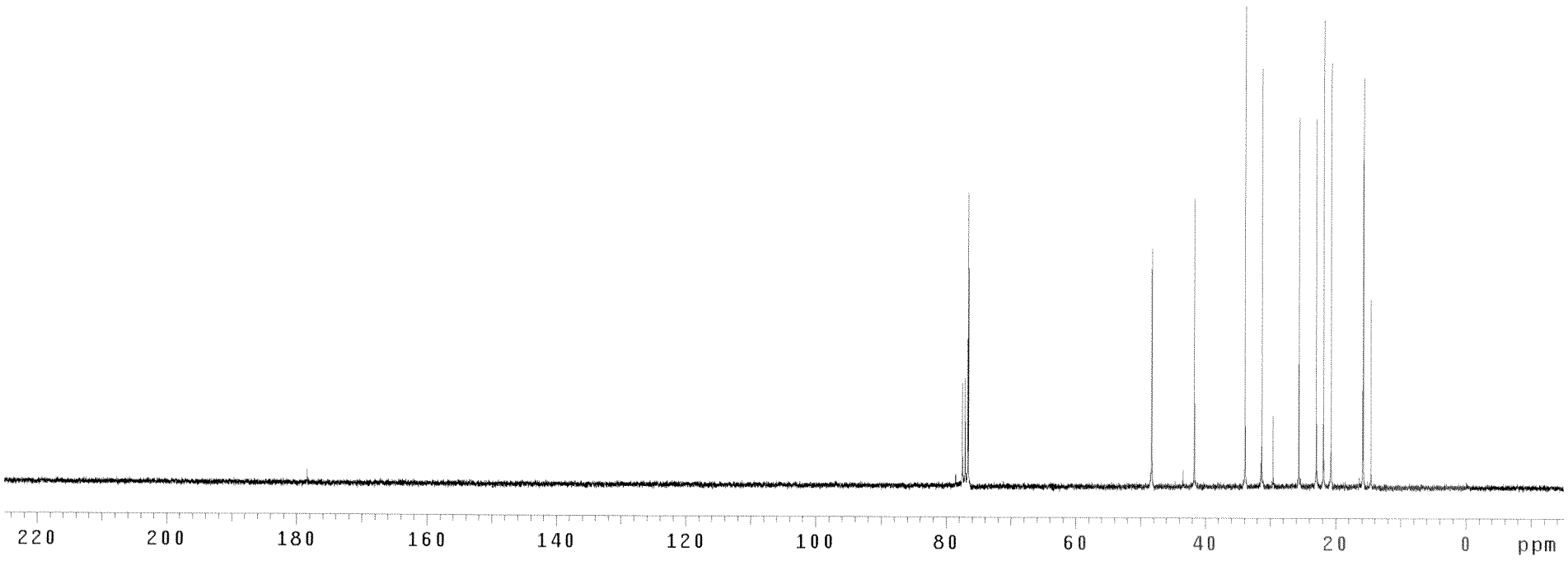
OB 970

exp10 Carbon

SAMPLE		SPECIAL	
date	Feb 20 2013	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2013.02.14/s_2013~		pw90	18.500
0220_28/data/cdcl3~		alfa	10.000
_06.fid		FLAGS	
ACQUISITION		il	n
sw	18115.9	in	n
at	1.301	dp	y
np	47120	hs	nn
fb	10000	PROCESSING	
bs	64	lb	0.50
d1	2.000	fn	not used
nt	800	DISPLAY	
ct	800	sp	-1135.5
TRANSMITTER		wp	18115.4
tn	C13	rfl	1136.1
sfrq	75.454	rfp	0
tof	766.0	rp	55.8
tpwr	58	lp	-160.4
pw	9.250	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	256
dm	yyy	th	4
dmm	w	ai cdc ph	
dpwr	35		
dmf	6700		



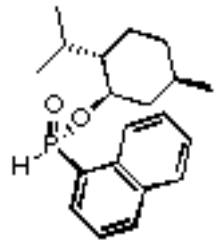
Compound (S_p)-16
^{13}C NMR



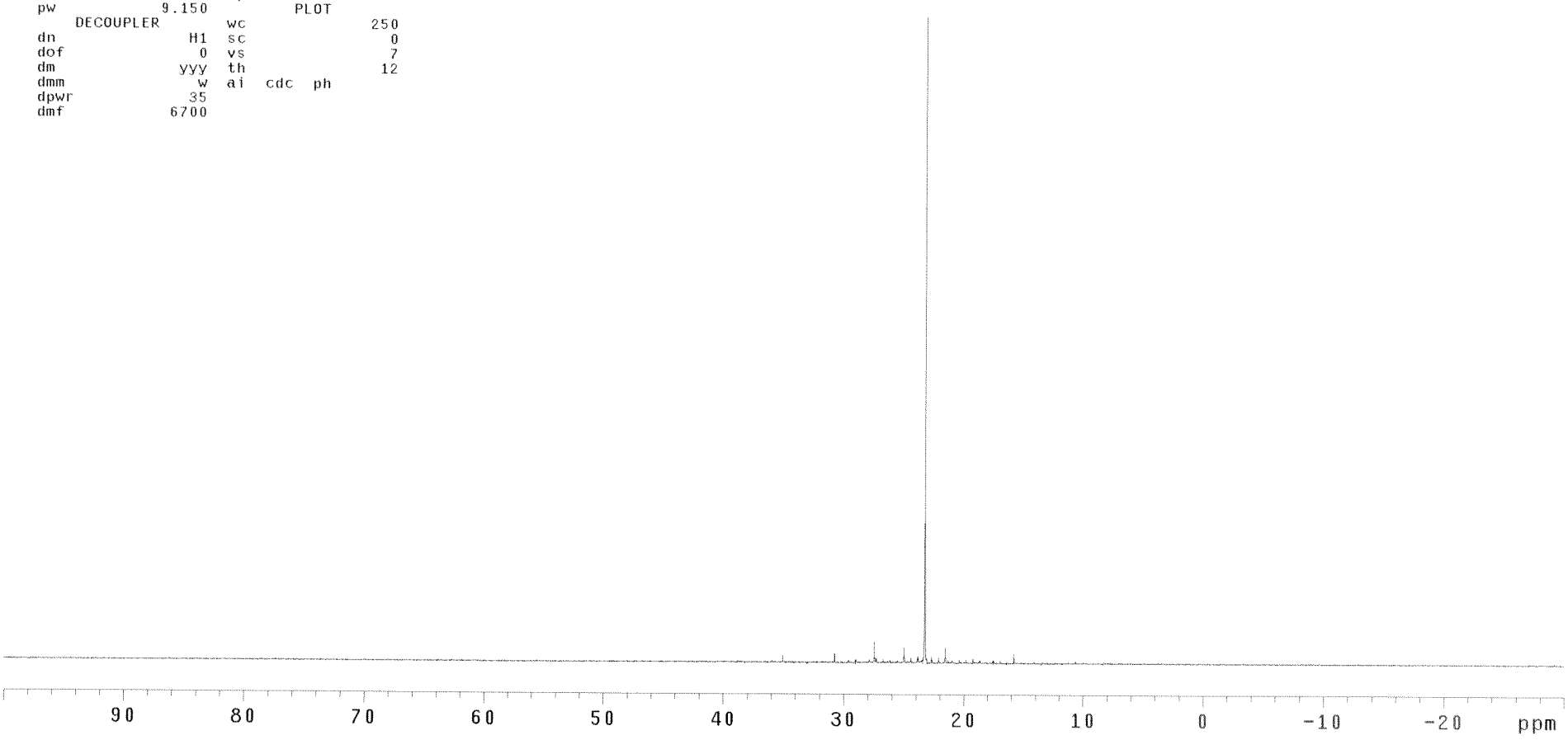
OB 949
pad=10 run with findz0 before acquisitio
n

exp1 Phosphorus

SAMPLE		SPECIAL	
date	Feb 9 2013	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2013.02.09/s_2013~		pw90	18.300
0209_05/data/cdcl3~		alfa	10.000
02.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	136.0
tpwr	55	lp	-124.4
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	7
dm	yyy	th	12
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



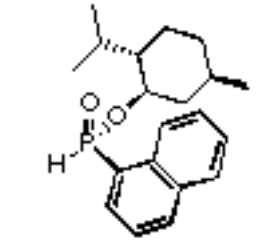
Compound (S_p)-17
$^{31}P\{^1H\}$ NMR decoupled



OB 949

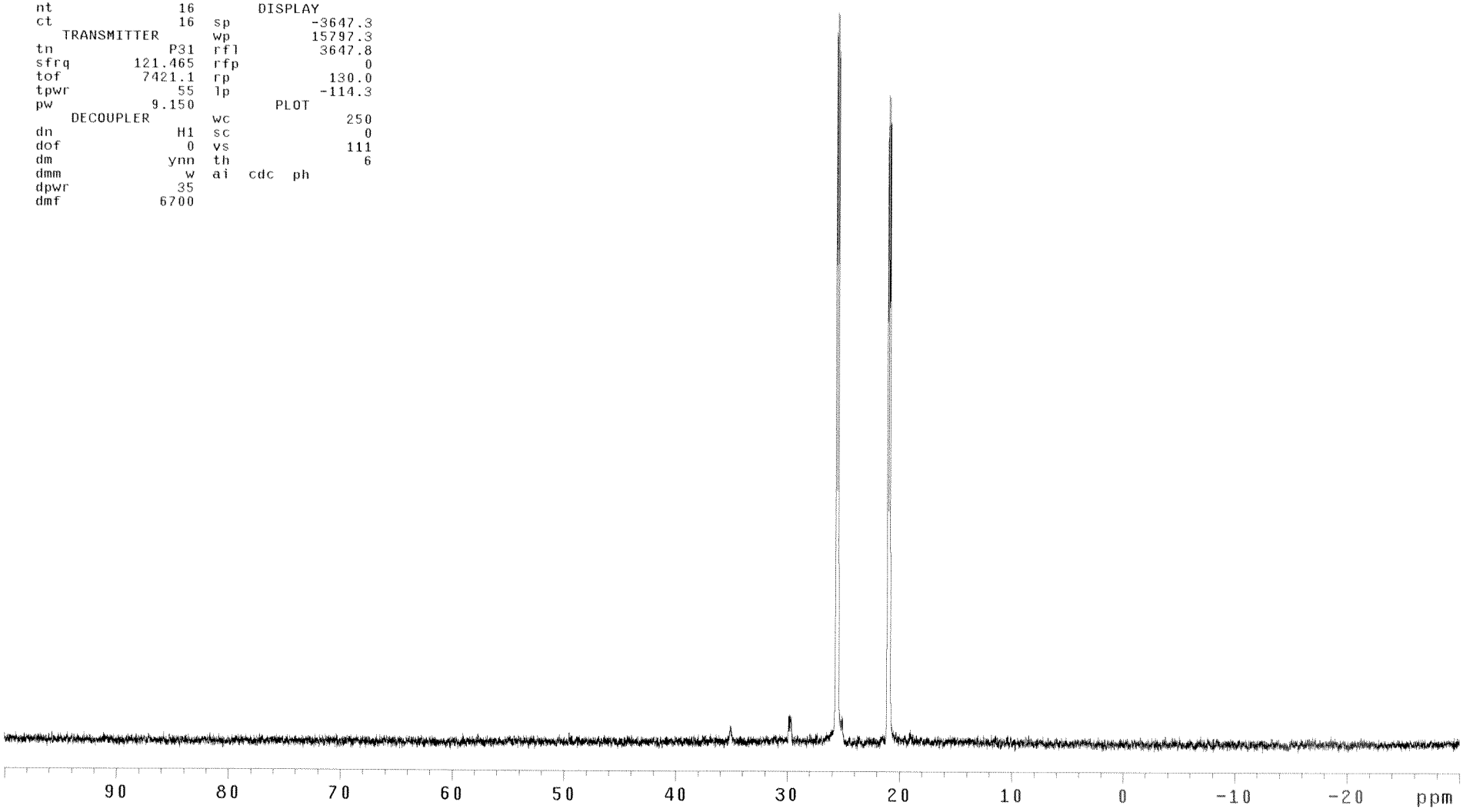
exp1 Phosphorus

SAMPLE		SPECIAL	
date	Feb 9 2013	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
	/vnmrsys/data/auto~	hst	0.008
	_2013.02.09/s_2013~	pw90	18.300
	0209_05/data/cdcl3~	alfa	10.000
	_04.fid	FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	130.0
tpwr	55	lp	-114.3
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	111
dm	ynn	th	6
dmm	w	ai cdc ph	
dpwr	35		
dmf	6700		



Compound (S_p)-17

$^{31}P\{^1H\}$ NMR coupled

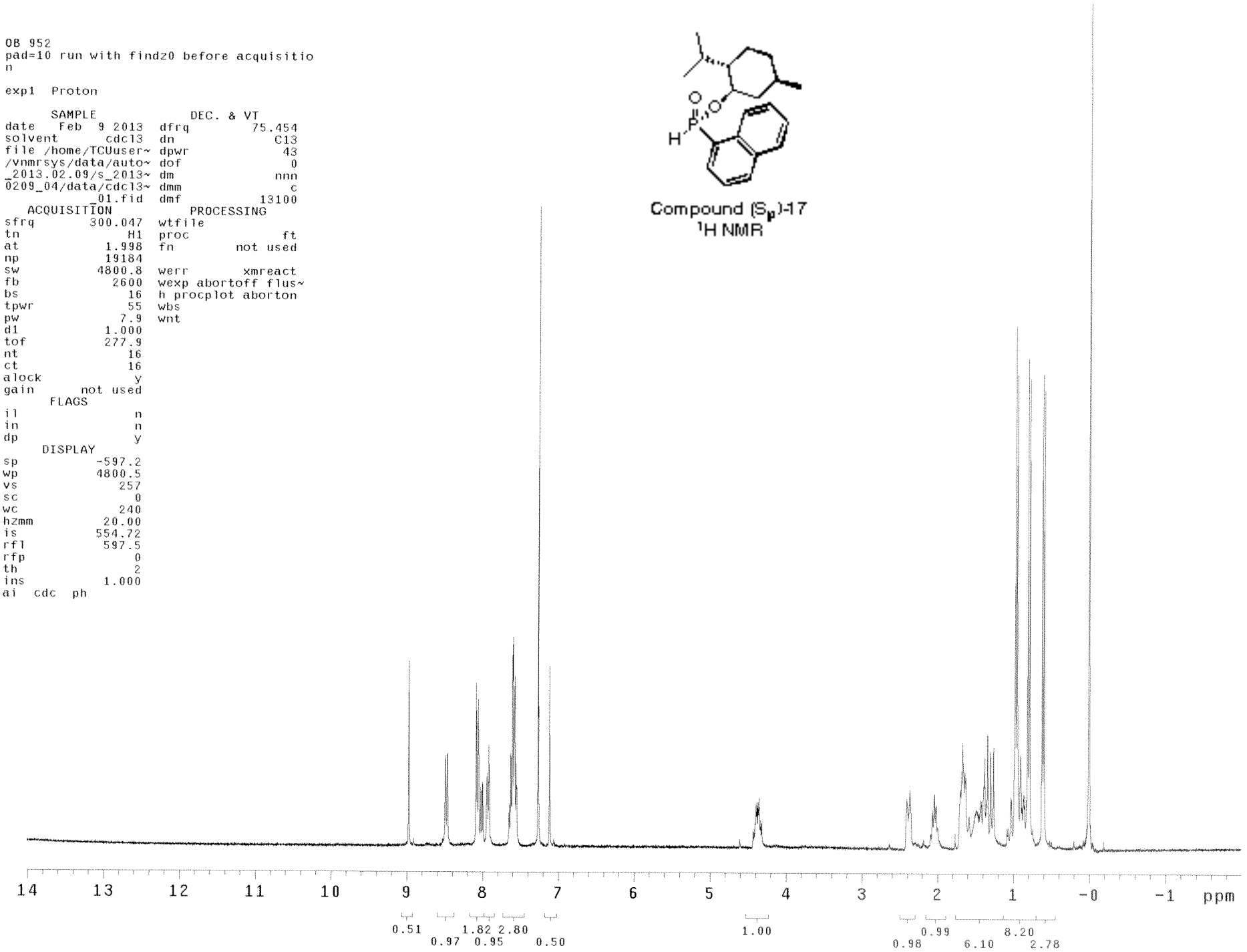


OB 952
pad=10 run with findz0 before acquisitio
n

exp1 Proton

SAMPLE		DEC. & VT	
date	Feb 9 2013	dfrq	75.454
solvent	cdcl3	dn	C13
file	/home/TCUuser~	dpwr	43
	/vnmrsys/data/auto~	dof	0
	_2013.02.09/s_2013~	dm	nnn
	0209_04/data/cdcl3~	dmm	c
	01.fid	dmf	13100
ACQUISITION		**PROCESSING**	
sfrq	300.047	wtfile	
tn	H1	proc	ft
at	1.998	fn	not used
np	19184		
sw	4800.8	werr	xmreact
fb	2600	wexp	abortoff flus~
bs	16	h procplot	aborton
tpwr	55	wbs	
pw	7.9	wnt	
d1	1.000		
tof	277.9		
nt	16		
ct	16		
alock	y		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-597.2		
wp	4800.5		
vs	257		
sc	0		
wc	240		
hzmm	20.00		
is	554.72		
rfl	597.5		
rfp	0		
th	2		
ins	1.000		
ai cdc ph			

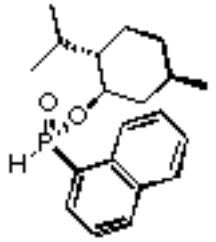
Compound (S_p)-17
^{1}H NMR



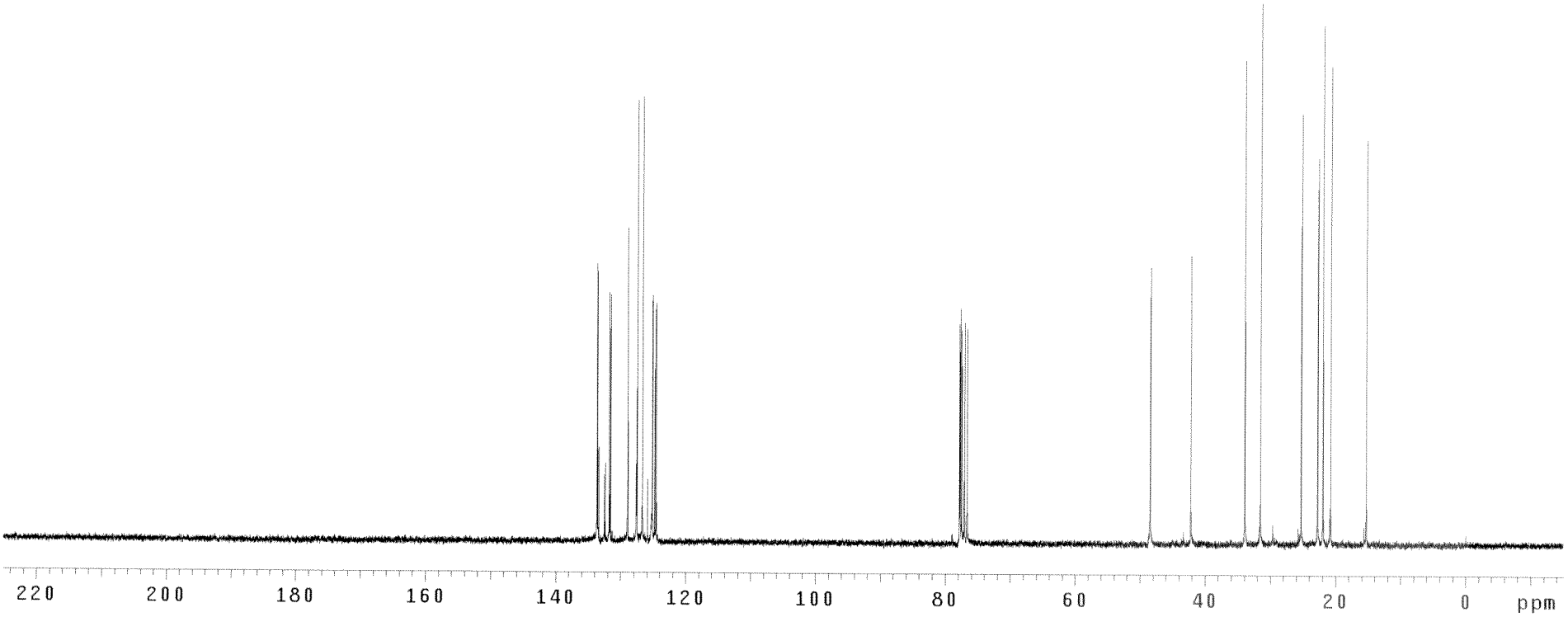
OB 949

exp1 Carbon

SAMPLE		SPECIAL	
date	Feb 9 2013	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2013.02.09/s_2013~		pw90	18.500
0209_05/data/cdcl3~		alfa	10.000
_05.fid		FLAGS	
ACQUISITION		il	n
sw	18115.9	in	n
at	1.301	dp	y
np	47120	hs	nn
fb	10000	PROCESSING	
bs	64	lb	0.50
d1	2.000	fn	not used
nt	1600	DISPLAY	
ct	1600	sp	-1135.5
TRANSMITTER		wp	18115.4
tn	C13	rfl	1136.1
sfrq	75.454	rfp	0
tof	766.0	rp	61.2
tpwr	58	lp	-197.6
pw	9.250	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	396
dm	yyy	th	6
dmm	w	ai cdc ph	
dpwr	35		
dmf	6700		



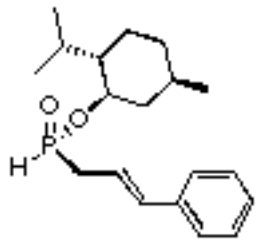
Compound (S_p)-17
^{13}C NMR



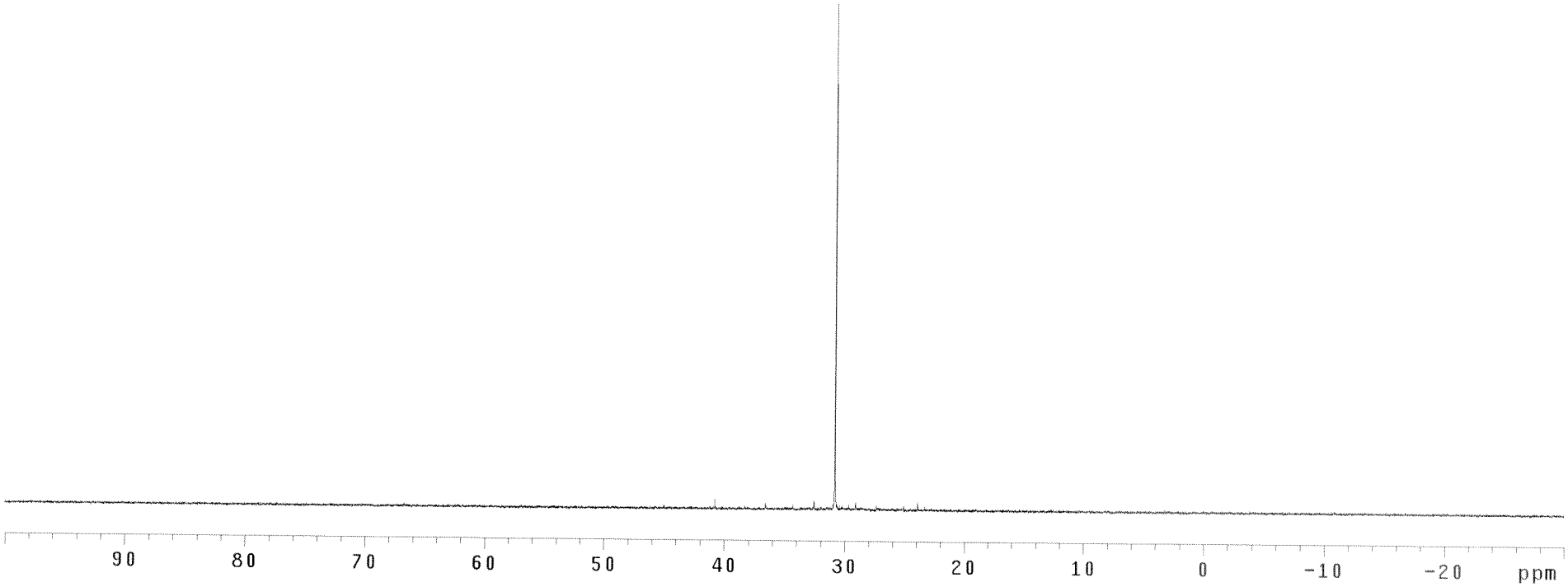
OB 948
pad=10 run with findz0 before acquisitio
n

exp1 Phosphorus

SAMPLE		SPECIAL	
date	Feb 11 2013	temp	not used
solvent	cdcl3	gain	25
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2013.02.09/s_2013~		pw90	18.300
0211_20/data/cdcl3~		alfa	10.000
_01.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	113.6
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	14
dm	yyy	th	11
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



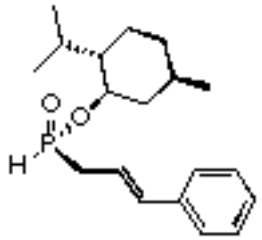
Compound (S_p)-18
$^{31}P\{^1H\}$ NMR decoupled



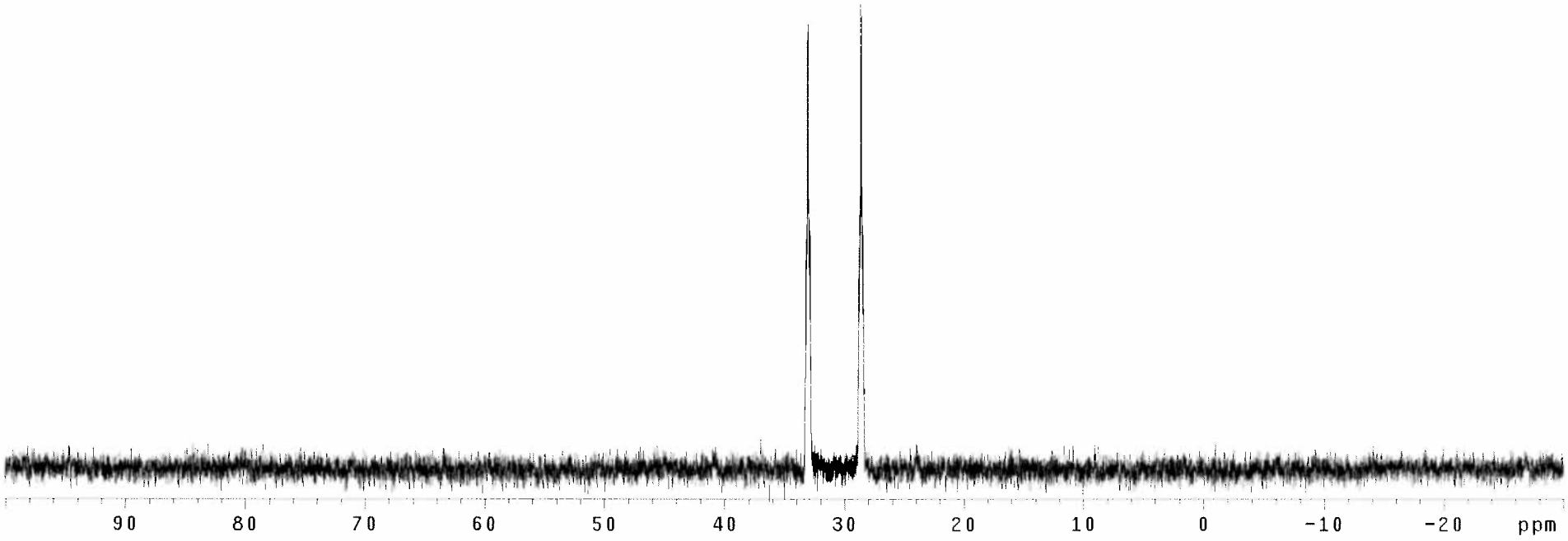
OB 948

exp1 Phosphorus

SAMPLE		SPECIAL	
date	Feb 11 2013	temp	not used
solvent	cdcl3	gain	25
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2013.02.09/s_2013~		pw90	18.300
0211_20/data/cdcl3~		alfa	10.000
_02.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	-142.4
tpwr	55	lp	-336.5
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	178
dm	ynn	th	12
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



Compound (S_p)-18
$^{31}P\{^1H\}$ NMR coupled



OB 948
pad=10 run with findz0 before acquisitio
n

exp1 Proton

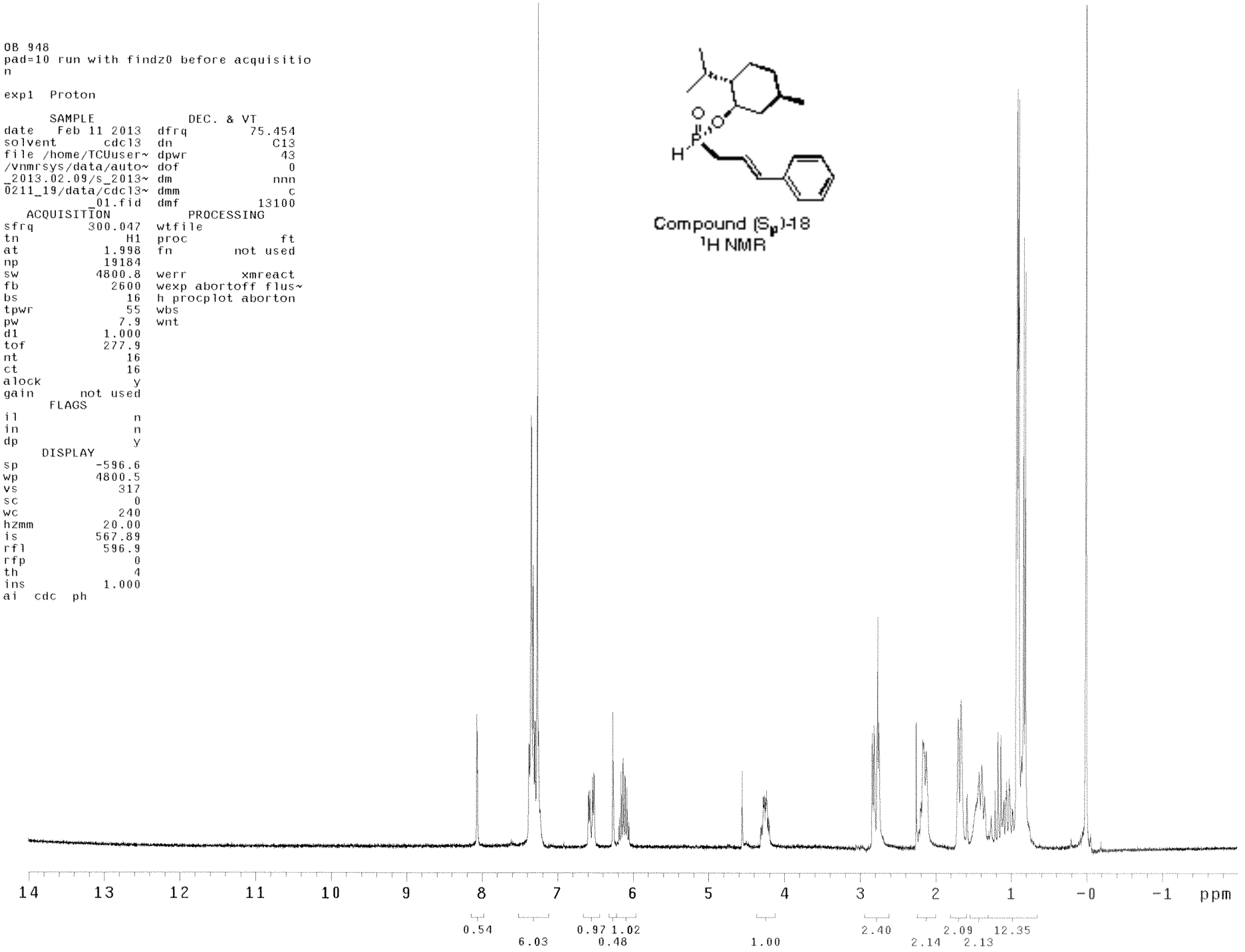
SAMPLE		DEC. & VT	
date	Feb 11 2013	dfrq	75.454
solvent	cdcl3	dn	C13
file	/home/TCUuser~	dpwr	43
	/vnmrsys/data/auto~	dof	0
	_2013.02.09/s_2013~	dm	nnn
	0211_19/data/cdcl3~	dmm	c
	_01.fid	dmf	13100
ACQUISITION		PROCESSING	
sfrq	300.047	wtfile	
tn	H1	proc	ft
at	1.998	fn	not used
np	19184		
sw	4800.8	werr	xmreact
fb	2600	wexp	abortoff flus~
bs	16	h procplot	aborton
tpwr	55	wbs	
pw	7.9	wnt	
d1	1.000		
tof	277.9		
nt	16		
ct	16		
alock	y		
gain	not used		

FLAGS	
il	n
in	n
dp	y

DISPLAY	
sp	-596.6
wp	4800.5
vs	317
sc	0
wc	240
hzmm	20.00
is	567.89
rfl	596.9
rfp	0
th	4
ins	1.000

ai cdc ph

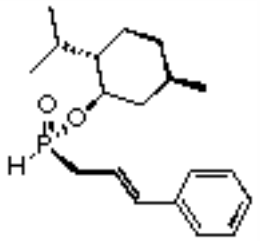
Compound (S_p)-18
^{1}H NMR



OB 948

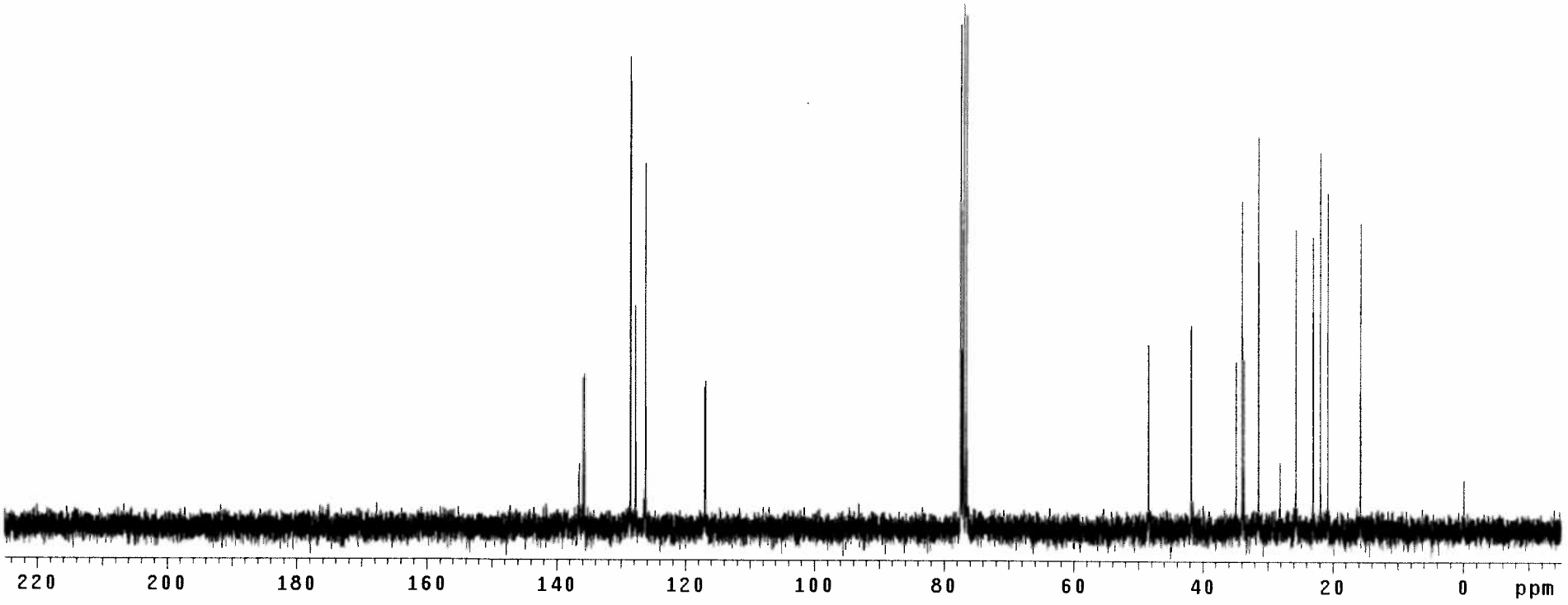
exp1 Carbon

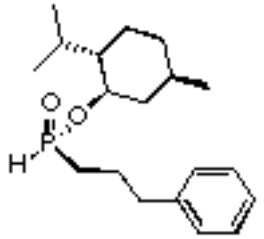
SAMPLE		SPECIAL	
date	Feb 11 2013	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2013.02.08/s_2013~		pw90	18.500
0211_20/data/cdcl3~		alfa	10.000
_03.fid		FLAGS	
ACQUISITION		il	n
sw	18115.9	in	n
at	1.301	dp	y
np	47120	hs	nn
fb	10000	PROCESSING	
bs	64	lb	0.50
d1	2.000	fn	not used
nt	650	DISPLAY	
ct	650	sp	-1135.5
TRANSMITTER		wp	18115.4
tn	C13	rfl	1136.1
sfrq	75.454	rfp	0
tof	766.0	rp	55.8
tpwr	58	lp	-193.3
pw	9.250	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	1156
dm	yyy	th	10
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



Compound (S_p)-18

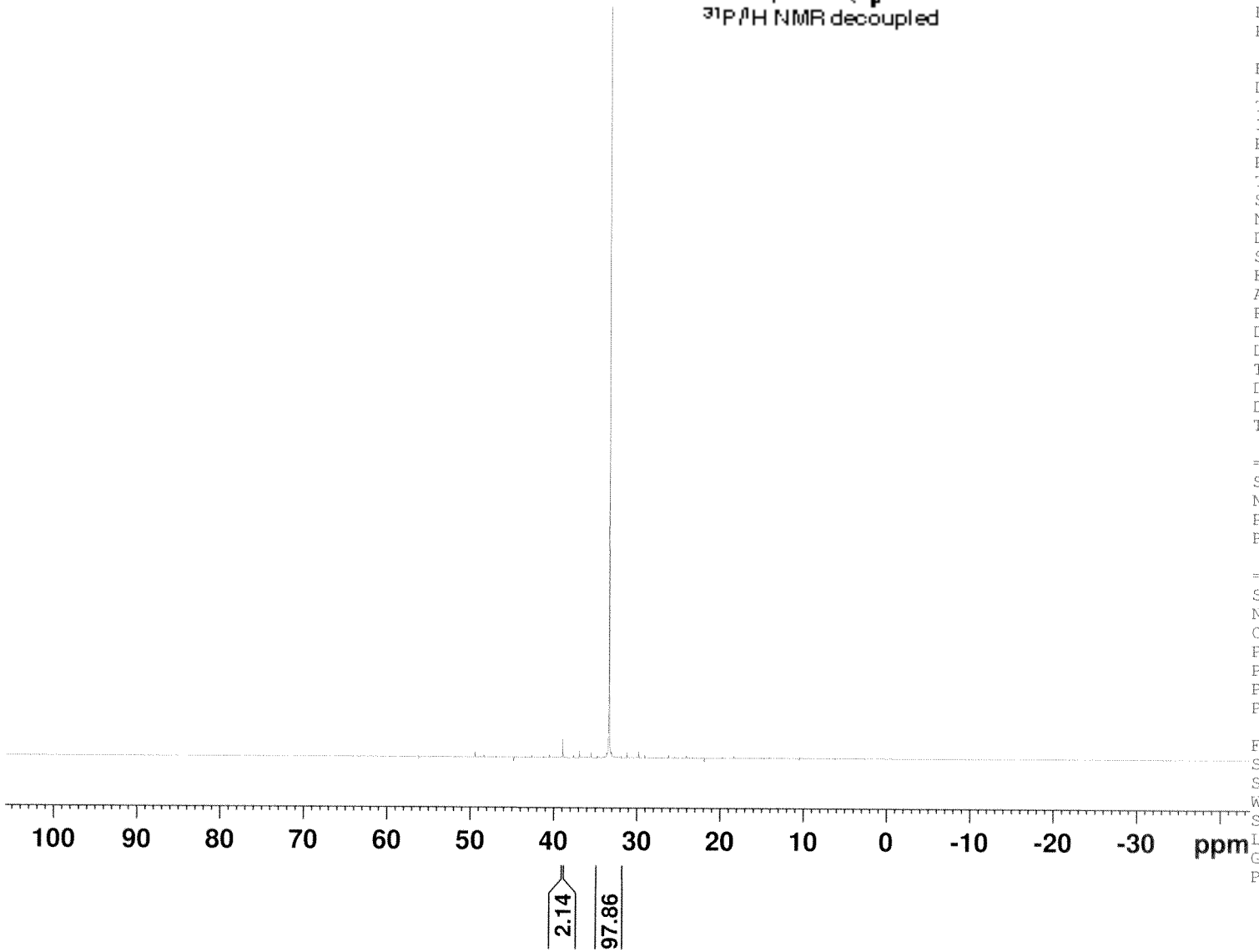
^{13}C NMR



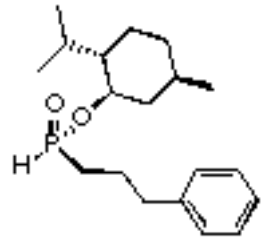


Compound (S_p)-19

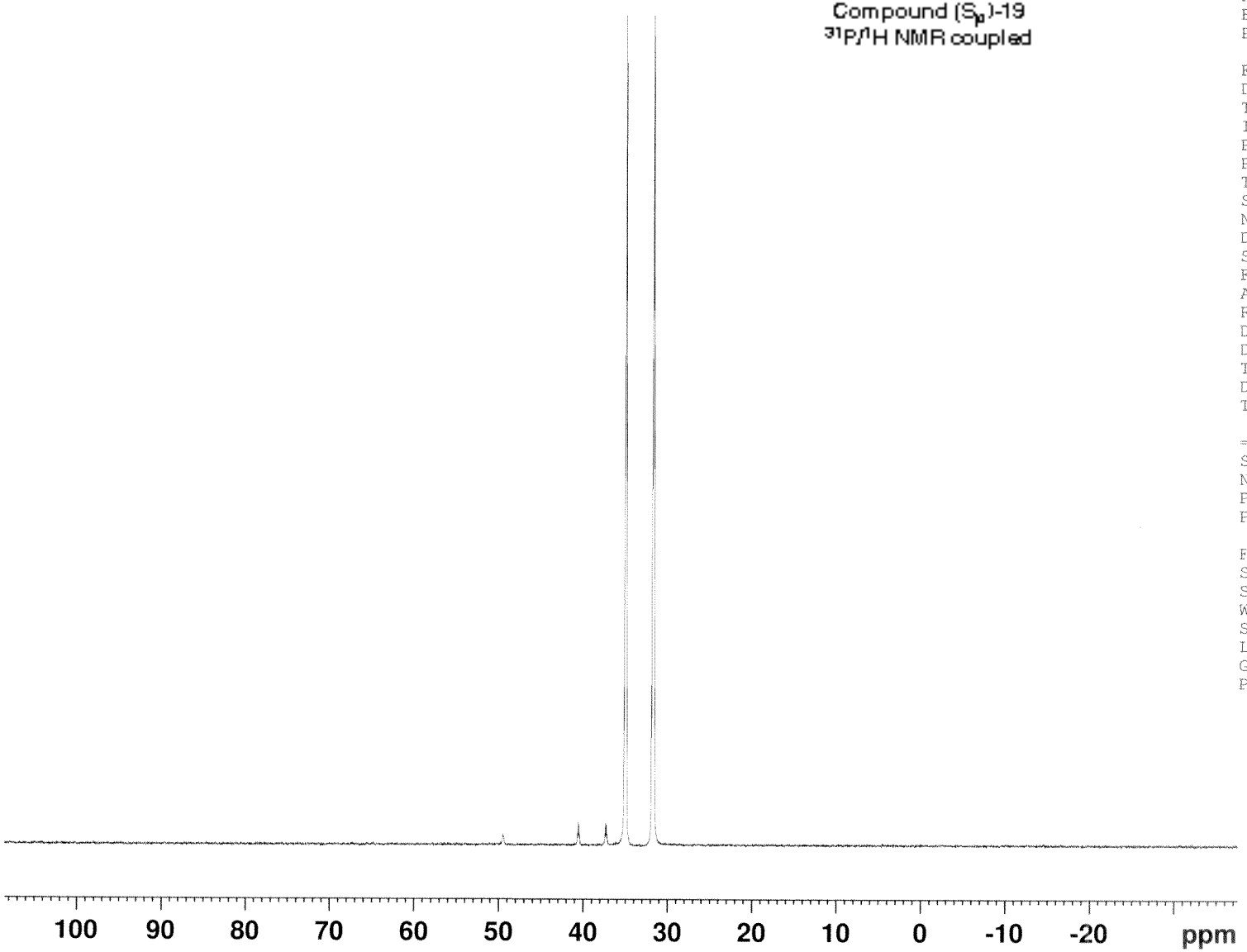
$^{31}P\{^1H\}$ NMR decoupled



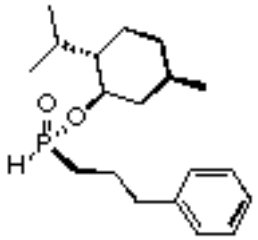

```
Current Data Parameters
NAME      OB 2095 after column
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150630
Time                9.38
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                 7.800 usec
DE                 6.50 usec
TE                294.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1         15.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                  32768
SF          161.9755930 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



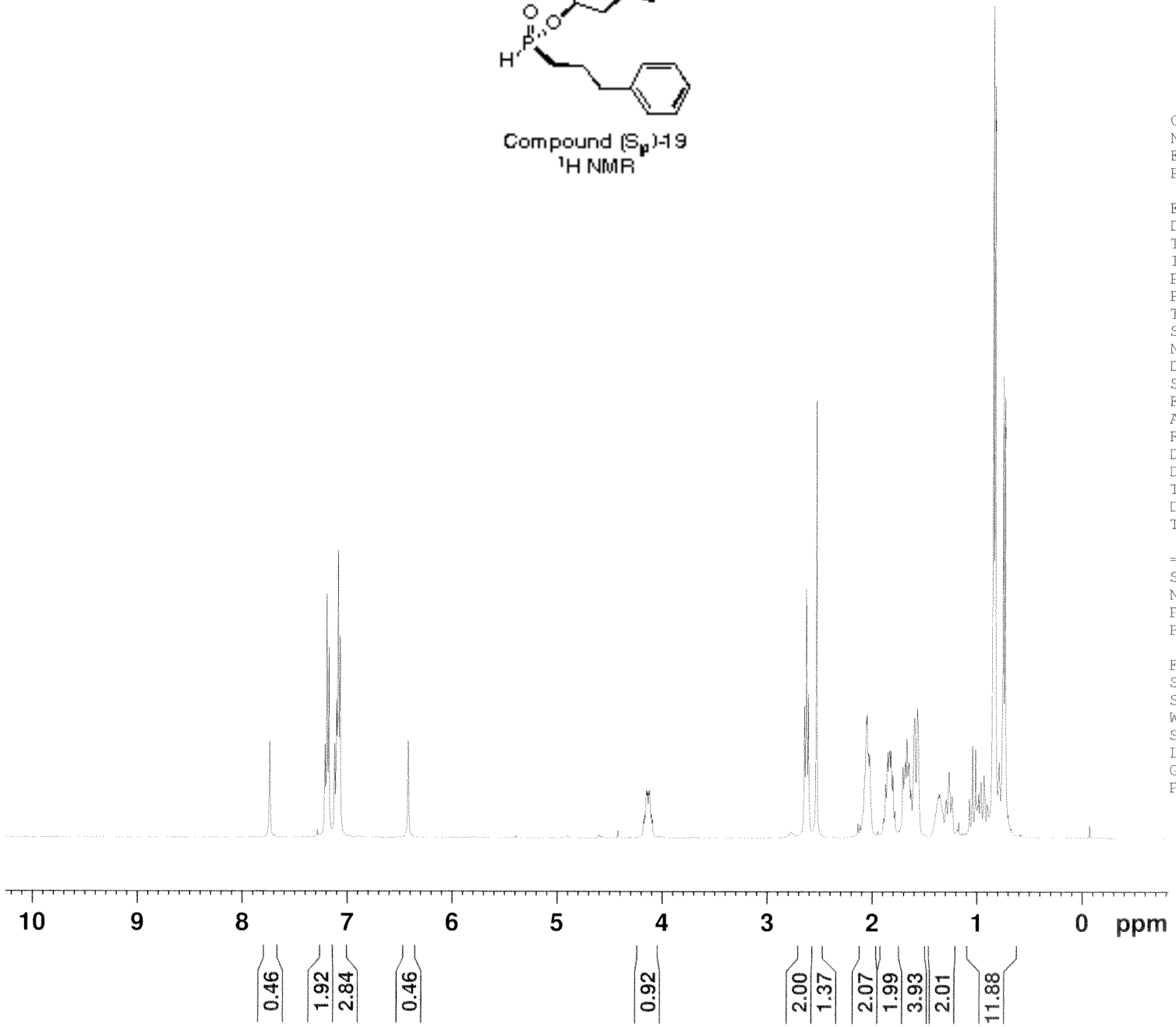
Compound (S_p)-19
$^{31}P\{^1H\}$ NMR coupled



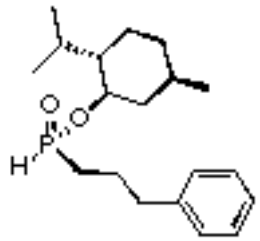

```
Current Data Parameters
NAME      OB 2095 after column
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150630
Time                9.40
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH            64102.563 Hz
FIDRES          0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                7.800 usec
DE                6.50 usec
TE                294.0 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 32768
SF           161.9755930 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



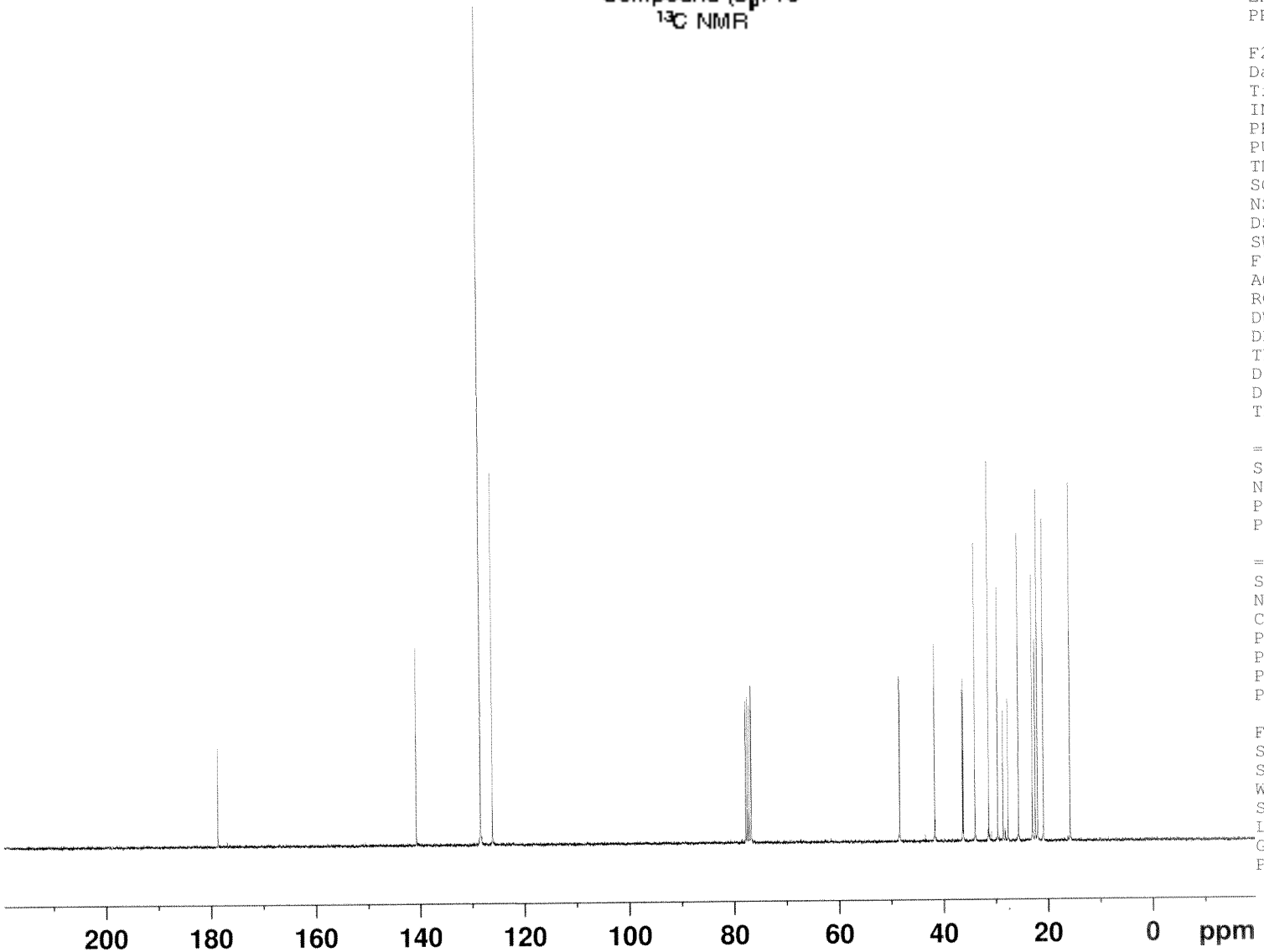
Compound (S_p)-19
^{1}H NMR



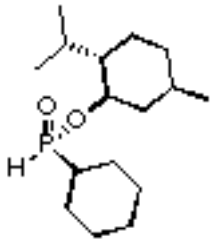

```
Current Data Parameters
NAME      OB 2095 after column
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150630
Time               9.42
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                 8.79
DW                62.400 usec
DE                 6.50 usec
TE                293.9 K
D1             1.00000000 sec
TD0                   1

======== CHANNEL f1 =========
SFO1         400.1324710 MHz
NUC1                1H
P1                10.00 usec
PLW1         25.00300026 W

F2 - Processing parameters
SI                65536
SF           400.1300000 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



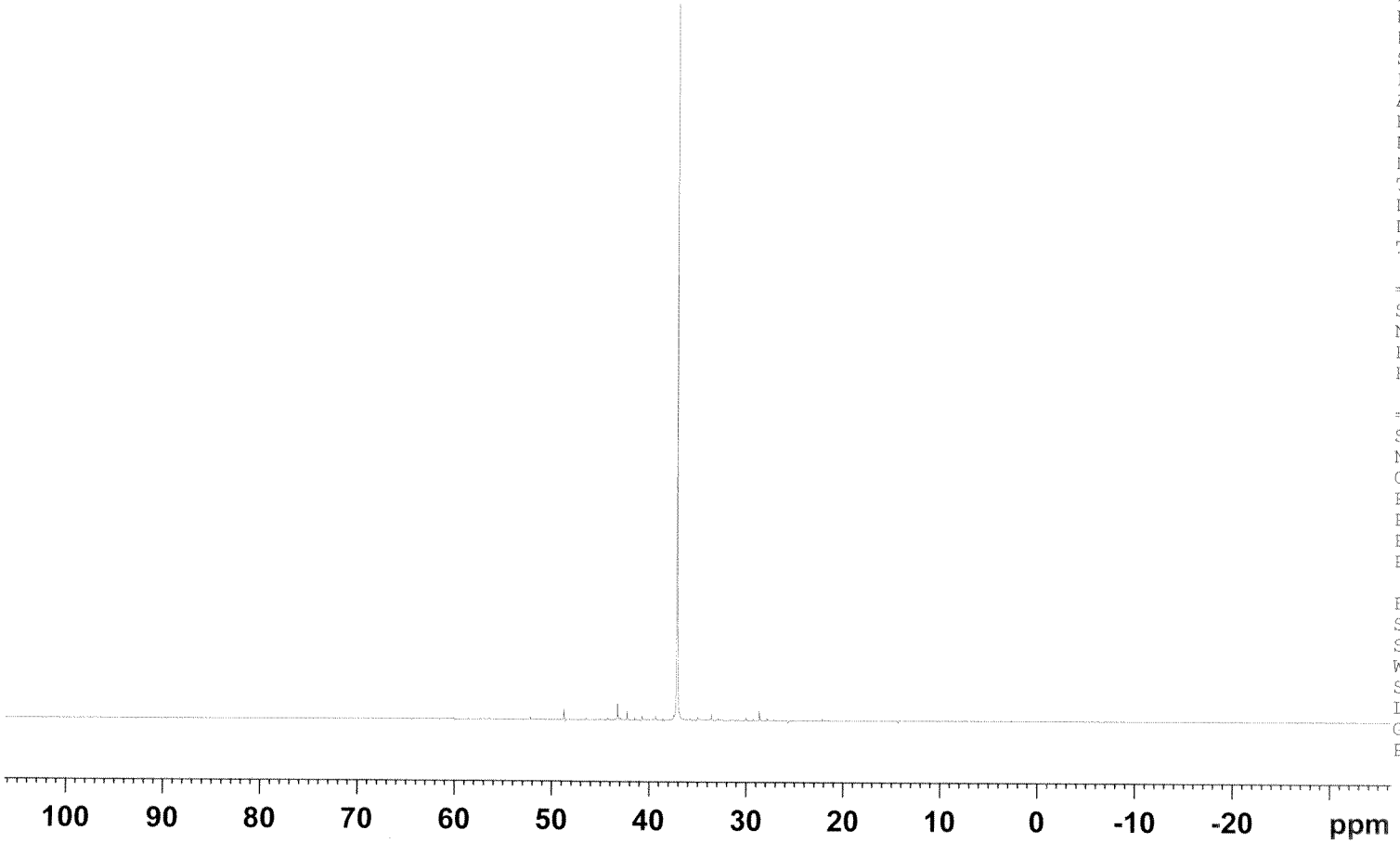
Compound (S_p)-19
^{13}C NMR



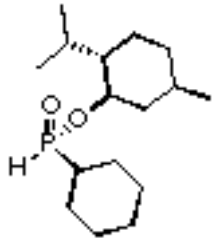

```
Current Data Parameters
NAME      OB 2095 after column
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150630
Time                9.51
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  101
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                295.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

========== CHANNEL f1 =========
SFO1         100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1         45.00000000 W

========== CHANNEL f2 =========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



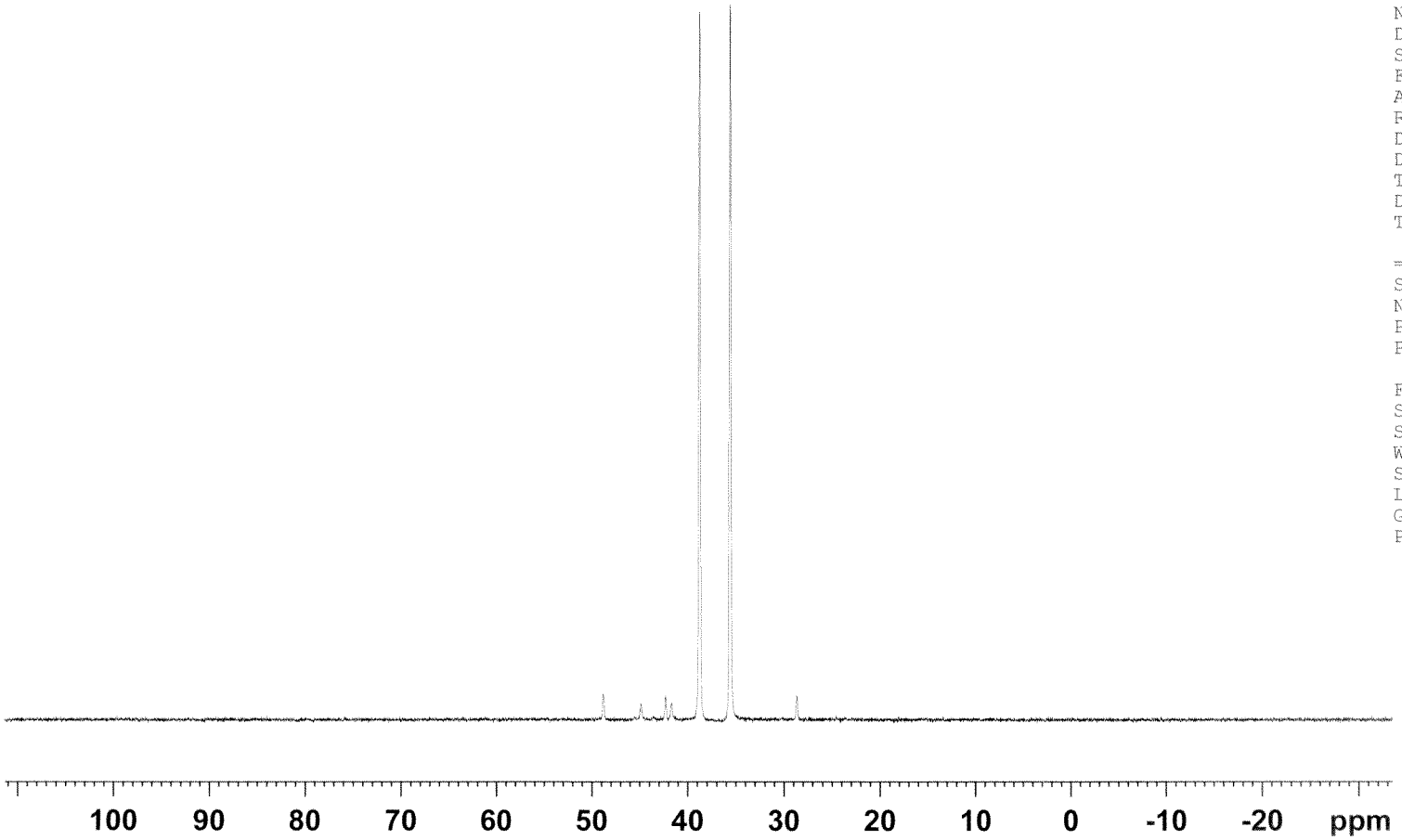
Compound (S_p)-20
$^{31}P\{^1H\}$ NMR decoupled



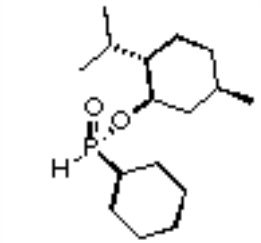

```
Current Data Parameters
NAME      OB 1826 after column
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20141112
Time                  9.44
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       4
SWH              64102.563 Hz
FIDRES            0.978127 Hz
AQ                0.5111808 sec
RG                   203.57
DW                    7.800 usec
DE                    6.50 usec
TE                   298.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          161.9674942 MHz
NUC1                   31P
P1                   14.25 usec
PLW1          15.00000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2          10.00000000 W
PLW12          0.31604999 W
PLW13          0.25600001 W

F2 - Processing parameters
SI                    32768
SF            161.9755930 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```



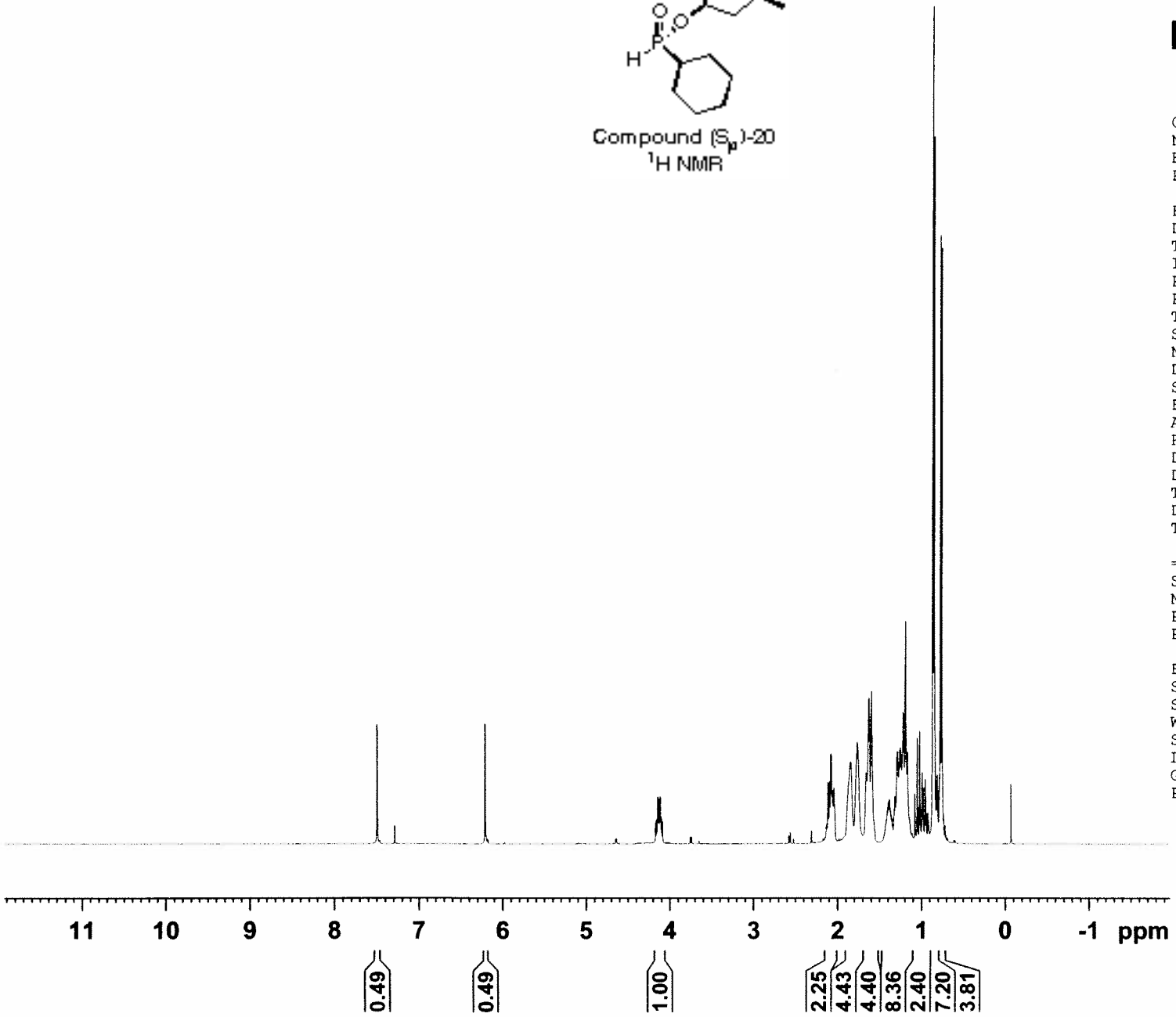
Compound (S_p)-20
$^{31}P/^{1}H$ NMR coupled




```
Current Data Parameters
NAME      OB 1826 after column
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20141112
Time                11.08
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH            64102.563 Hz
FIDRES          0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                 7.800 usec
DE                 6.50 usec
TE                297.8 K
D1           2.00000000 sec
TD0                    1

========= CHANNEL f1 ========
SFO1         161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 32768
SF           161.9755930 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



Compound (S_p)-20
^{1}H NMR

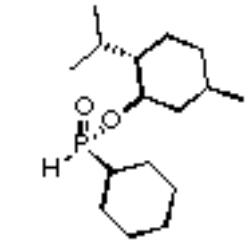


Current Data Parameters
NAME OB 1826 after column
EXPNO 3
PROCNO 1

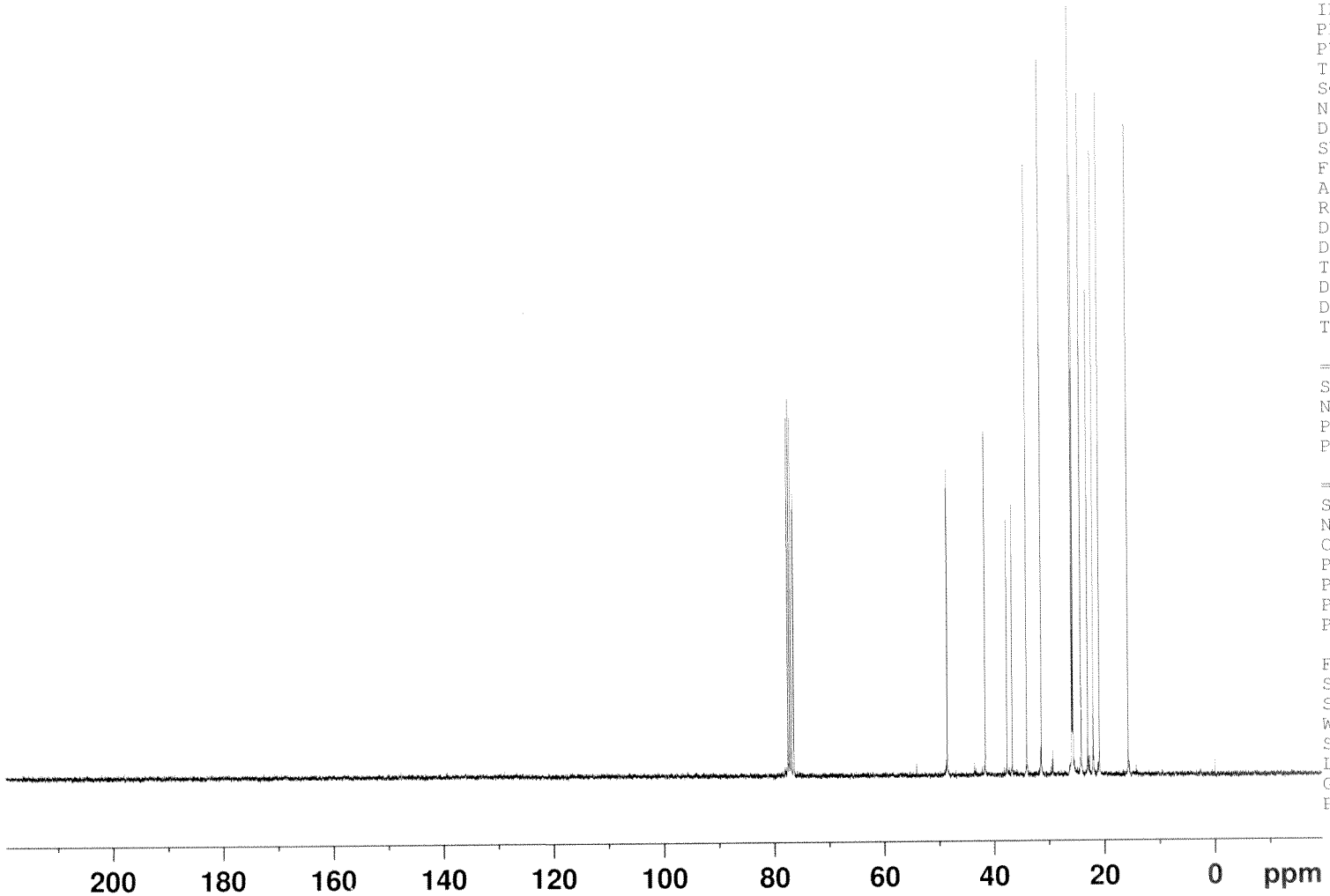
F2 - Acquisition Parameters
Date_ 20141112
Time 11.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 12.96
DW 62.400 usec
DE 6.50 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
PLW1 25.00300026 W

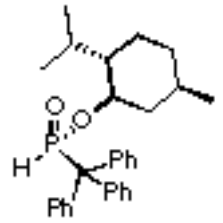
F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



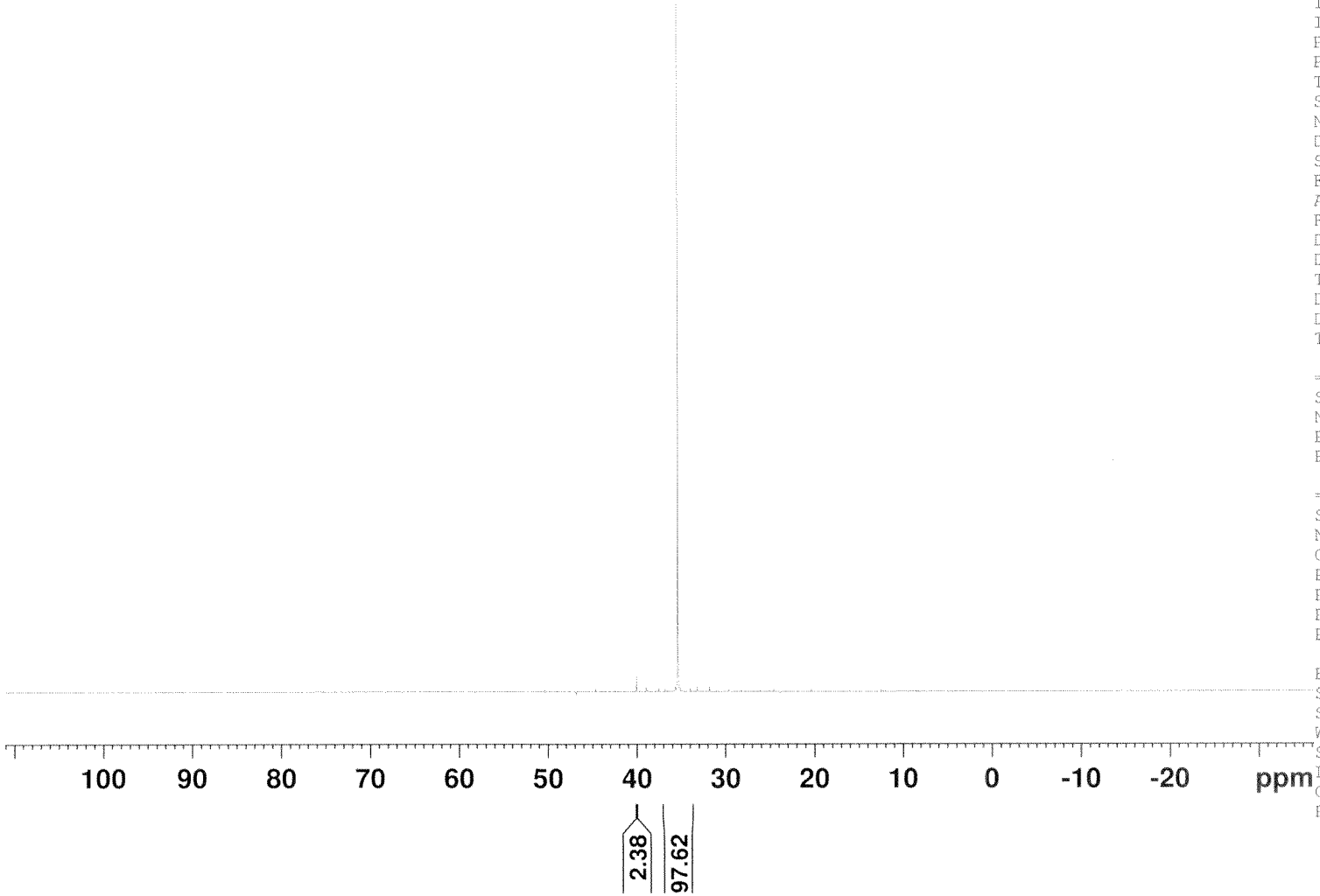
Compound (S_p)-20
^{13}C NMR



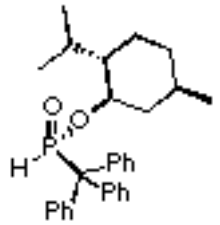

```
Current Data Parameters
NAME      OB 1826 after column
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20141112
Time                 12.46
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     268
DS                       4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631488 sec
RG                   203.57
DW                   20.800 usec
DE                    6.50 usec
TE                   298.3 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           100.6228293 MHz
NUC1                    13C
P1                   10.00 usec
PLW1           45.00000000 W

======== CHANNEL f2 ========
SFO2           400.1316005 MHz
NUC2                    1H
CPDPRG[2            waltz16
PCPD2                90.00 usec
PLW2           10.00000000 W
PLW12           0.31604999 W
PLW13           0.25600001 W

F2 - Processing parameters
SI                     32768
SF             100.6127685 MHz
WDW                       EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```



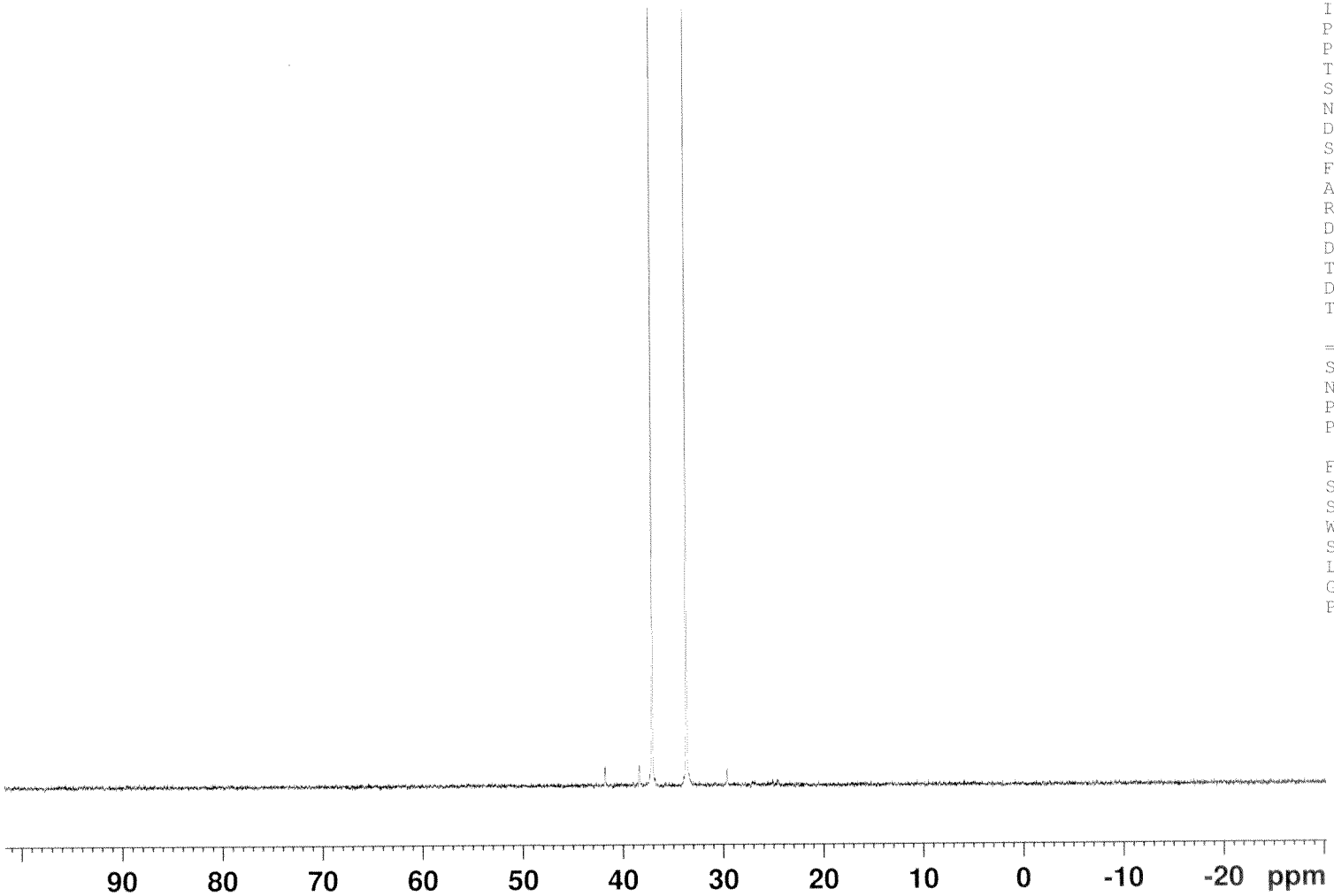
Compound (S_p)-21
$^{31}P\{^1H\}$ NMR decoupled



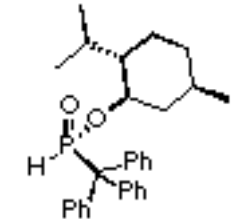

```
Current Data Parameters
NAME      OB 1843 after column
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20141124
Time               17.38
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                 7.800 usec
DE                 6.50 usec
TE                296.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1         15.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                  32768
SF           161.9755930 MHz
WDW                    EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



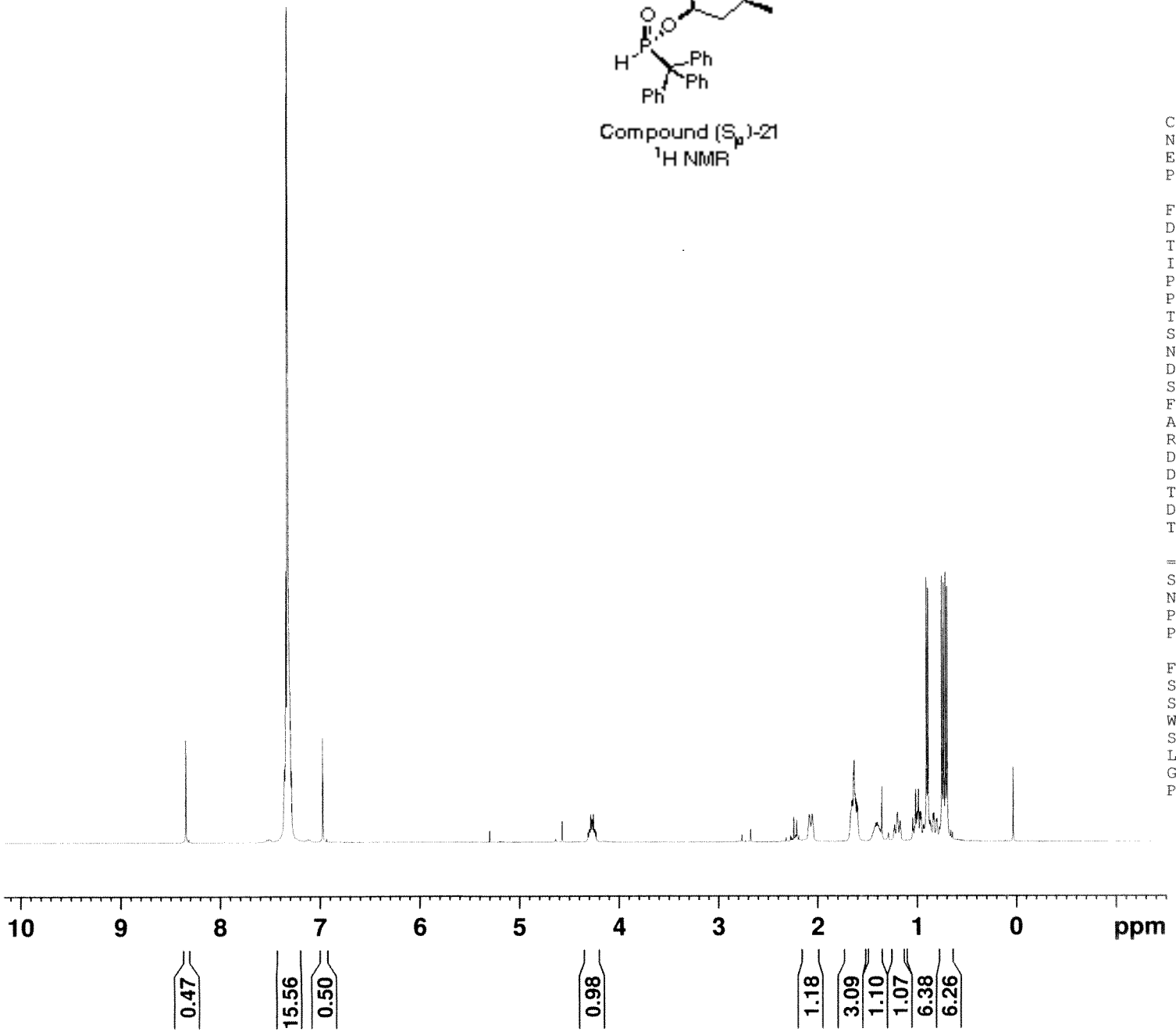
Compound (S_p)-21
$^{31}P/^{1}H$ NMR coupled



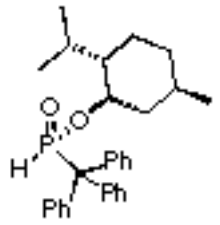

```
Current Data Parameters
NAME      OB 1843 after column
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20141124
Time                 17.40
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      4
SWH            64102.563 Hz
FIDRES          0.978127 Hz
AQ              0.5111808 sec
RG                 203.57
DW                  7.800 usec
DE                  6.50 usec
TE                 295.8 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         161.9674942 MHz
NUC1                  31P
P1                  14.25 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                   32768
SF           161.9755930 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



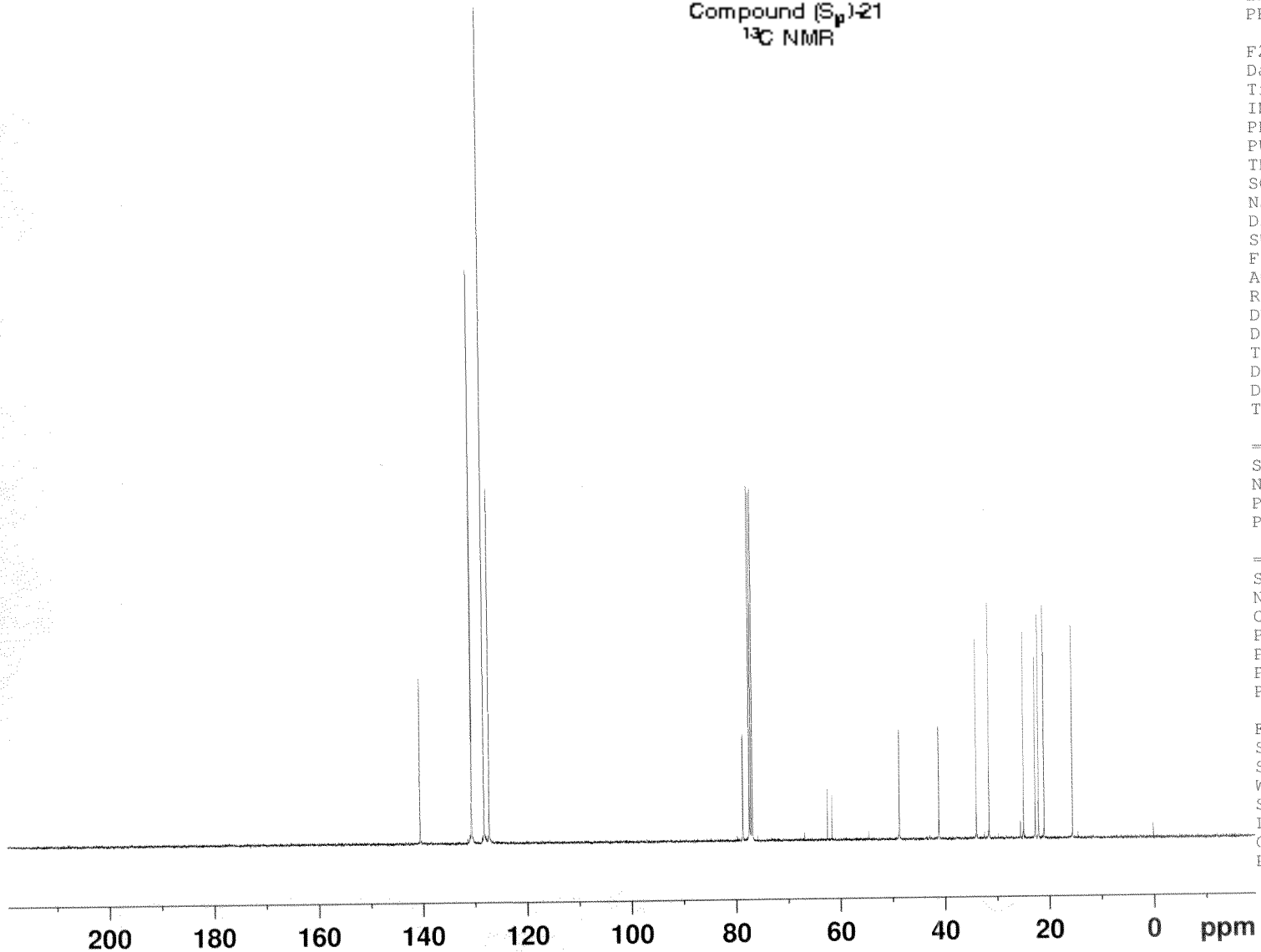
Compound (S_p)-21
^{1}H NMR



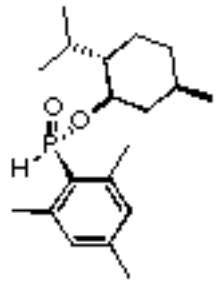

```
Current Data Parameters
NAME      OB 1843 after column
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_           20141124
Time               17.42
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     9
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 32.38
DW                62.400 usec
DE                 6.50 usec
TE                295.8 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 10.00 usec
PLW1         25.00300026 W

F2 - Processing parameters
SI                 65536
SF           400.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



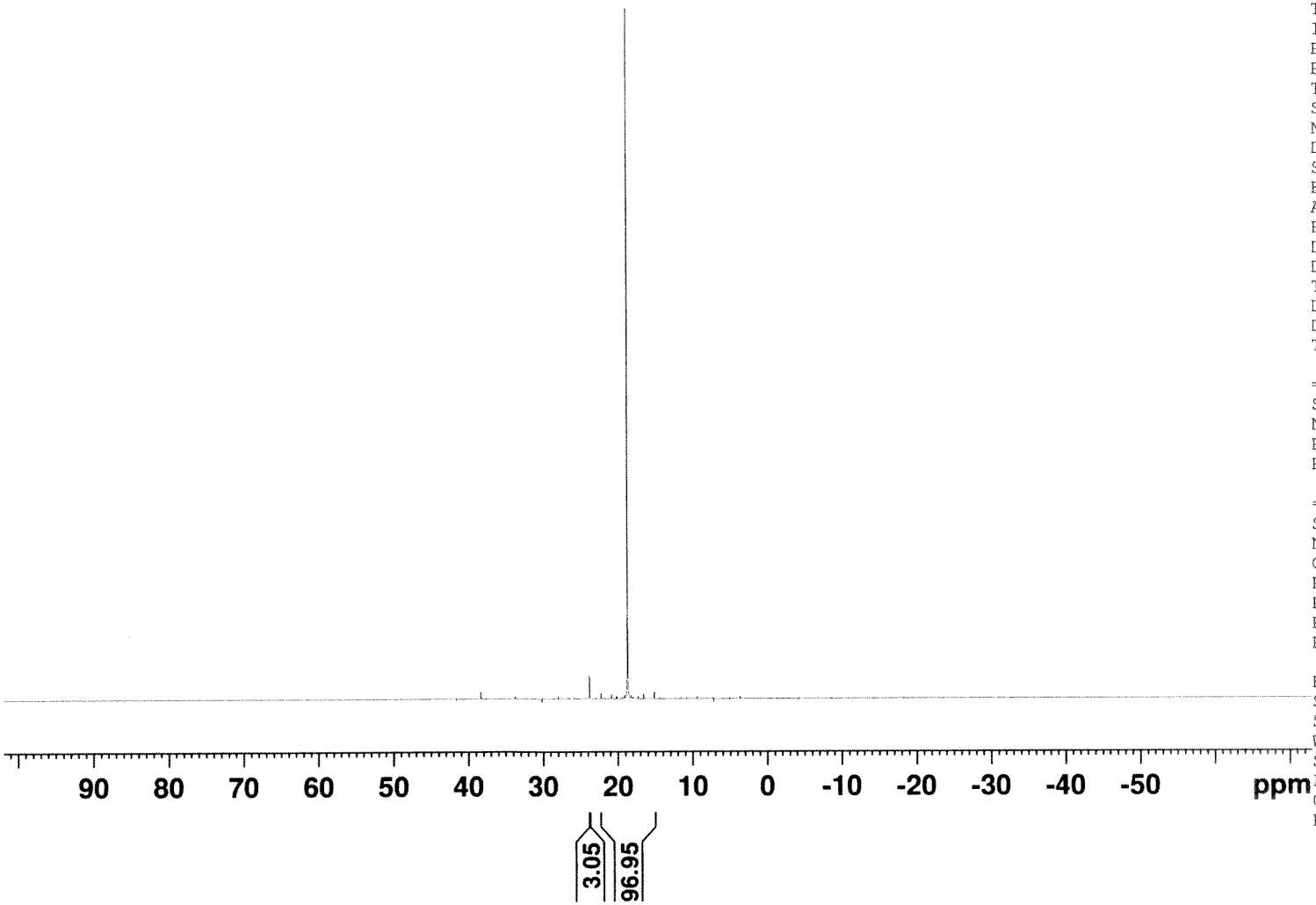
Compound (S_p)-21
^{13}C NMR



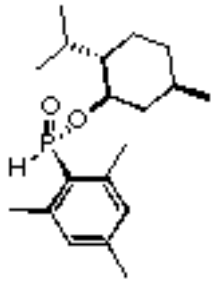

```
Current Data Parameters
NAME      OB 1843 after column
EXPNO                        4
PROCNO                       1

F2 - Acquisition Parameters
Date_              20141124
Time                 18.31
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     819
DS                       4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                  203.57
DW                20.800 usec
DE                 6.50 usec
TE                 296.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

========== CHANNEL f1 =========
SFO1          100.6228293 MHz
NUC1                   13C
P1                  10.00 usec
PLW1          45.00000000 W

========== CHANNEL f2 =========
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2               90.00 usec
PLW2          10.00000000 W
PLW12          0.31604999 W
PLW13          0.25600001 W

F2 - Processing parameters
SI                     32768
SF            100.6127685 MHz
WDW                       EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```



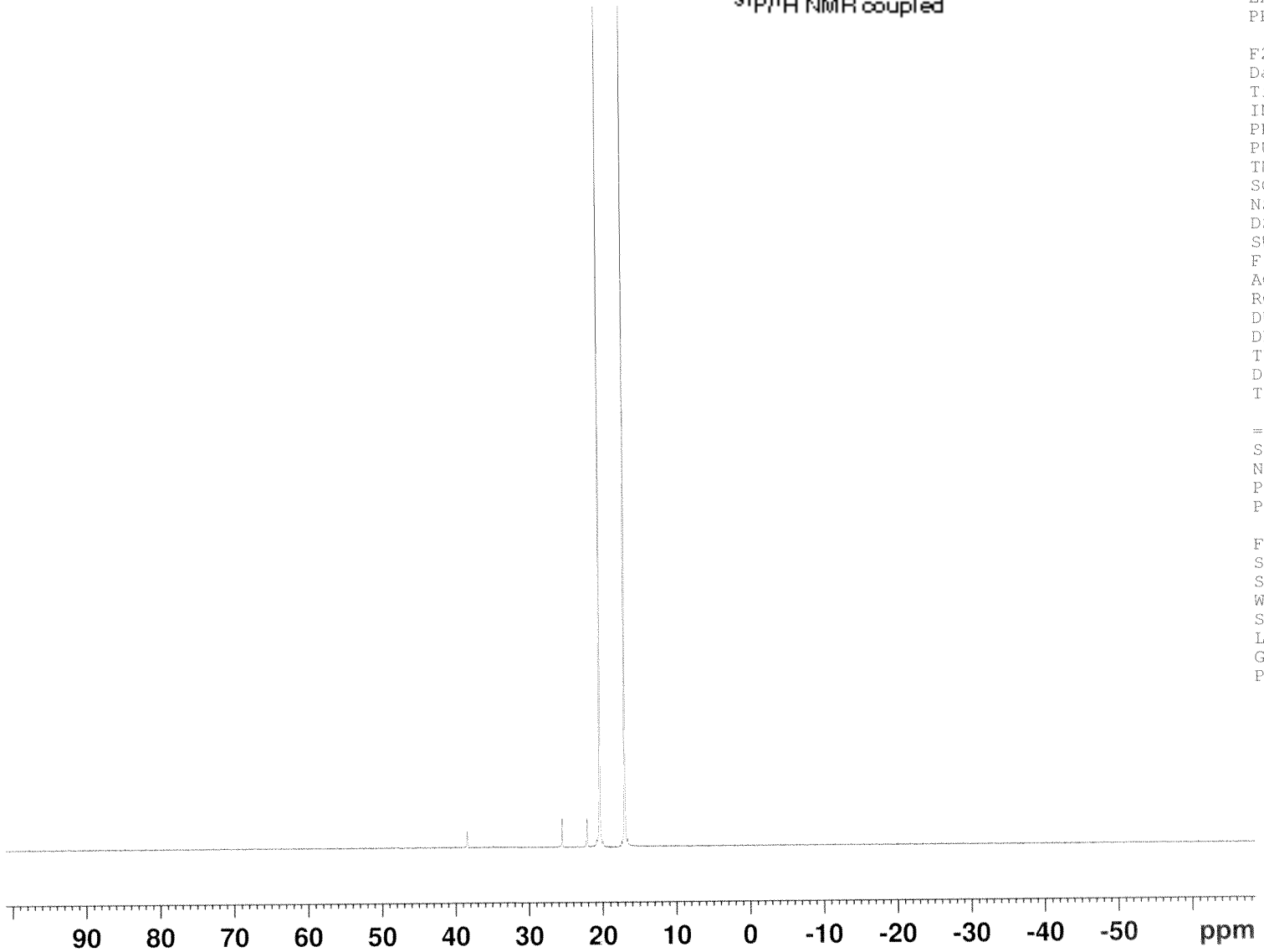
Compound (S_p)-22
$^{31}P\{^1H\}$ NMR decoupled



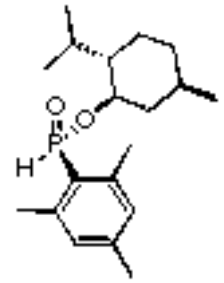

```
Current Data Parameters
NAME      OB 1866 after column
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20141210
Time                8.57
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                 7.800 usec
DE                 6.50 usec
TE                299.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1        15.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



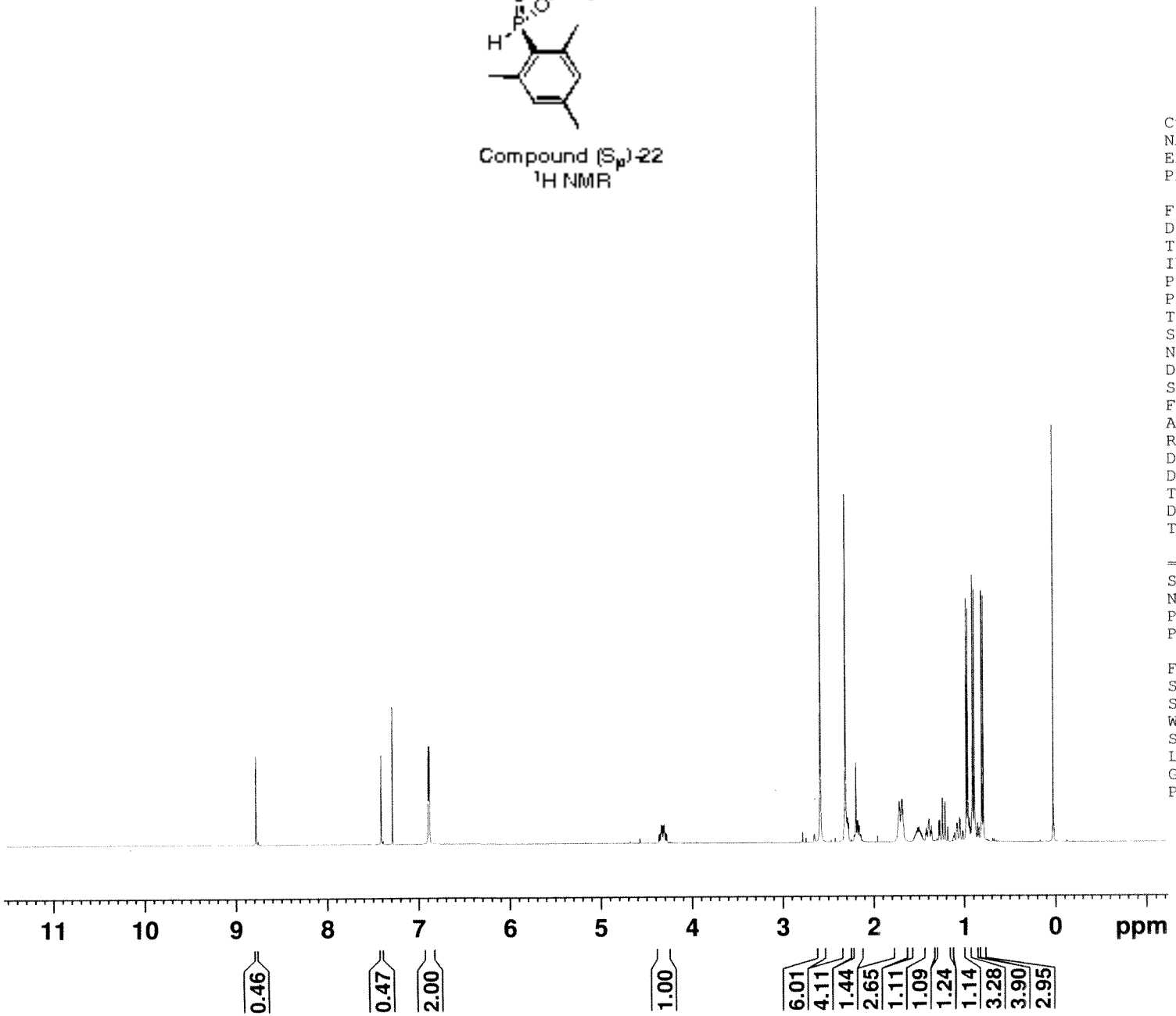
Compound (S_p)-22
$^{31}P\{^1H\}$ NMR coupled



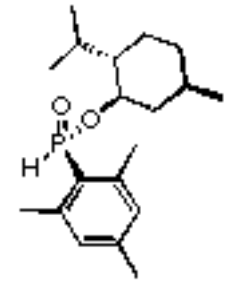

```
Current Data Parameters
NAME      OB 1866 after column
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20141210
Time                8.59
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH            64102.563 Hz
FIDRES          0.978127 Hz
AQ              0.5111808 sec
RG                 203.57
DW                  7.800 usec
DE                  6.50 usec
TE                 298.6 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         161.9674942 MHz
NUC1                  31P
P1                  14.25 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 32768
SF           161.9755930 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



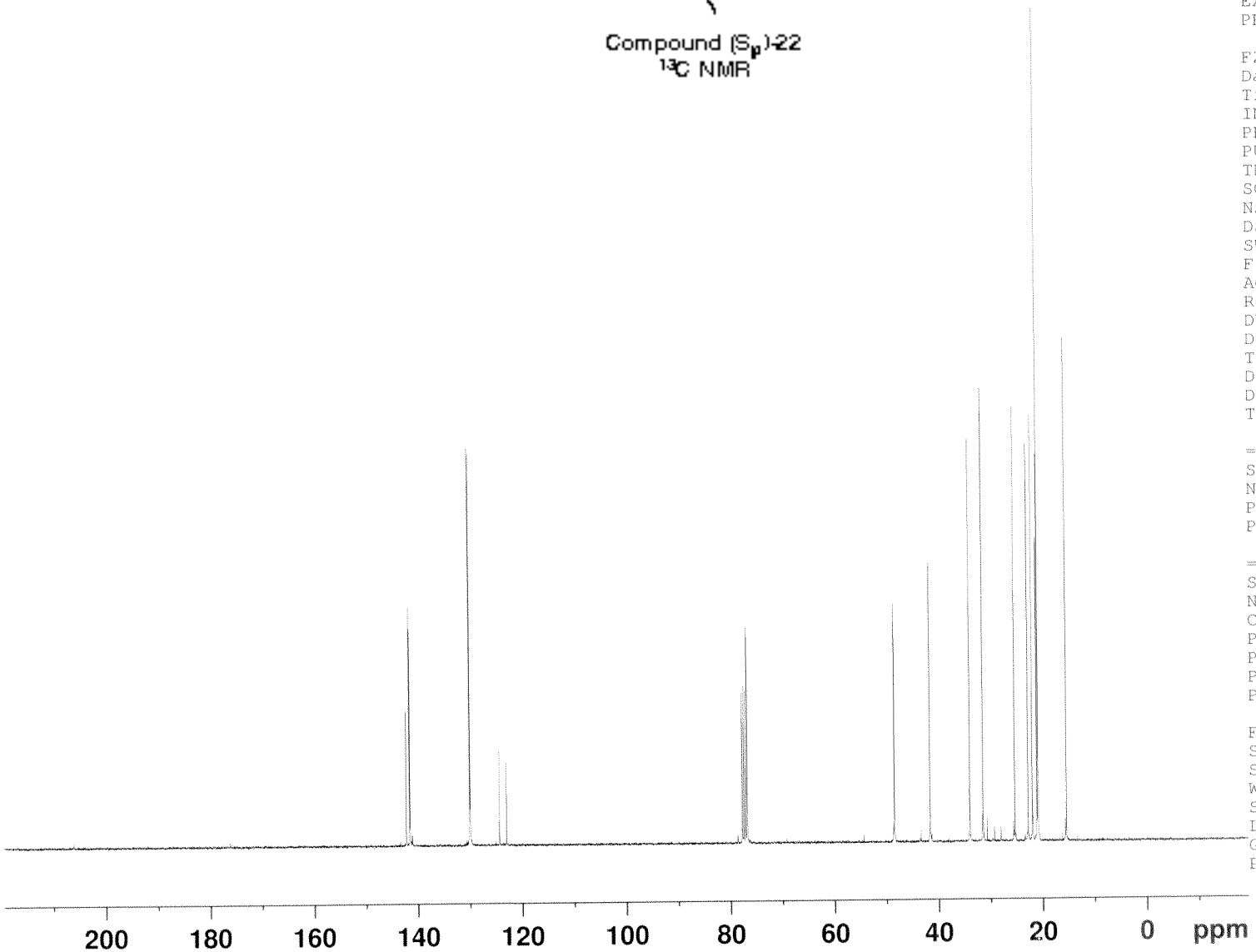
Compound (S_p)-22
^{1}H NMR




```
Current Data Parameters
NAME      OB 1866 after column
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_           20141210
Time                8.50
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    12
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 113.32
DW                62.400 usec
DE                 6.50 usec
TE                298.5 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 10.00 usec
PLW1         25.00300026 W

F2 - Processing parameters
SI                 65536
SF          400.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



Compound (S_p)-22
^{13}C NMR

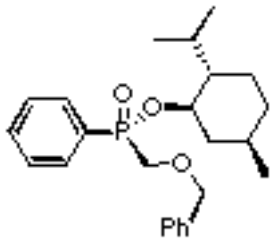



```
Current Data Parameters
NAME      OB 1866 after column
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20141210
Time                 9.17
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDC13
NS                    282
DS                      4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                299.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         45.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                   32768
SF           100.6127685 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

OB 396

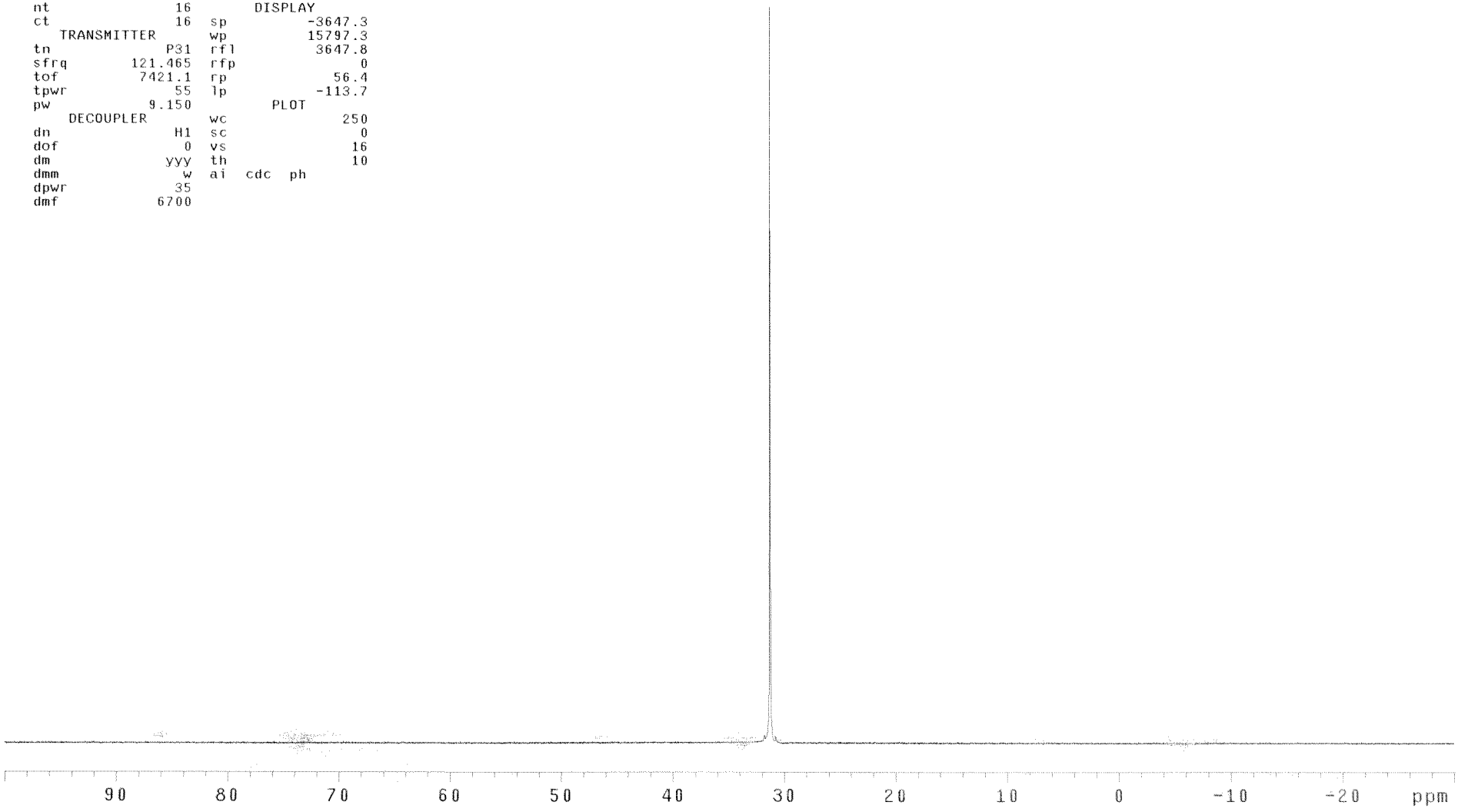
exp1 Phosphorus

SAMPLE		SPECIAL	
date	Apr 25 2012	temp	not used
solvent	D2O	gain	20
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2012.04.23/s_2012~		pw90	18.300
0425_05/data/D2O_0~		alfa	10.000
2.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	56.4
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	16
dm	yyy	th	10
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



Compound (S_p)-23

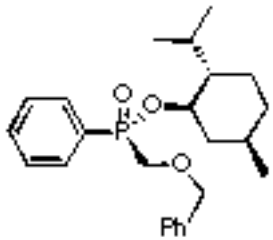
$^{31}P\{^{1}H\}$ NMR decoupled



OB 396

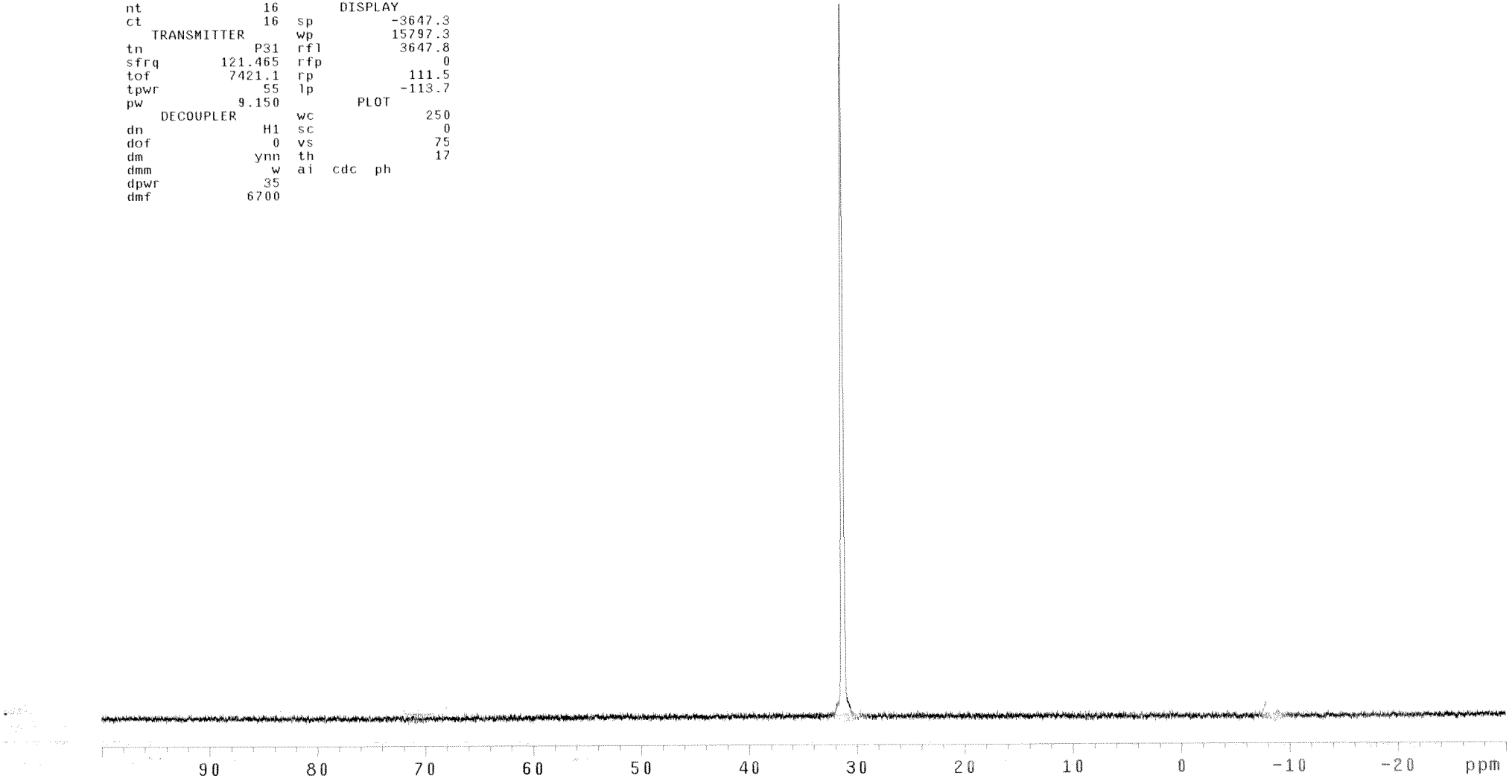
exp1 Phosphorus

SAMPLE		SPECIAL	
date	Apr 25 2012	temp	not used
solvent	D2O	gain	20
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2012.04.23/s_2012~		pw90	18.300
0425_05/data/D2O_0~		alfa	10.000
4.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	111.5
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	75
dm	ynn	th	17
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



Compound (S_p)-23

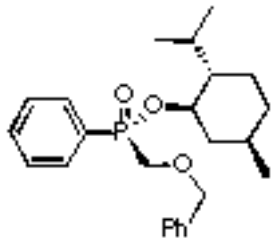
$^{31}P/^{1}H$ NMR coupled



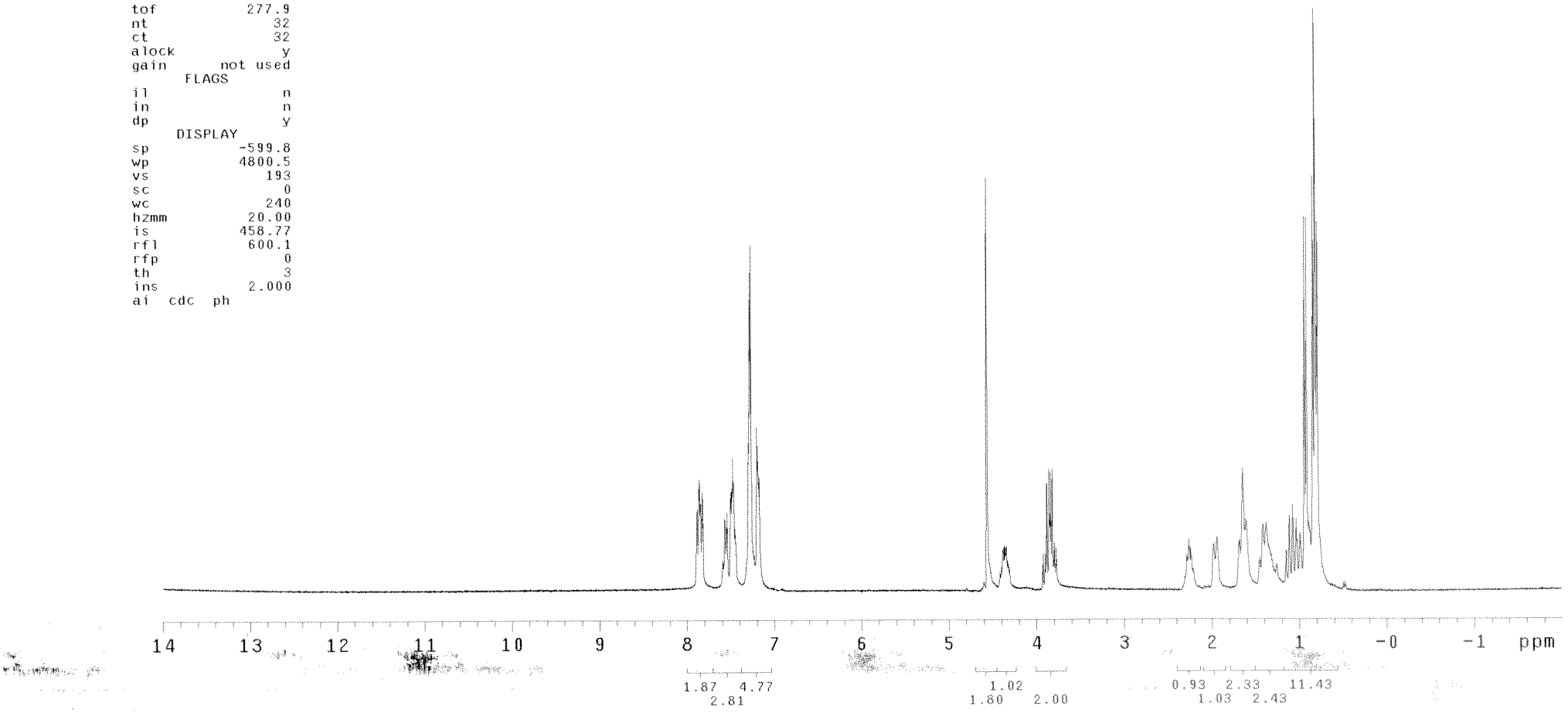
OB 396
pad=10 run with findz0 before acquisitio
n

exp1 Proton

SAMPLE		DEC. & VT	
date	Apr 25 2012	dfrq	75.454
solvent	cdcl3	dn	C13
file	/home/TCUuser~	dpwr	43
	/vnmrsys/data/auto~	dof	0
	_2012.04.23/s_2012~	dm	nnn
	0425_02/data/cdcl3~	dmm	c
	01.fid	dmf	13100
ACQUISITION		PROCESSING	
sfrq	300.047	wtfile	
tn	H1	proc	ft
at	1.998	fn	not used
np	19184		
sw	4800.8	werr	xmreact
fb	2600	wexp	abortoff flus~
bs	16	h procplot	aborton
tpwr	55	wbs	
pw	7.9	wnt	
d1	1.000		
tof	277.9		
nt	32		
ct	32		
alock	y		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-599.8		
wp	4800.5		
vs	193		
sc	0		
wc	240		
hzmm	20.00		
is	458.77		
rfl	600.1		
rfp	0		
th	3		
ins	2.000		
ai cdc ph			



Compound (S_p)-23
^{1}H NMR

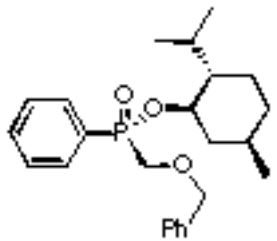


OB 396

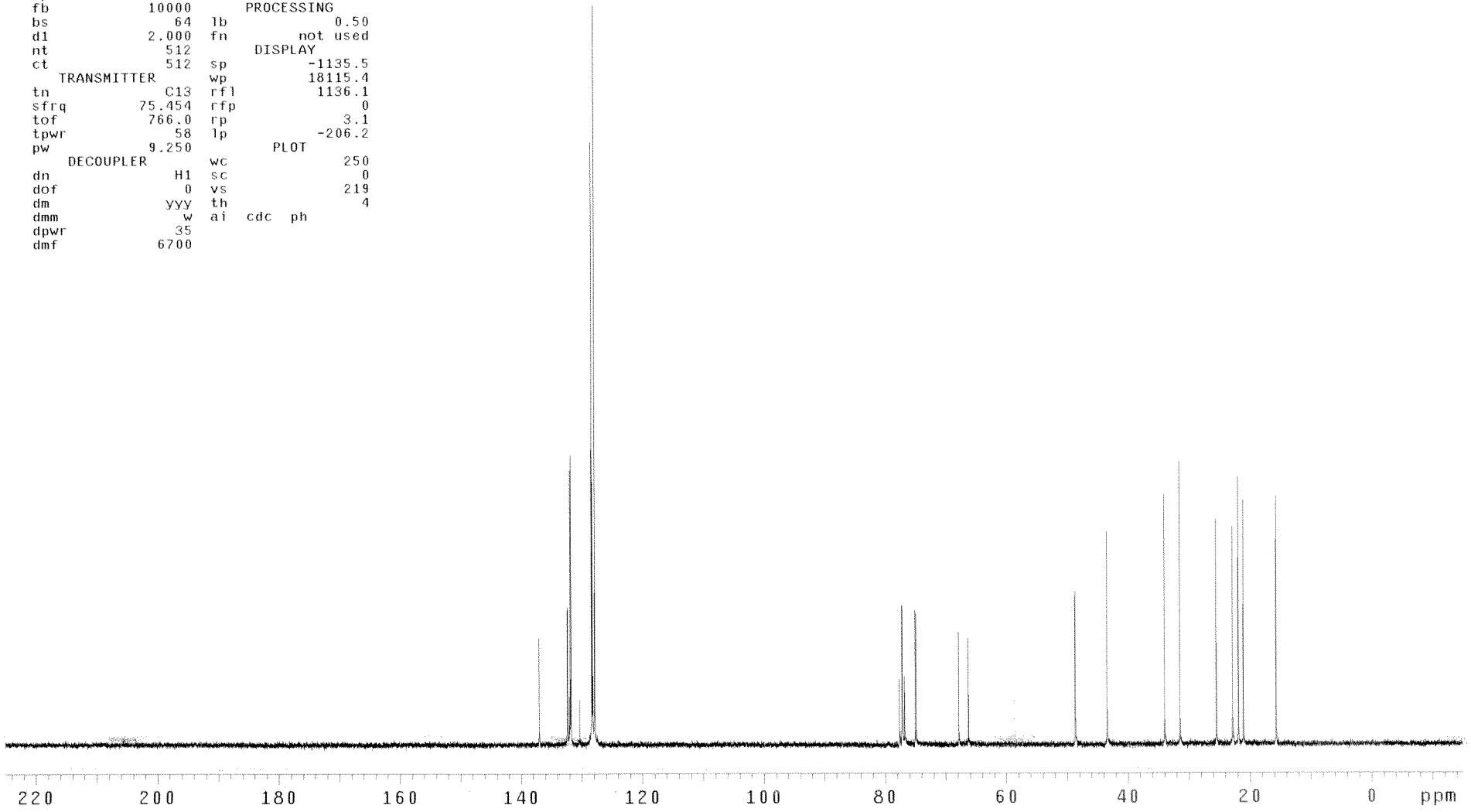
pad=10 run with findz0 before acquisitio
n

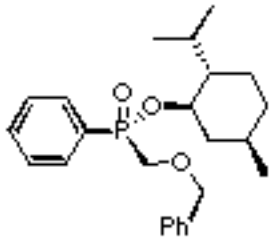
exp1 Carbon

SAMPLE		SPECIAL	
date	Apr 25 2012	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2012.04.23/s_2012~		pw90	18.500
0425_06/data/cdcl3~		alfa	10.000
_01.fid		FLAGS	
ACQUISITION		il	n
sw	18115.9	in	n
at	1.301	dp	y
np	47120	hs	nn
fb	10000	PROCESSING	
bs	64	lb	0.50
d1	2.000	fn	not used
nt	512	DISPLAY	
ct	512	sp	-1135.5
TRANSMITTER		wp	18115.4
tn	C13	rfl	1136.1
sfrq	75.454	rfp	0
tof	766.0	rp	3.1
tpwr	58	lp	-206.2
pw	9.250	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	219
dm	yyy	th	4
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		

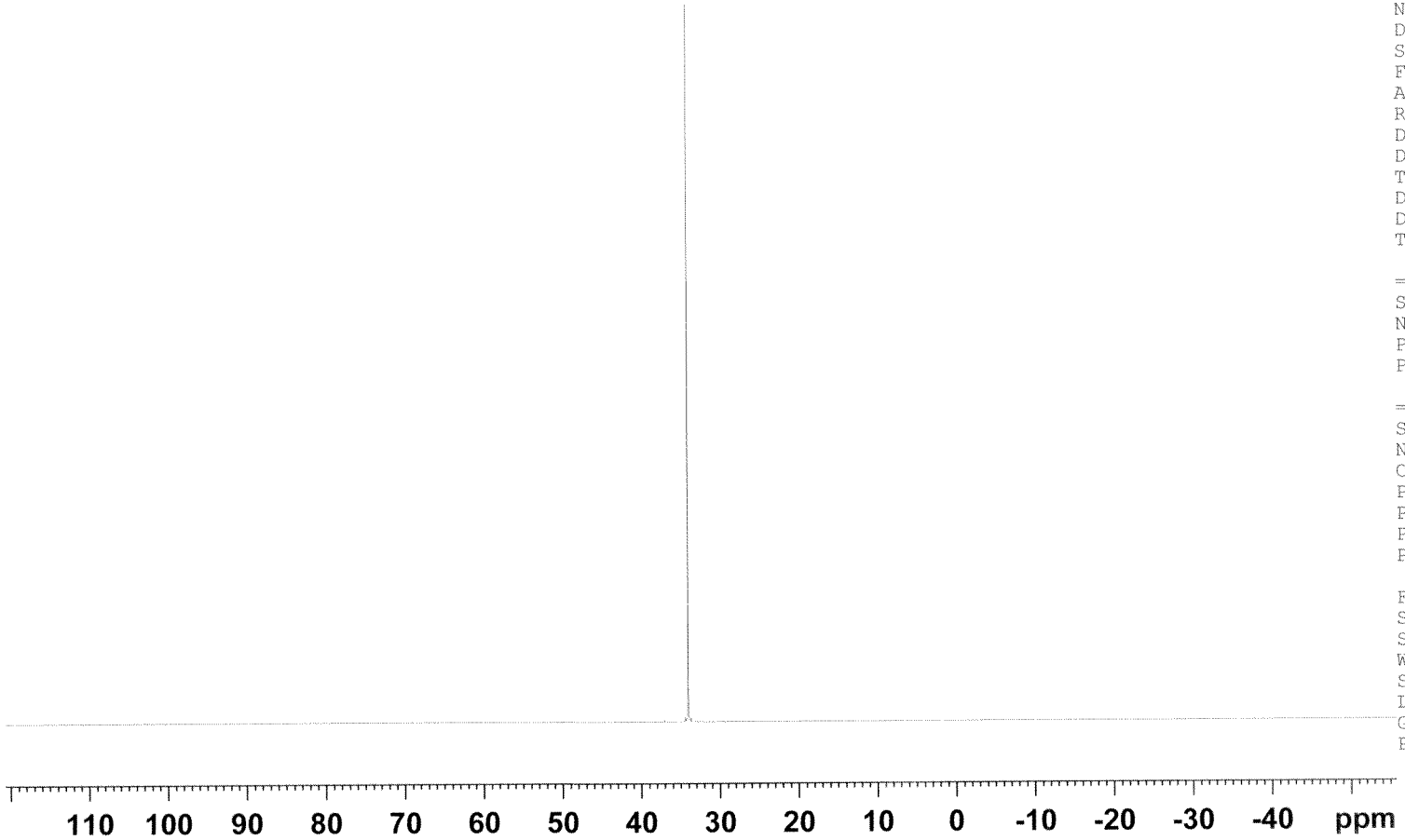


Compound (S_p)-23
^{13}C NMR

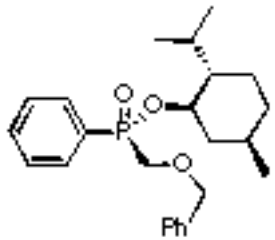




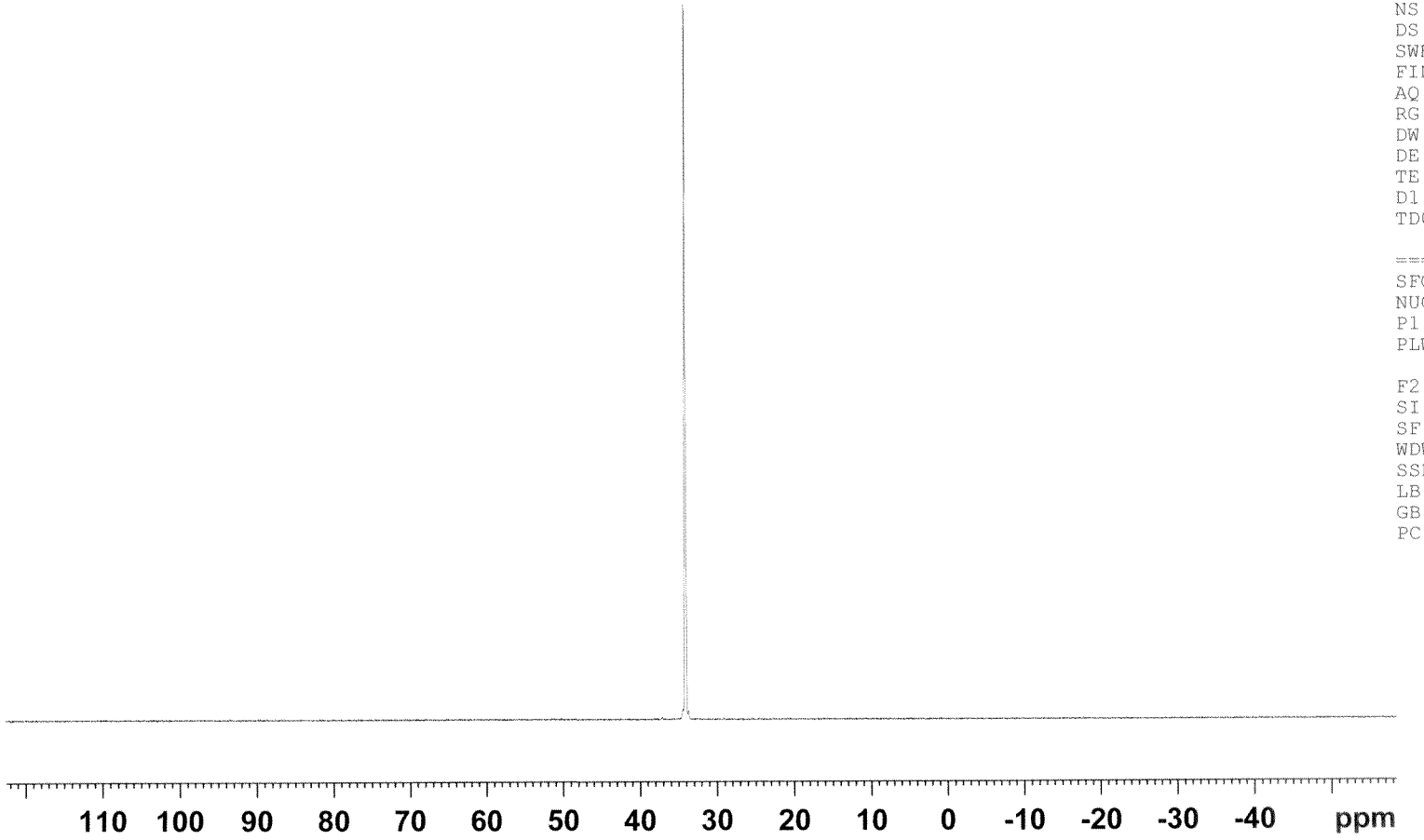
Compound (S_p)-23
$^{31}P\{^1H\}$ NMR decoupled



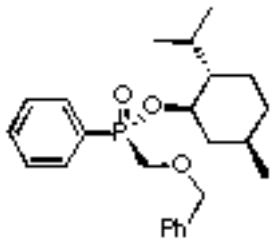

```
Current Data Parameters
NAME      OB 1816 after column
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20141107
Time                 17.36
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       4
SWH              64102.563 Hz
FIDRES            0.978127 Hz
AQ                0.5111808 sec
RG                   203.57
DW                  7.800 usec
DE                  6.50 usec
TE                   295.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

========== CHANNEL f1 =========
SFO1          161.9674942 MHz
NUC1                   31P
P1                  14.25 usec
PLW1          15.00000000 W

========== CHANNEL f2 =========
SFO2          400.1316005 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2          10.00000000 W
PLW12          0.31604999 W
PLW13          0.25600001 W

F2 - Processing parameters
SI                   32768
SF            161.9755930 MHz
WDW                     EM
SSB       0
LB                    2.00 Hz
GB        0
PC                    1.40
```



Compound (S_p)-23
$^{31}P/^{1}H$ NMR coupled

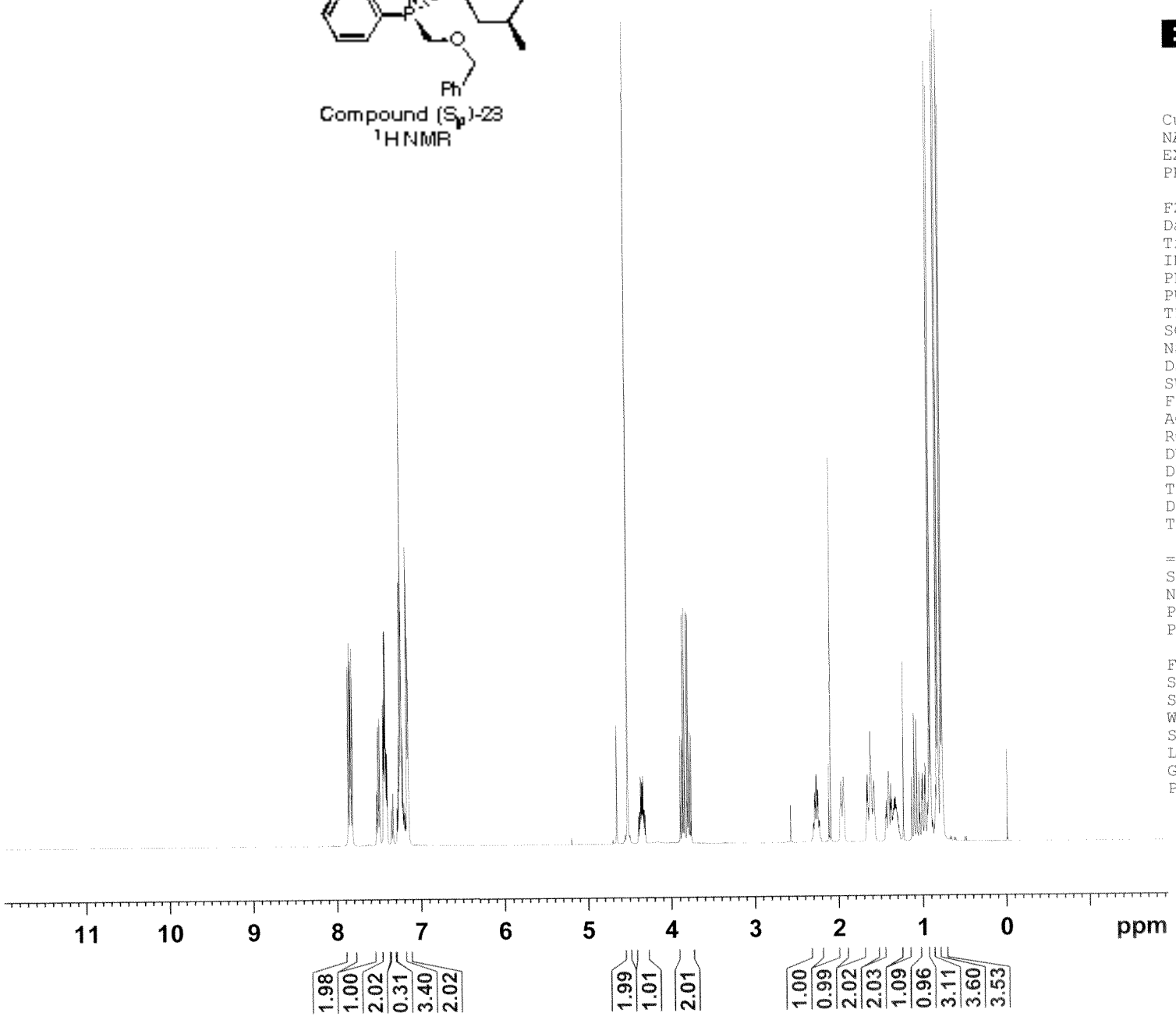



```
Current Data Parameters
NAME      OB 1816 after column
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20141107
Time                17.38
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    22
DS                     4
SWH             64102.563 Hz
FIDRES          0.978127 Hz
AQ              0.5111808 sec
RG                 203.57
DW                 7.800 usec
DE                 6.50 usec
TE                 295.5 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         161.9674942 MHz
NUC1                  31P
P1                 14.25 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 32768
SF           161.9755930 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



Compound (S_p)-23

^{1}H NMR

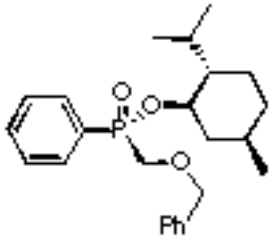


Current Data Parameters
NAME OB 1816 after column
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20141107
Time 17.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 9
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 11.05
DW 62.400 usec
DE 6.50 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1

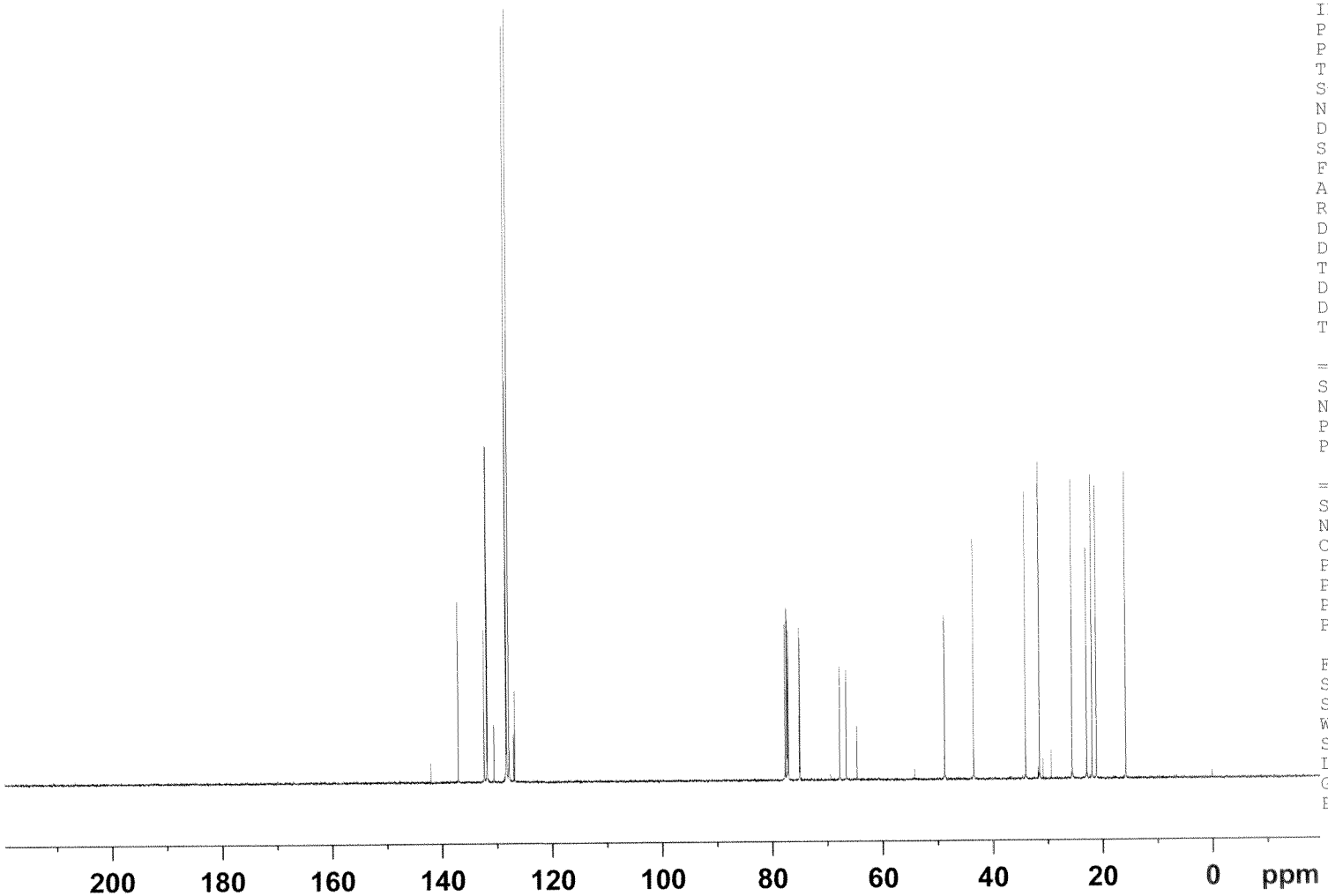
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 10.00 usec
PLW1 25.00300026 W

F2 - Processing parameters
SI 65536
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



Compound (S_P)-23

^{13}C NMR



Current Data Parameters

NAME	OB 1816 after column
EXPNO	4
PROCNO	1

F2 - Acquisition Parameters

Date_	20141107
Time	17.53
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	174
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631488 sec
RG	203.57
DW	20.800 usec
DE	6.50 usec
TE	296.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

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

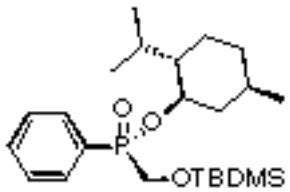
SFO1	100.6228293 MHz
NUC1	13C
P1	10.00 usec
PLW1	45.00000000 W

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

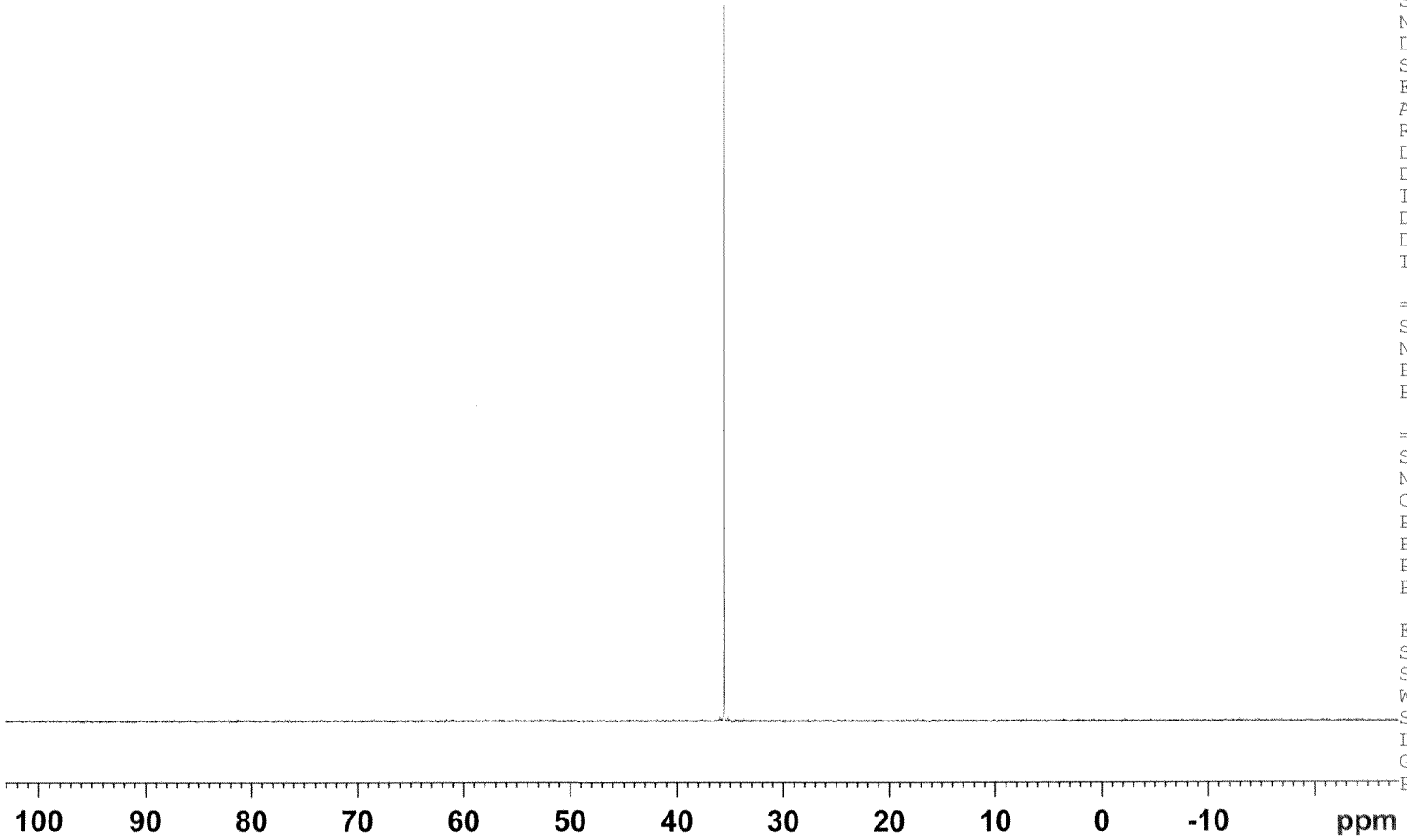
SFO2	400.1316005 MHz
NUC2	1H
CPDPRG[2	waltz16
PCPD2	90.00 usec
PLW2	10.00000000 W
PLW12	0.31604999 W
PLW13	0.25600001 W

F2 - Processing parameters

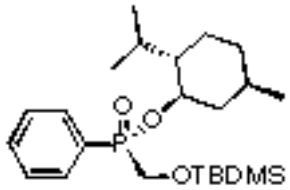
SI	32768
SF	100.6127685 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



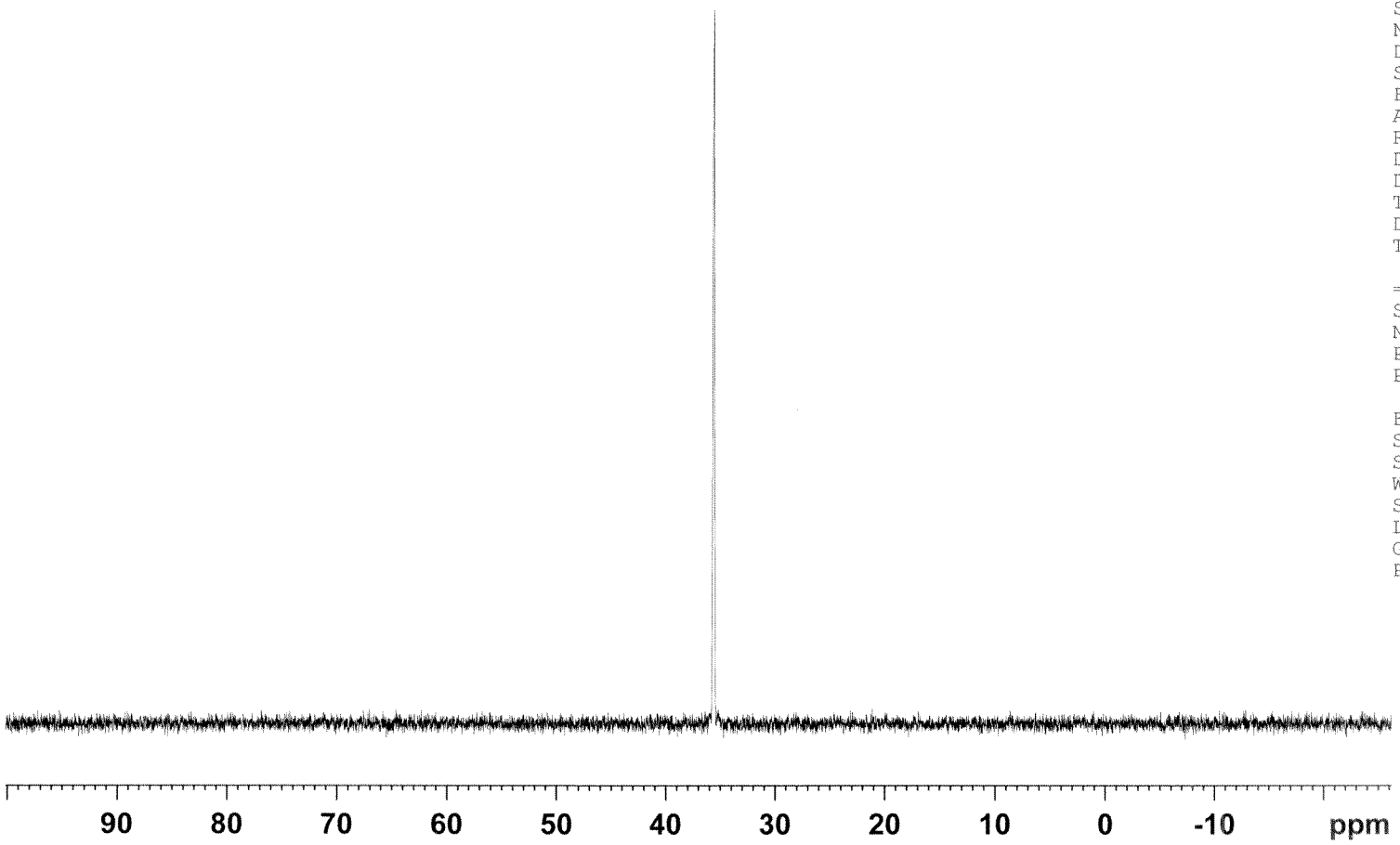
Compound (S_p)-24
$^{31}P\{^1H\}$ NMR decoupled



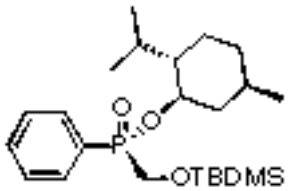

```
Current Data Parameters
NAME        OB 1584 pure
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140716
Time               10.20
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                 7.800 usec
DE                 6.50 usec
TE                294.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

========== CHANNEL f1 =========
SFO1         161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1         15.00000000 W

========== CHANNEL f2 =========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                 32768
SF           161.9755930 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



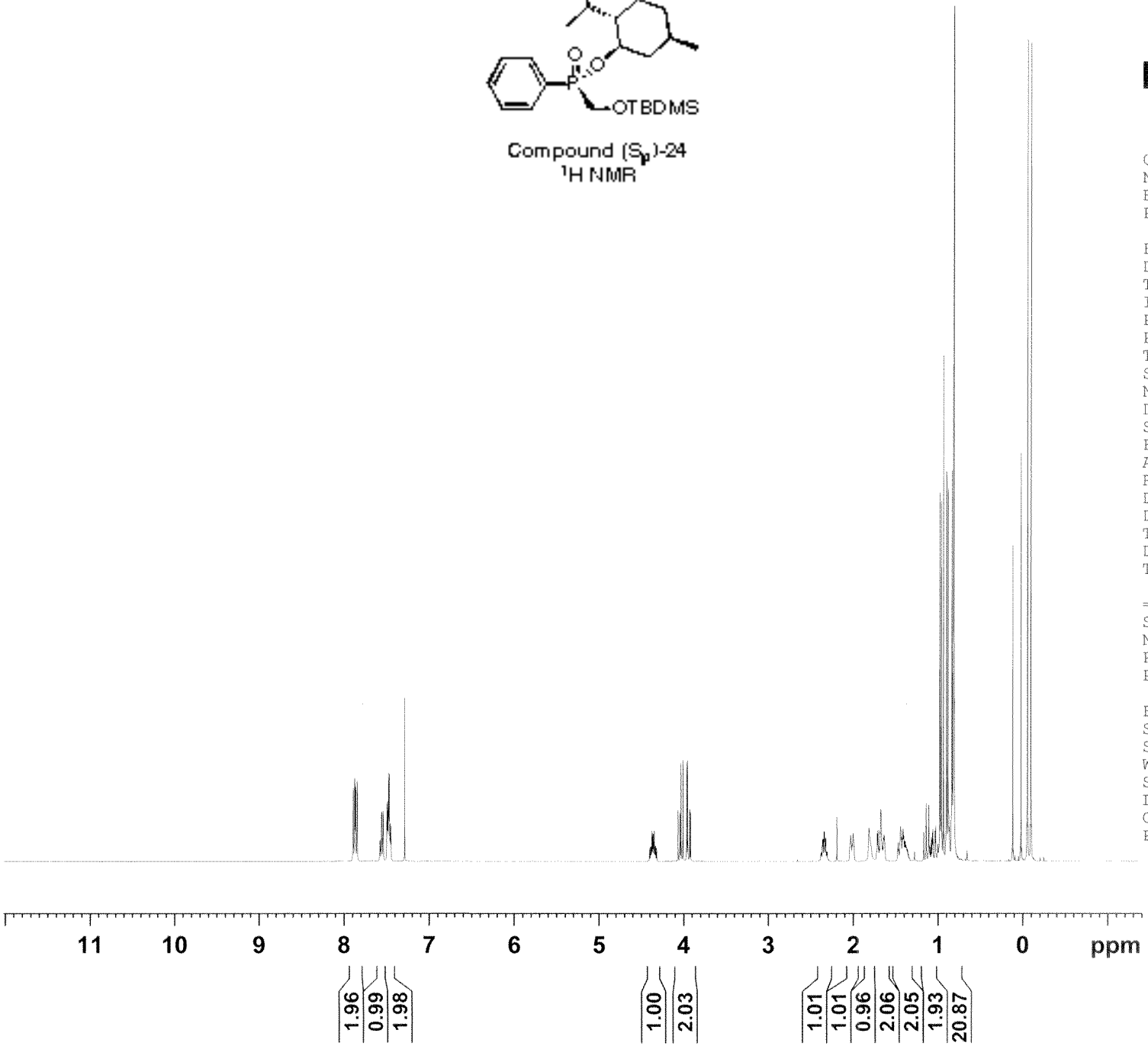
Compound (S_p)-24
$^{31}P\{^{1}H\}$ NMR coupled




```
Current Data Parameters
NAME        OB 1584 pure
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140716
Time               10.22
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                 7.800 usec
DE                 6.50 usec
TE                294.5 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         161.9674942 MHz
NUC1                 31P
P1                14.25 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                32768
SF           161.9755930 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



Compound (S_p)-24
^{1}H NMR



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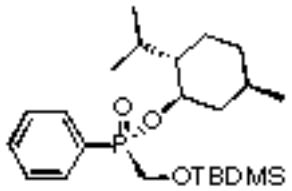
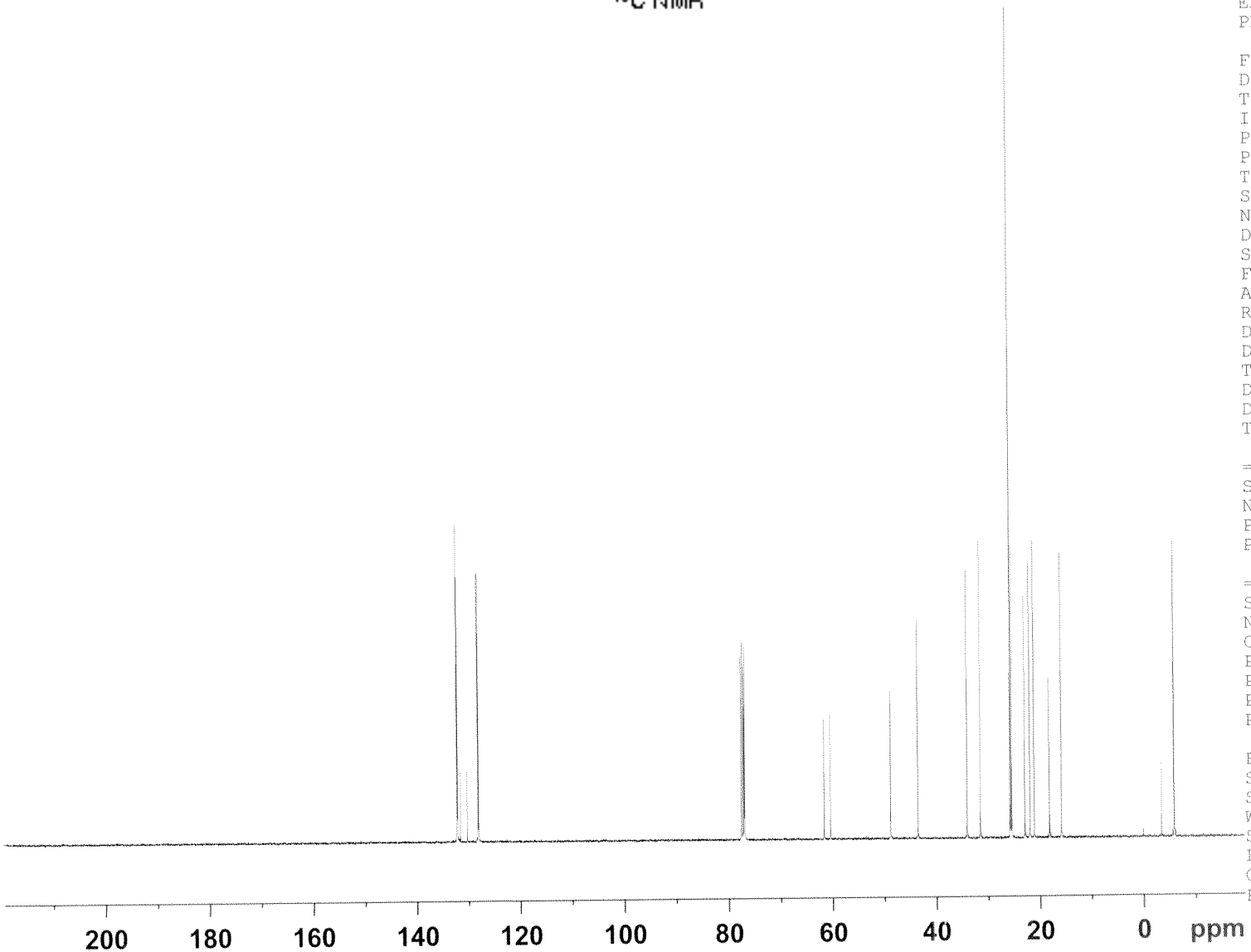
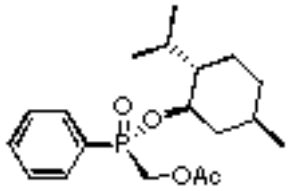
```
Current Data Parameters
NAME        OB 1582 pure
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140716
Time                10.25
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 81.67
DW                62.400 usec
DE                 6.50 usec
TE                294.4 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 10.00 usec
PLW1         25.00300026 W

F2 - Processing parameters
SI                 65536
SF           400.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



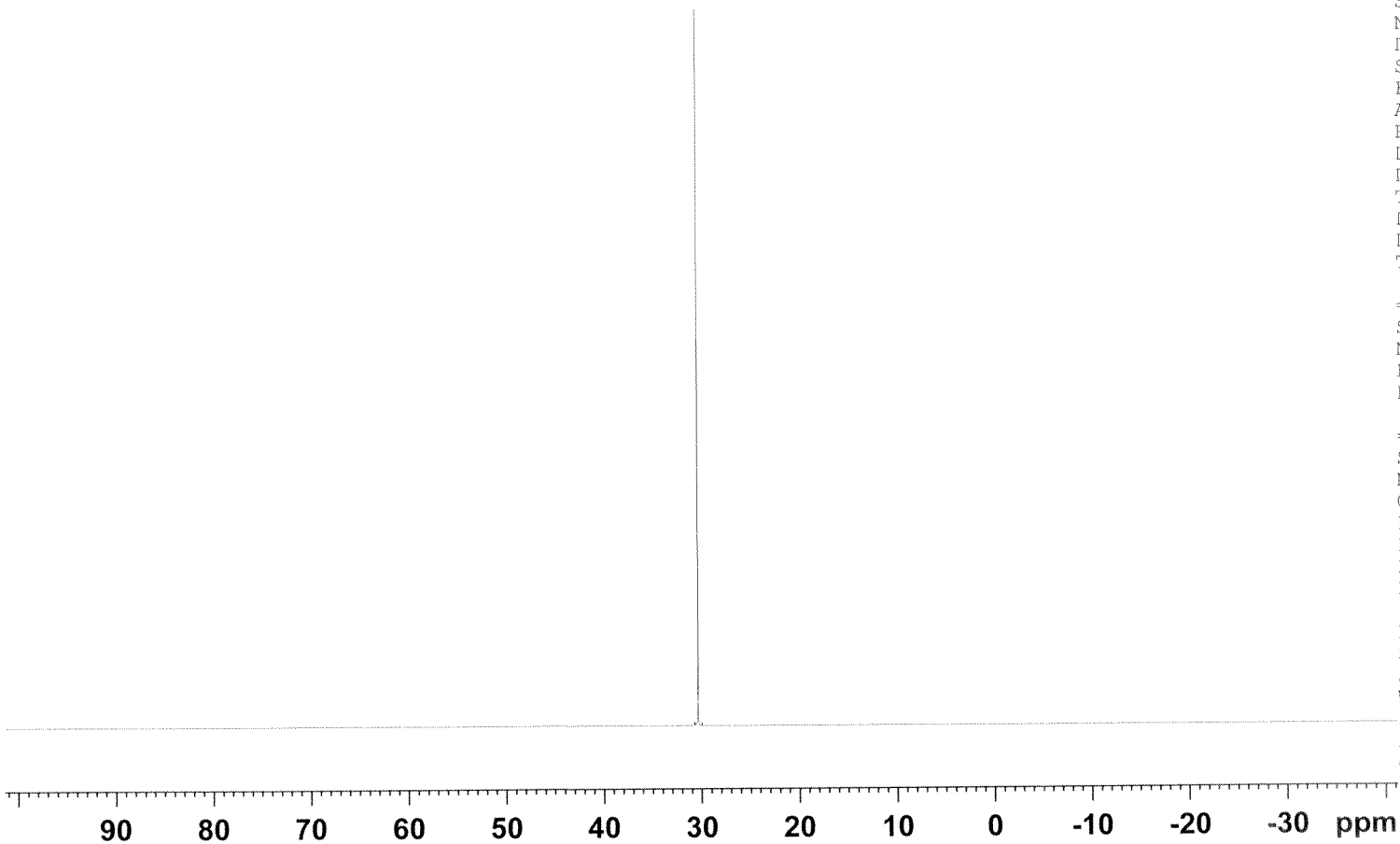
Compound (S_p)-24
^{13}C NMR



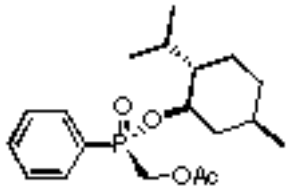

```
Current Data Parameters
NAME          OB 1584 pure
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20151123
Time_                18.16
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    273
DS                      4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                292.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

========== CHANNEL f1 ==========
SFO1          100.6228293 MHz
NUC1                    13C
P1                  10.00 usec
PLW1          45.00000000 W

========== CHANNEL f2 ==========
SFO2          400.1316005 MHz
NUC2                    1H
CPDPRG[2          waltz16
PCPD2               90.00 usec
PLW2          10.00000000 W
PLW12          0.31604999 W
PLW13          0.25600001 W

F2 - Processing parameters
SI                    32768
SF            100.6127685 MHz
WDW                      EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```



Compound (S_p)-25
$^{31}P\{^1H\}$ NMR decoupled




```
Current Data Parameters
NAME                  OB 1591
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_                20140722
Time                     9.29
INSTRUM                 spect
PROBHD   5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                Acetone
NS                          16
DS                           4
SWH                  64102.563 Hz
FIDRES                0.978127 Hz
AQ                    0.5111808 sec
RG                       203.57
DW                        7.800 usec
DE                        6.50 usec
TE                       295.6 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1               161.9674942 MHz
NUC1                       31P
P1                       14.25 usec
PLW1               15.00000000 W

======== CHANNEL f2 ========
SFO2               400.1316005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2               10.00000000 W
PLW12               0.31604999 W
PLW13               0.25600001 W

F2 - Processing parameters
SI                       32768
SF                 161.9755930 MHz
WDW                         EM
SSB                  0
LB                       1.00 Hz
GB                   0
PC                       1.40
```



Compound (S_p)-25
$^{31}P/^{1}H$ NMR coupled

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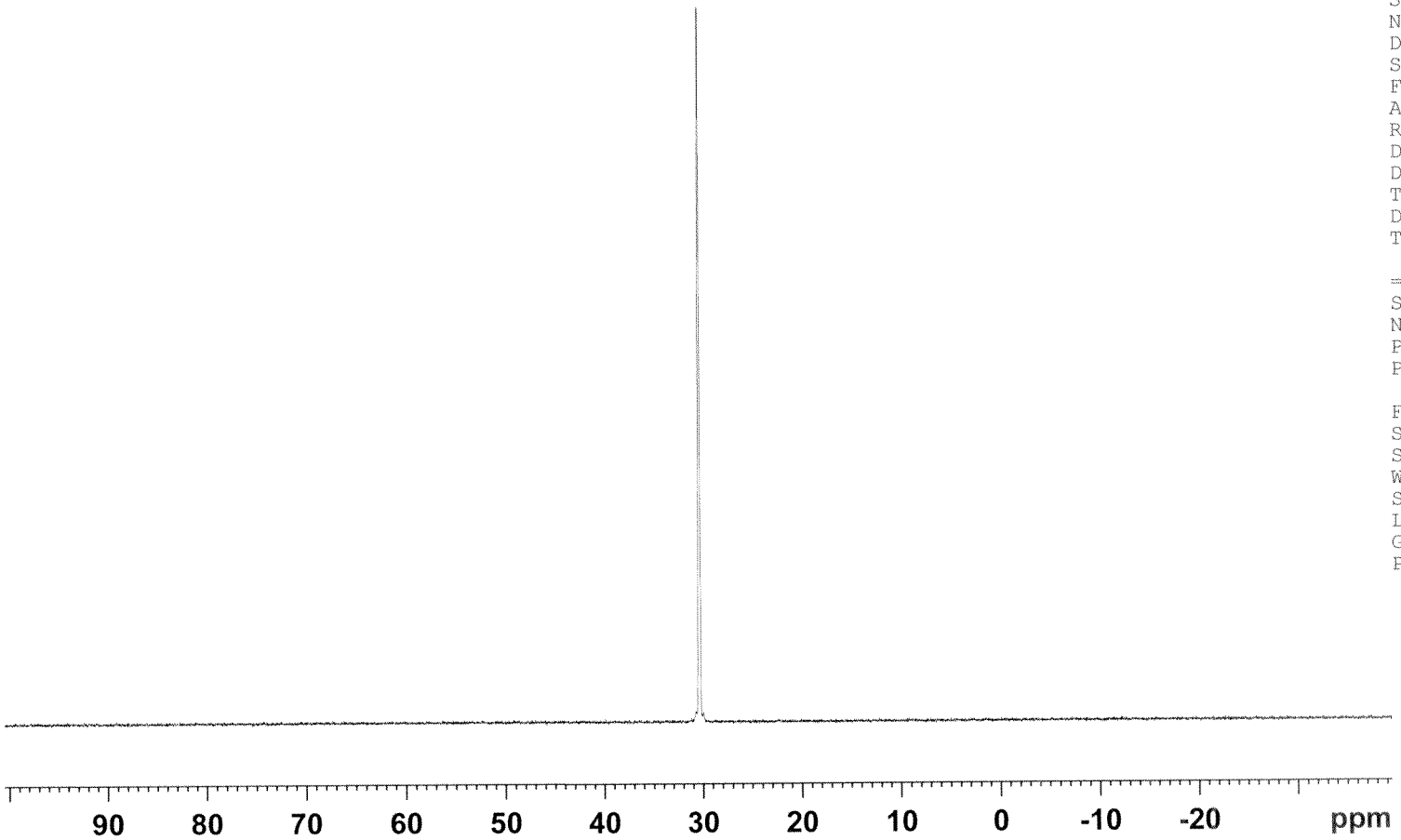
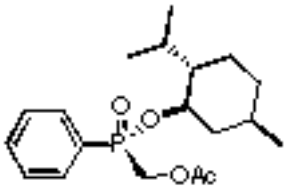
```
Current Data Parameters
NAME                  OB 1591
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20140722
Time                   9.31
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT             Acetone
NS                         22
DS                          4
SWH               64102.563 Hz
FIDRES             0.978127 Hz
AQ                 0.5111808 sec
RG                    203.57
DW                   7.800 usec
DE                    6.50 usec
TE                   295.2 K
D1                2.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1            161.9674942 MHz
NUC1                     31P
P1                    14.25 usec
PLW1           15.00000000 W

F2 - Processing parameters
SI                      32768
SF              161.9755930 MHz
WDW                        EM
SSB         0
LB                     1.00 Hz
GB          0
PC                     1.40
```





Compound (S_p)-25
$^{31}P/^{1}H$ NMR coupled

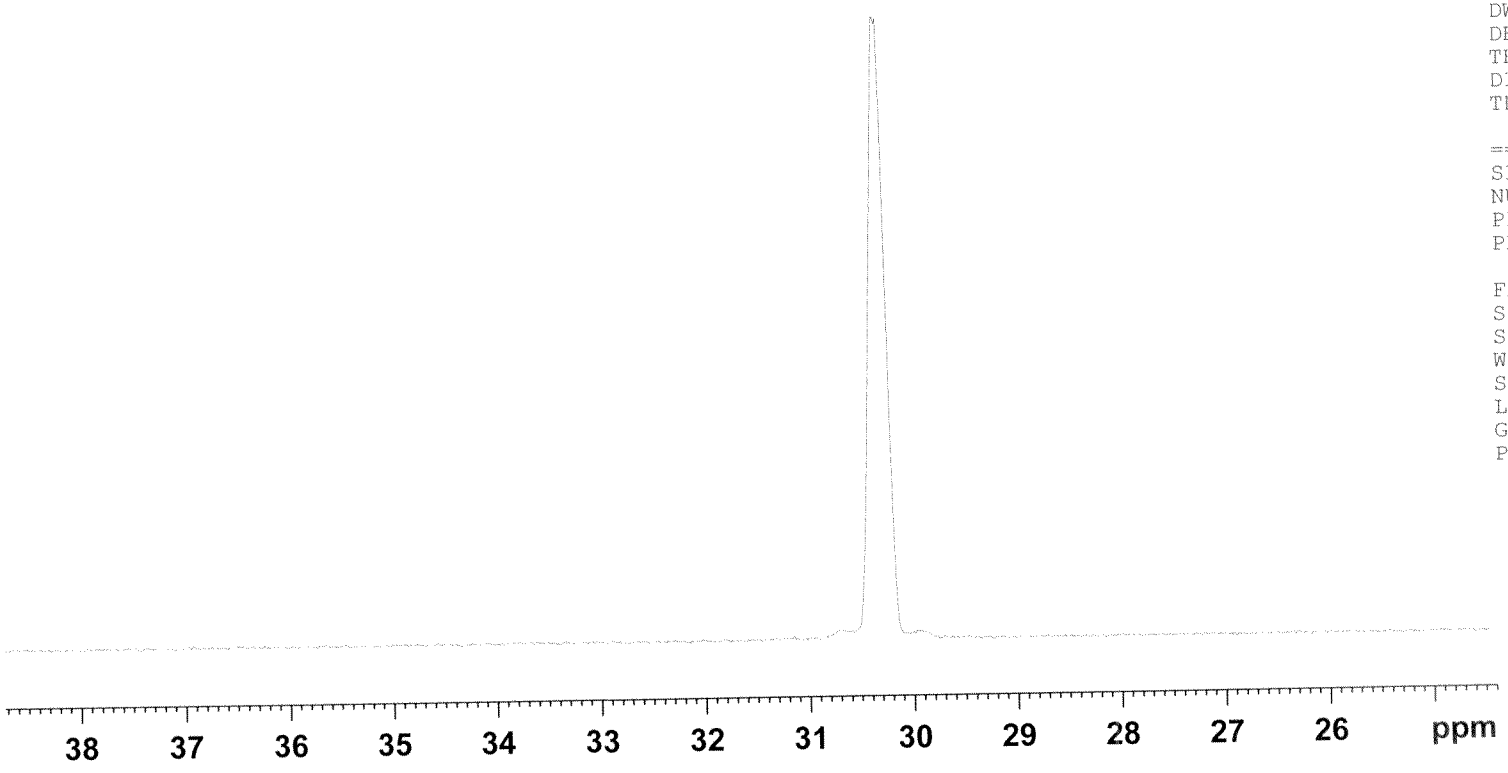
BRUKER

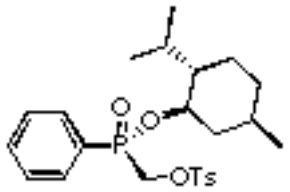
Current Data Parameters
NAME OB 1591
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20140722
Time 9.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 22
DS 4
SWH 64102.563 Hz
FIDRES 0.978127 Hz
AQ 0.5111808 sec
RG 203.57
DW 7.800 usec
DE 6.50 usec
TE 295.2 K
D1 2.00000000 sec
TD0 1

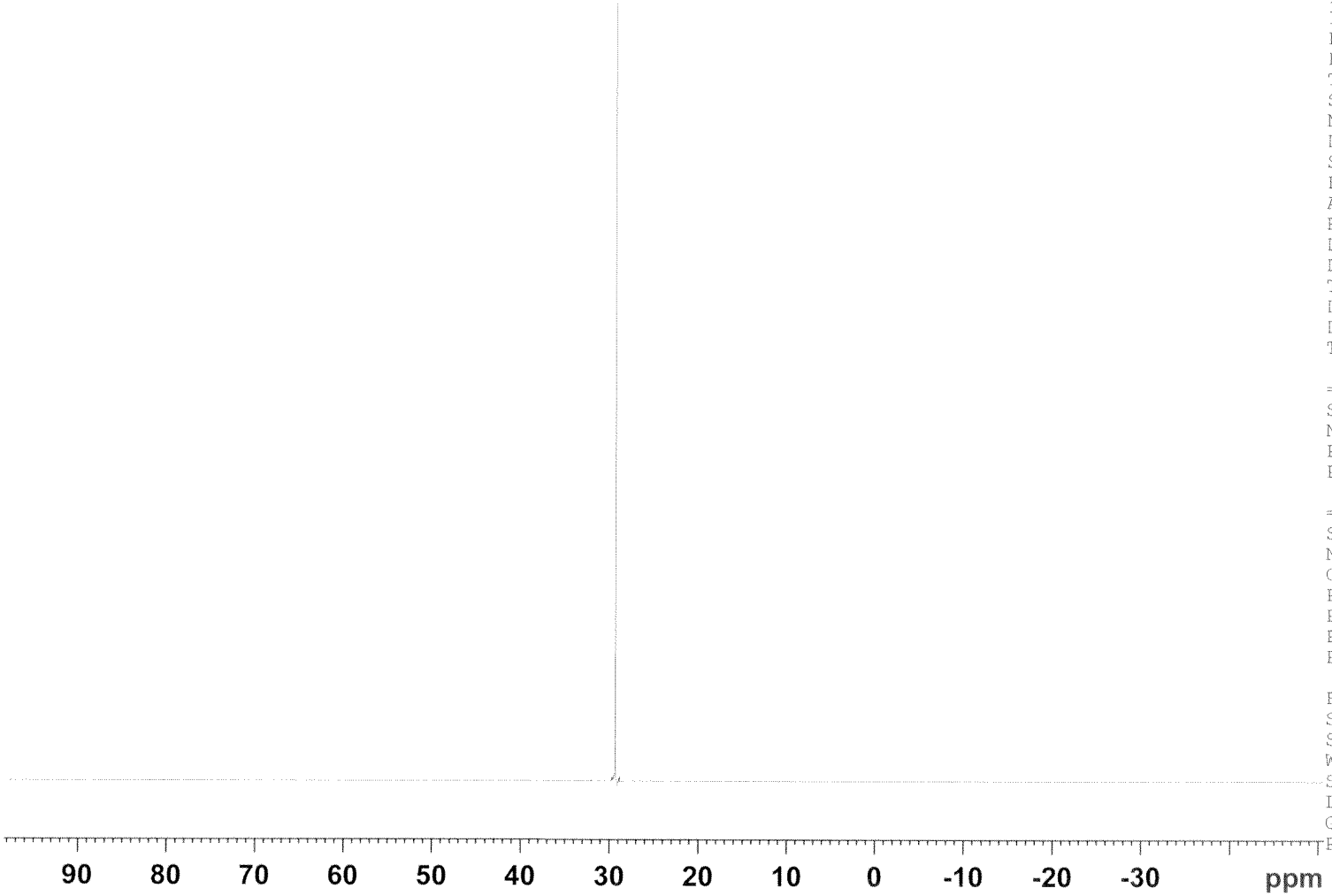
======== CHANNEL f1 ========
SFO1 161.9674942 MHz
NUC1 31P
P1 14.25 usec
PLW1 15.00000000 W

F2 - Processing parameters
SI 32768
SF 161.9755930 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

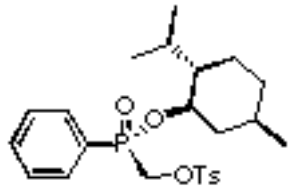




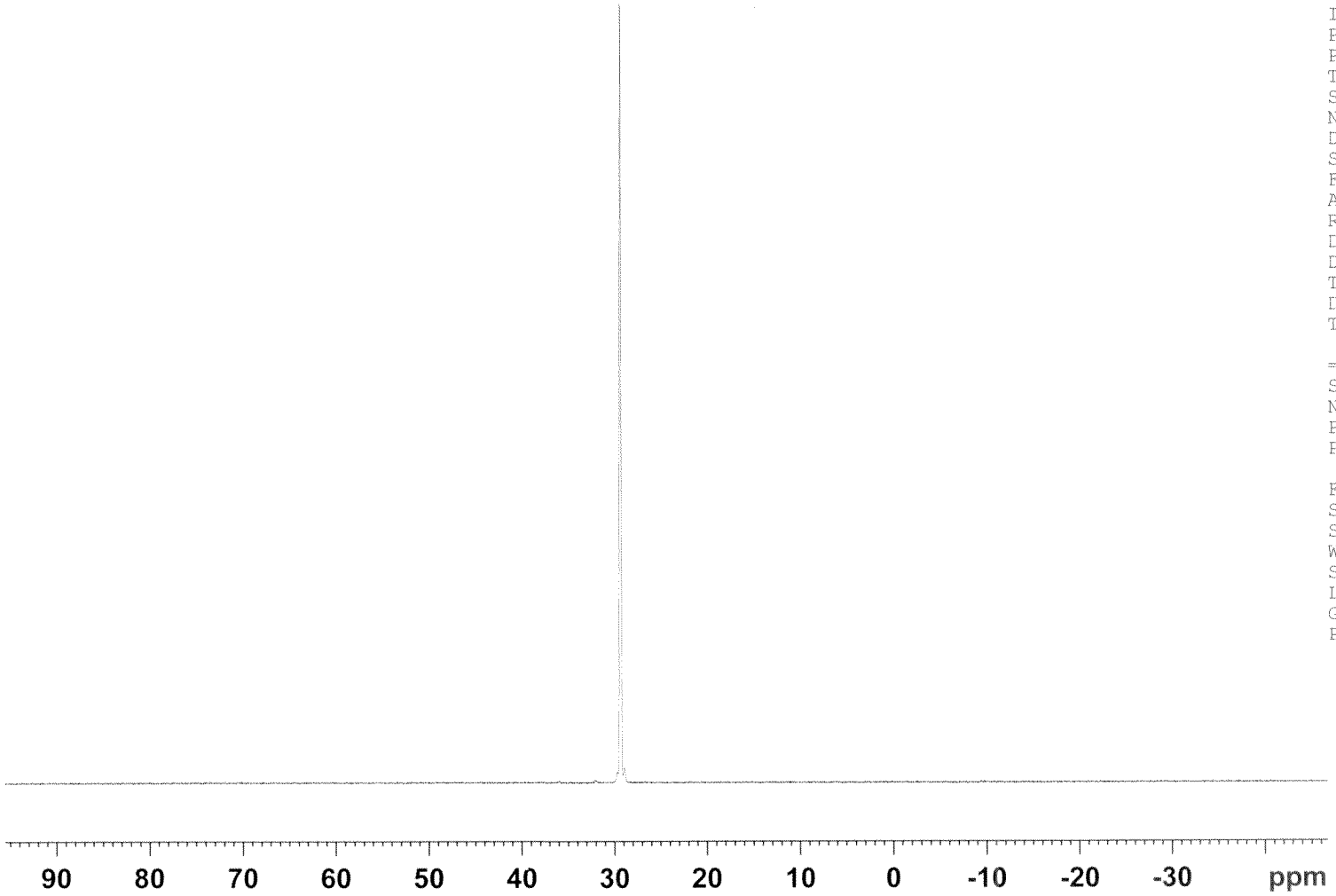
Compound (S_p)-26
$^{31}P\{^1H\}$ NMR decoupled




```
Current Data Parameters
NAME          OB 648 2nd
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151119
Time                9.45
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                 7.800 usec
DE                 6.50 usec
TE                295.2 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

========== CHANNEL f1 =========
SFO1         161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1         15.00000000 W

========== CHANNEL f2 =========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                 32768
SF           161.9755930 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



Compound (S_p)-26
$^{31}P/^{1}H$ NMR coupled



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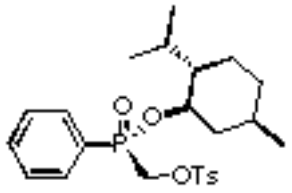
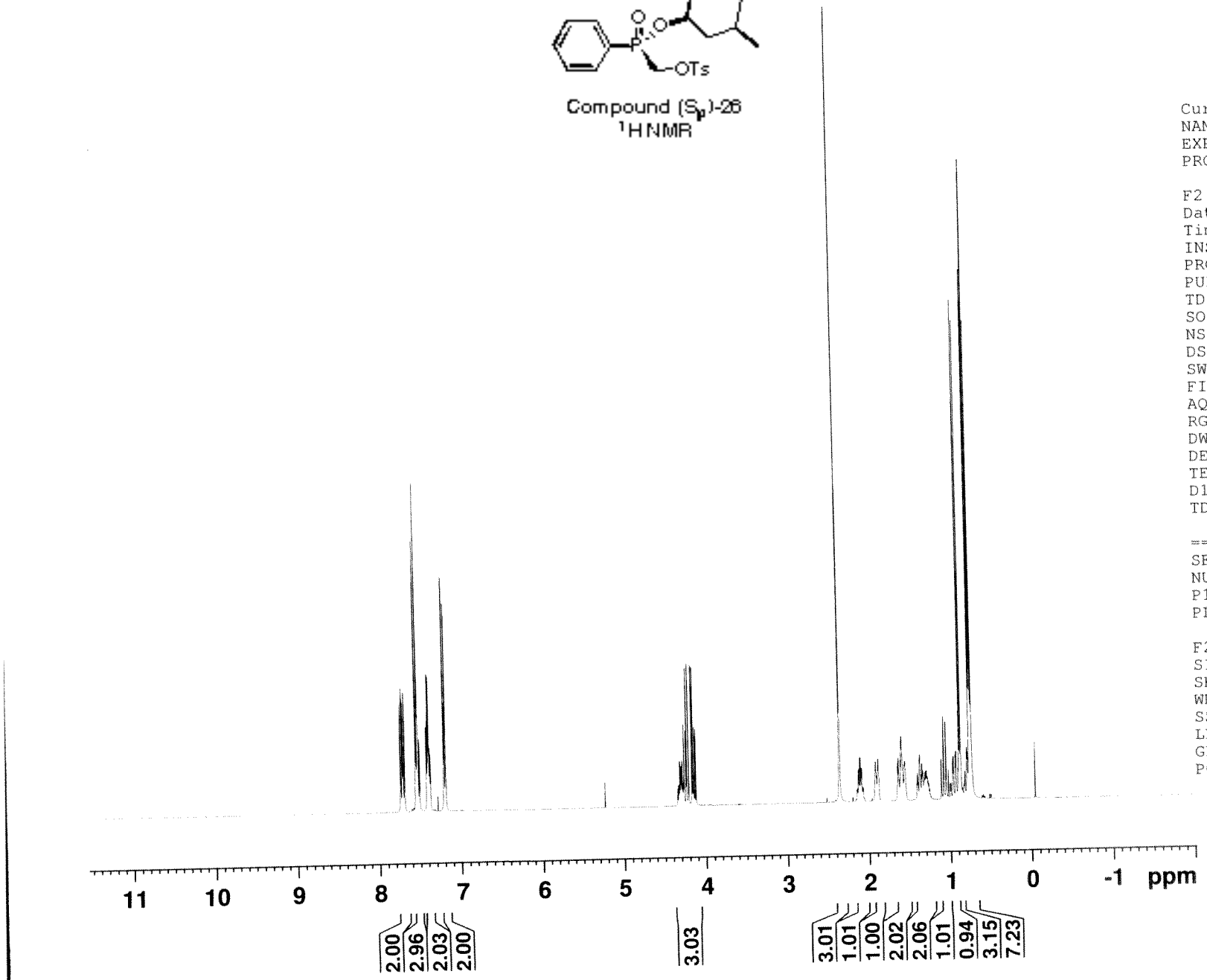
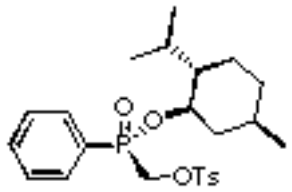
```
Current Data Parameters
NAME          OB 648 2nd
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20151119
Time                9.48
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH            64102.563 Hz
FIDRES          0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                7.800 usec
DE                6.50 usec
TE                295.2 K
D1            2.00000000 sec
TD0                    1

========== CHANNEL f1 =========
SFO1         161.9674942 MHz
NUC1                  31P
P1                 14.25 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 32768
SF           161.9755930 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



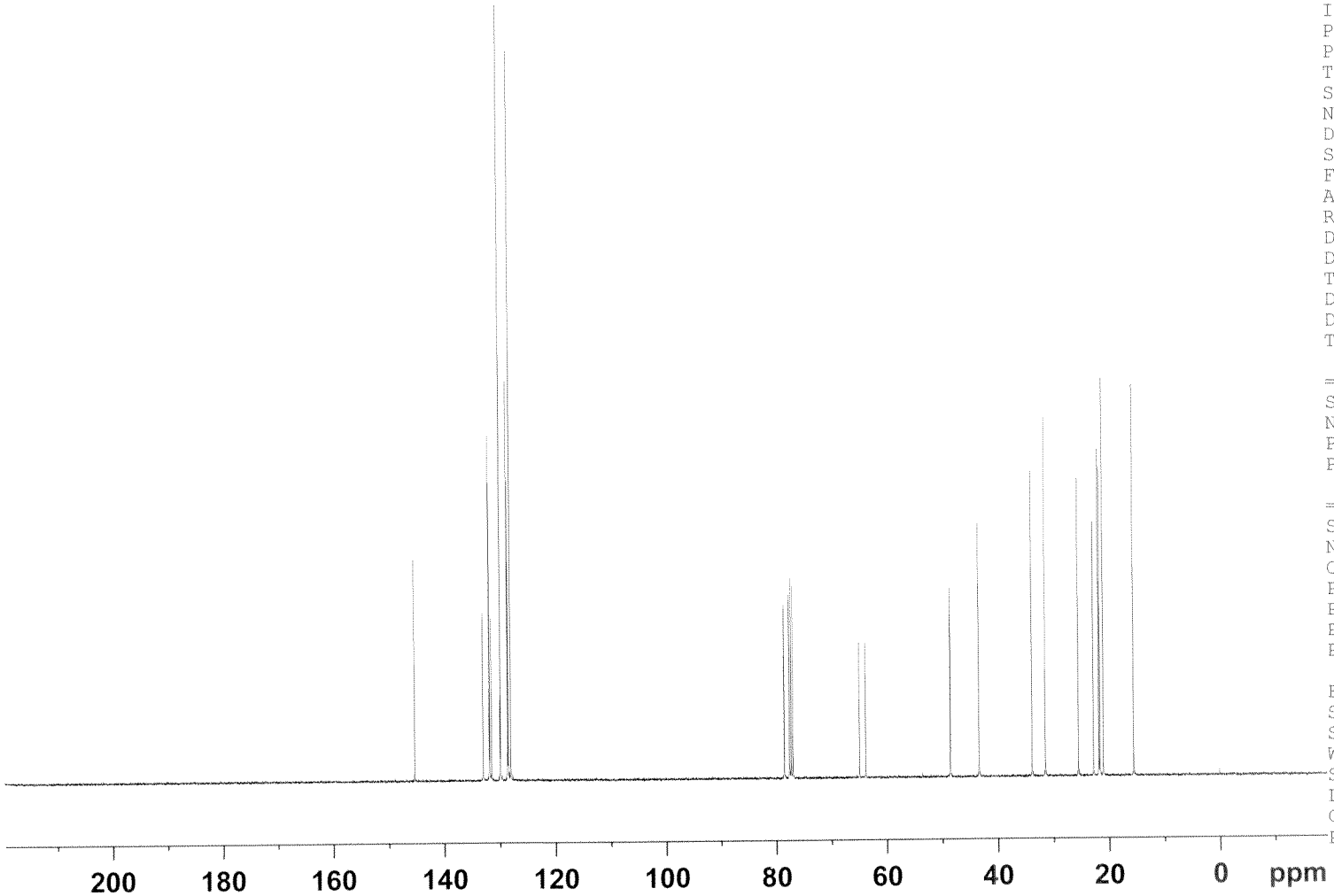
Compound (S_p)-26
^{1}H NMR




```
Current Data Parameters
NAME                  OB 648 2nd
EXPNO                          3
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20151119
Time                        9.50
INSTRUM                    spect
PROBHD    5 mm PABBO BB/
PULPROG                     zg30
TD                         65536
SOLVENT                    CDCl3
NS                            16
DS                             2
SWH                     8012.820 Hz
FIDRES                  0.122266 Hz
AQ                      4.0894465 sec
RG                         12.96
DW                        62.400 usec
DE                         6.50 usec
TE                        295.2 K
D1                      1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                  400.1324710 MHz
NUC1                          1H
P1                        10.00 usec
PLW1                  25.00300026 W

F2 - Processing parameters
SI                         65536
SF                    400.1300000 MHz
WDW                           EM
SSB                  0
LB                        0.30 Hz
GB                   0
PC                         1.00
```



Compound (S_p)-26
^{13}C NMR



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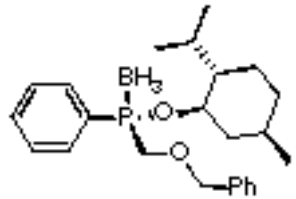
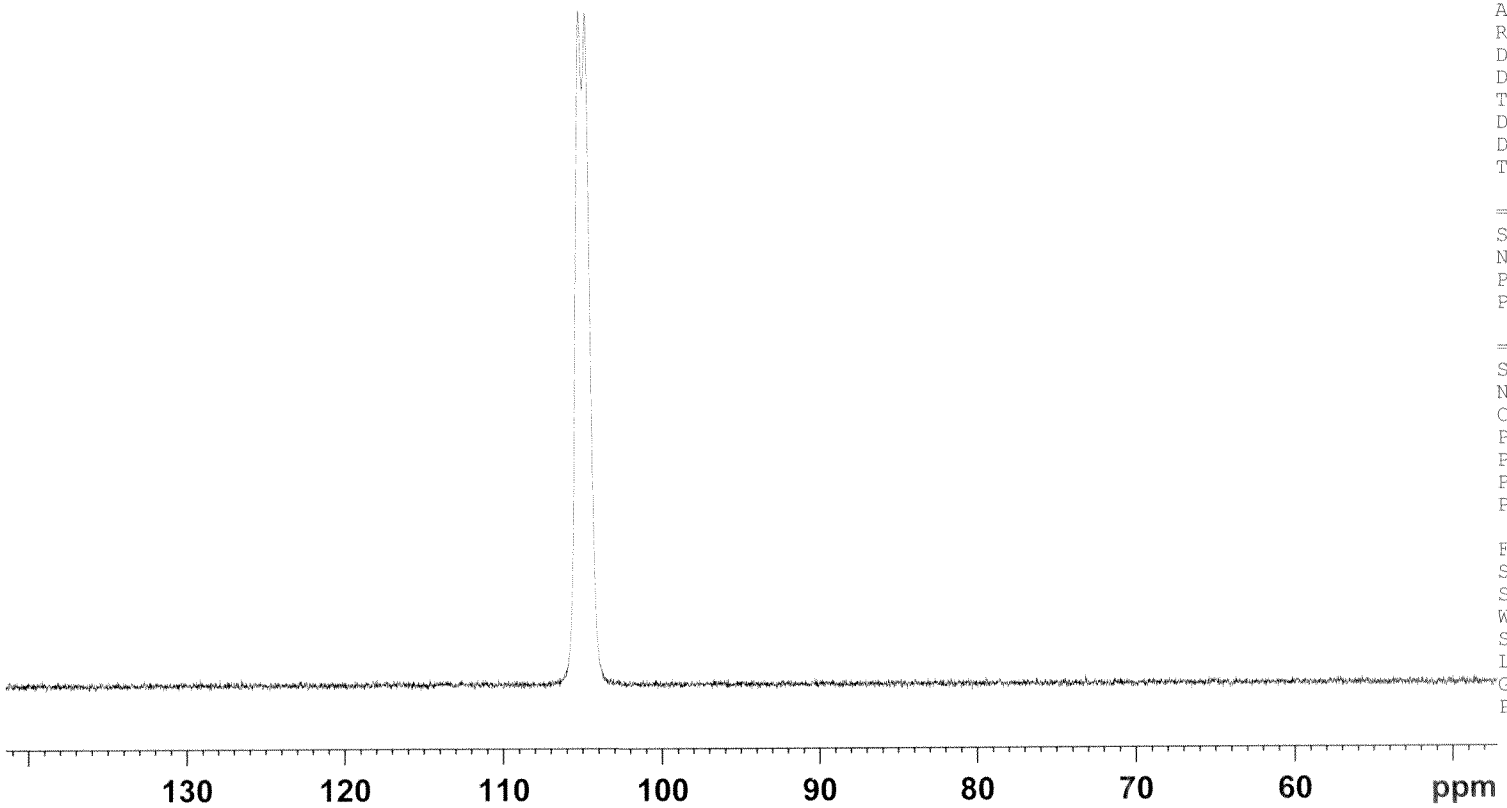
```
Current Data Parameters
NAME                  OB 648 2nd
EXPNO                          4
PROCNO                         1

F2 - Acquisition Parameters
Date_                   20151119
Time                       10.09
INSTRUM                    spect
PROBHD    5 mm PABBO BB/
PULPROG                  zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                          298
DS                            4
SWH                 24038.461 Hz
FIDRES               0.366798 Hz
AQ                   1.3631488 sec
RG                      203.57
DW                      20.800 usec
DE                       6.50 usec
TE                      295.1 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                           1

======== CHANNEL f1 =========
SFO1               100.6228293 MHz
NUC1                       13C
P1                      10.00 usec
PLW1                45.00000000 W

======== CHANNEL f2 =========
SFO2               400.1316005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                   90.00 usec
PLW2                10.00000000 W
PLW12                0.31604999 W
PLW13                0.25600001 W

F2 - Processing parameters
SI                        32768
SF                 100.6127685 MHz
WDW                          EM
SSB                 0
LB                       1.00 Hz
GB                  0
PC                       1.40
```

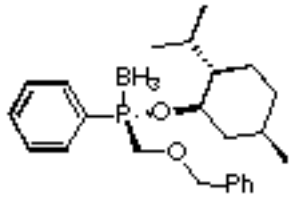


Compound (S_p)-27

$^{31}P\{^1H\}$ NMR decoupled



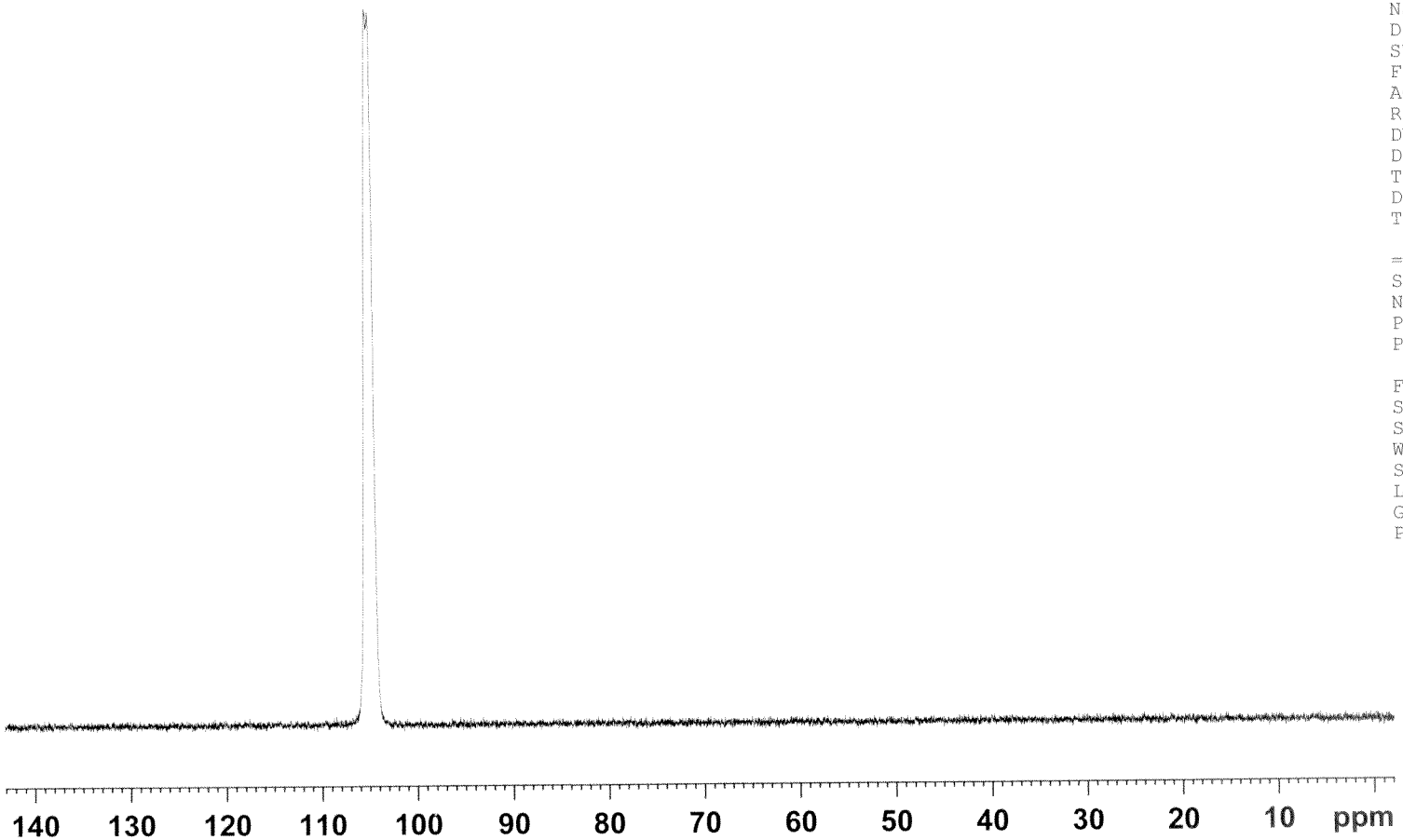

```
Current Data Parameters
NAME      OB 1707 after column
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_             20140911
Time                 17.17
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       4
SWH              64102.563 Hz
FIDRES           0.978127 Hz
AQ               0.5111808 sec
RG                  203.57
DW                  7.800 usec
DE                   6.50 usec
TE                  294.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          161.9674942 MHz
NUC1                  31P
P1                  14.25 usec
PLW1          15.00000000 W

======== CHANNEL f2 ========
SFO2          400.1316005 MHz
NUC2                  1H
CPDPRG[2           waltz16
PCPD2               90.00 usec
PLW2          10.00000000 W
PLW12          0.31604999 W
PLW13          0.25600001 W

F2 - Processing parameters
SI                   32768
SF            161.9755930 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

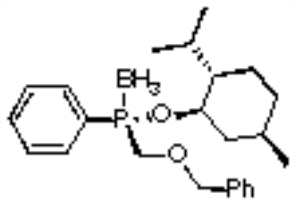


Compound (S_p)-27
$^{31}P\{^1H\}$ NMR coupled

BRUKER

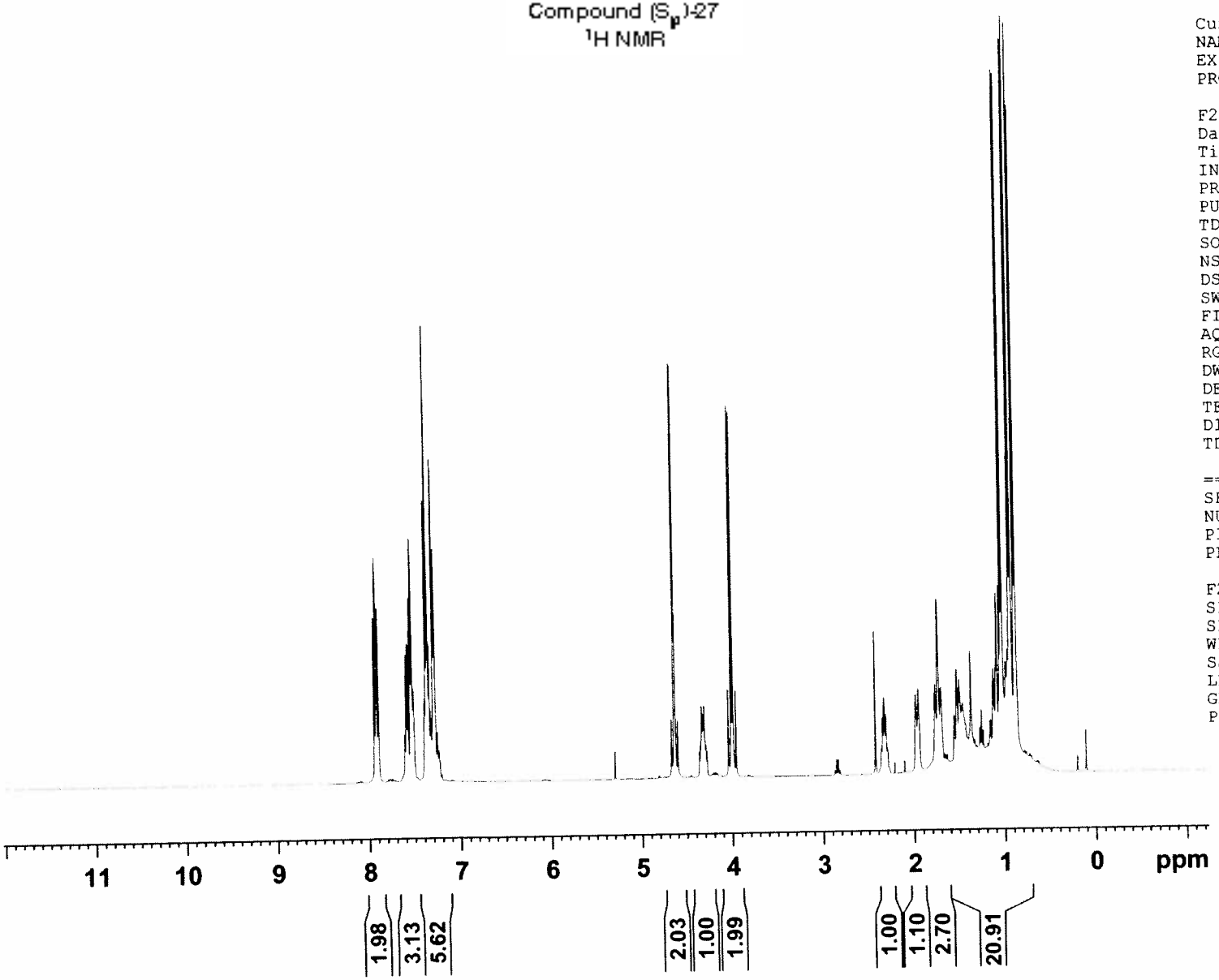
```
Current Data Parameters
NAME      OB 1707 after column
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20140911
Time                 17.20
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      4
SWH              64102.563 Hz
FIDRES            0.978127 Hz
AQ                0.5111808 sec
RG                   203.57
DW                  7.800 usec
DE                  6.50 usec
TE                  294.5 K
D1              2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          161.9674942 MHz
NUC1                  31P
P1                 14.25 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                   32768
SF            161.9755930 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



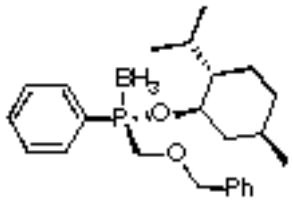


Compound (S_p)-27

^{1}H NMR

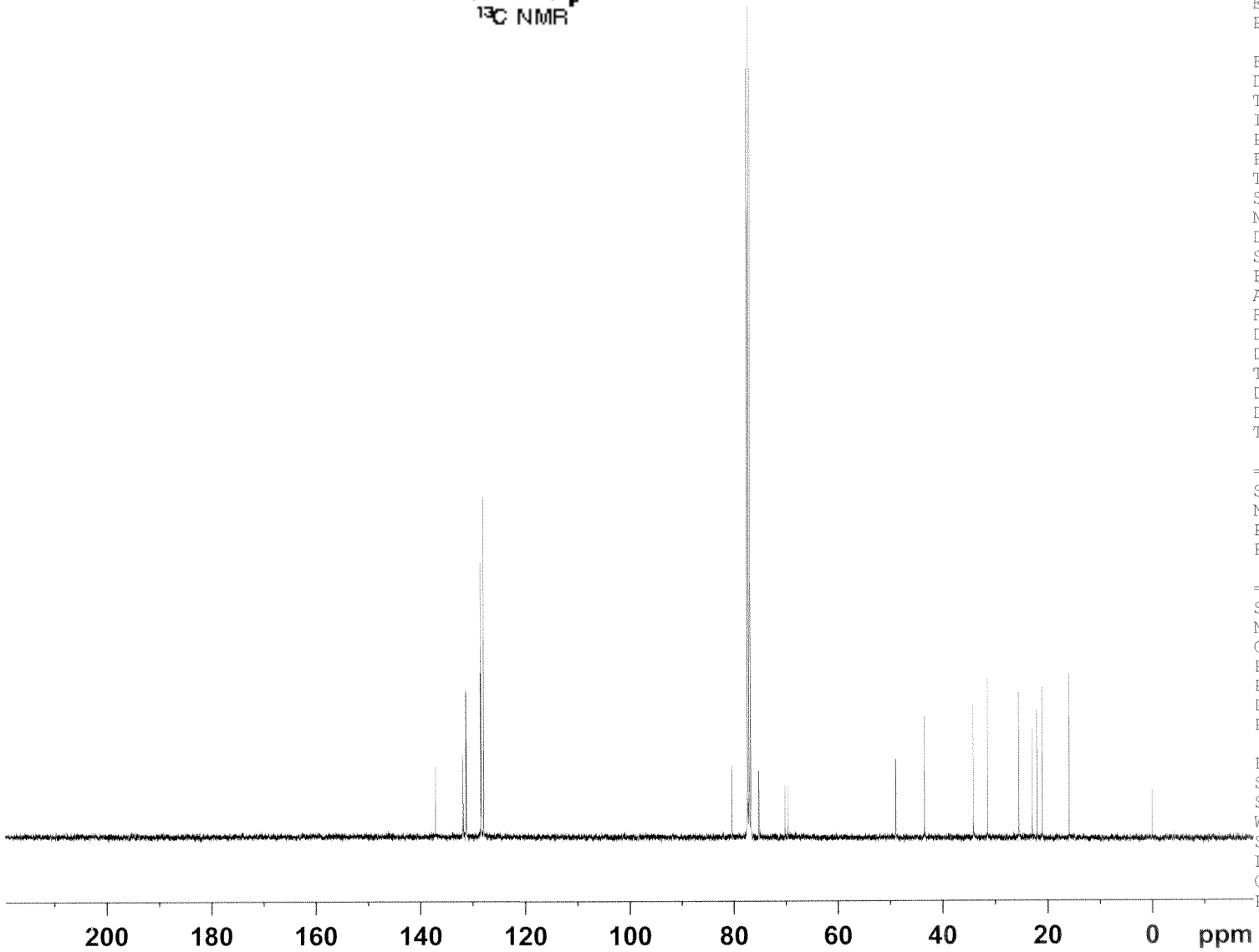



```
Current Data Parameters
NAME      OB 1707 after column
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_             20140911
Time                17.24
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDC13
NS                     11
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  10.22
DW                62.400 usec
DE                 6.50 usec
TE                294.6 K
D1             1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                   1H
P1                 10.00 usec
PLW1          25.00300026 W

F2 - Processing parameters
SI                  65536
SF            400.1300000 MHz
WDW                    EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

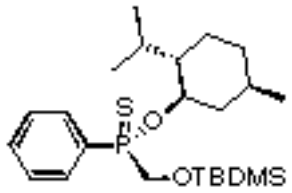


Compound (S_p)-27

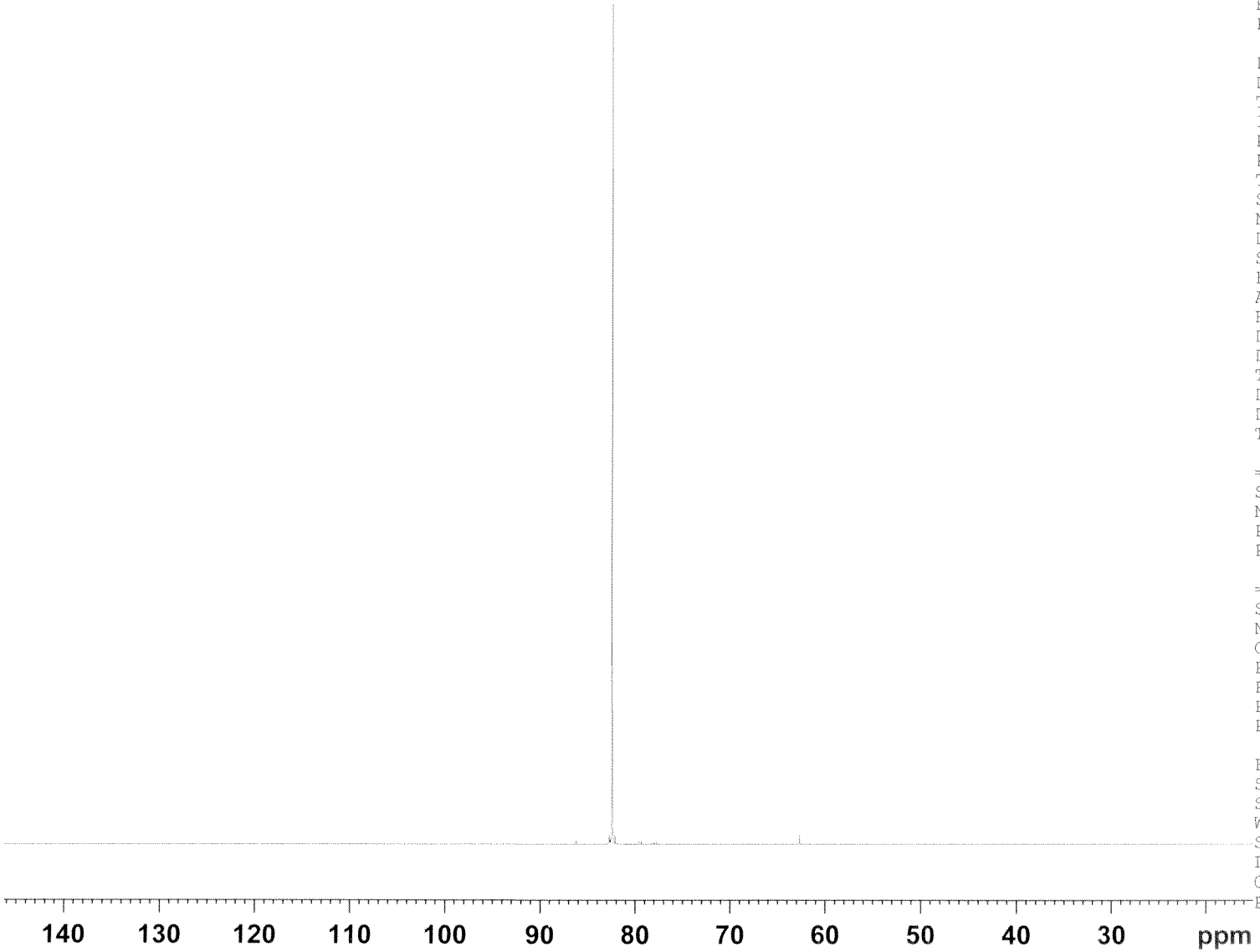
^{13}C NMR



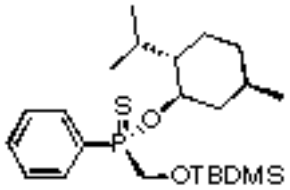

```
Current Data Parameters
NAME         OB1707 pure
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20141215
Time               18.54
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  773
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                296.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1        45.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                 32768
SF          100.6127685 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



Compound (P_S)-28a
$^{31}P\{^1H\}$ NMR decoupled




```
Current Data Parameters
NAME       OB 1588 pure2
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20151124
Time              11.00
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE                292.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1        15.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



Compound (R_p)-28a
$^{31}P\{^1H\}$ NMR coupled

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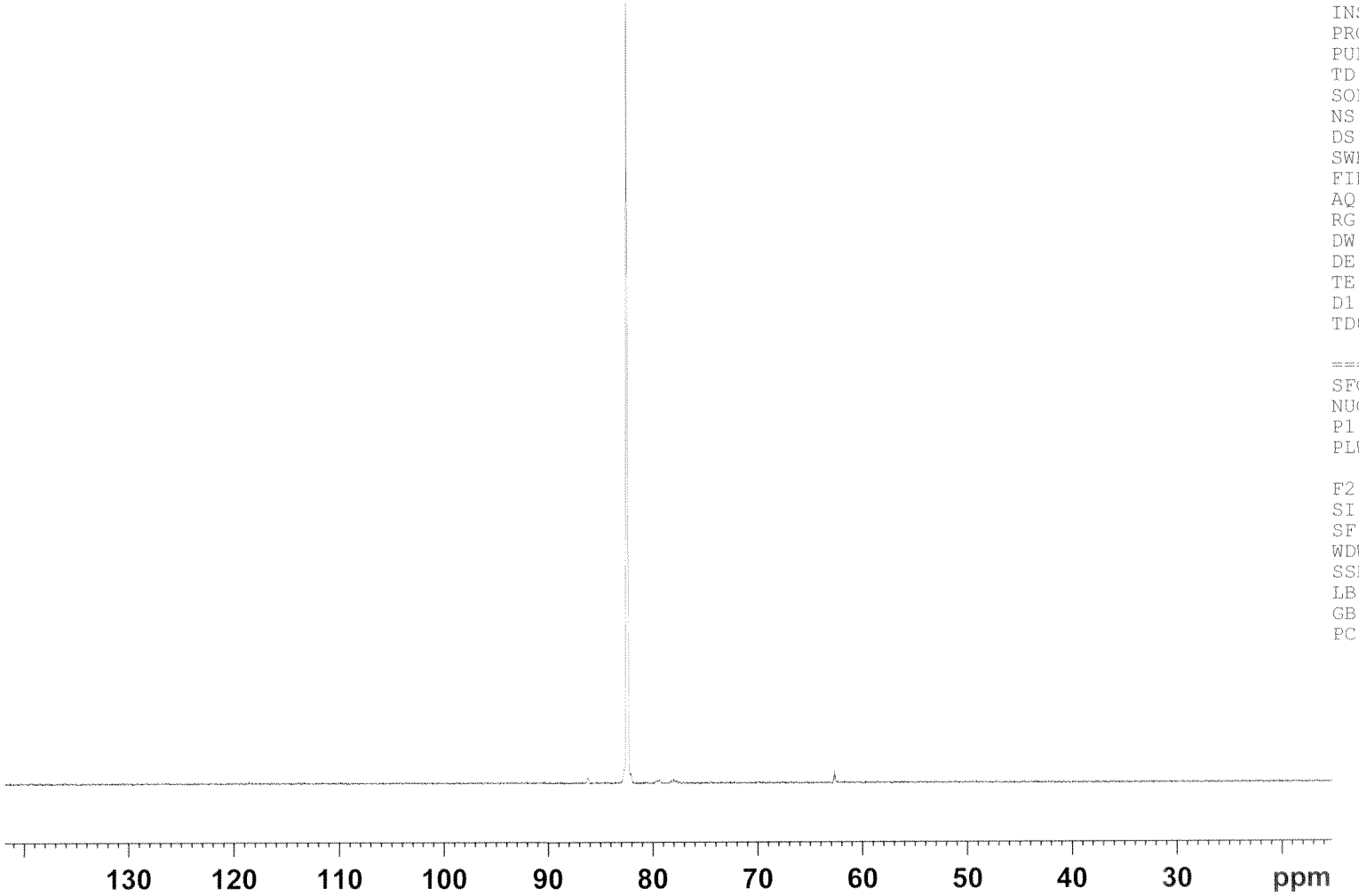
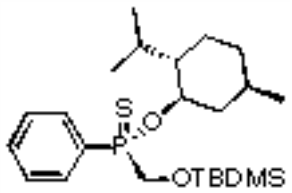
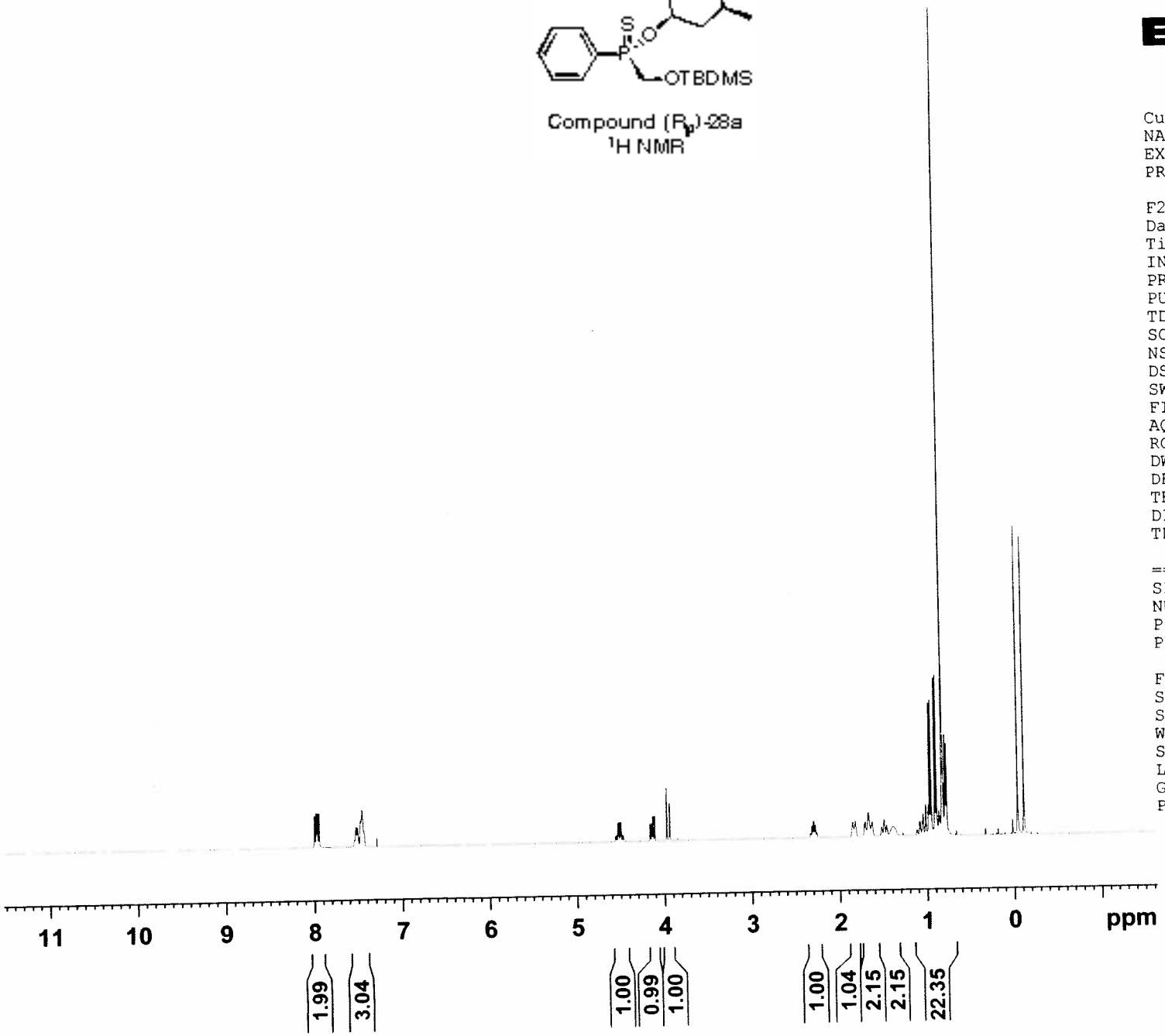
```
Current Data Parameters
NAME       OB 1588 pure2
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20151124
Time               11.02
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH            64102.563 Hz
FIDRES          0.978127 Hz
AQ              0.5111808 sec
RG                203.57
DW                7.800 usec
DE                6.50 usec
TE                291.7 K
D1             2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         161.9674942 MHz
NUC1                 31P
P1                14.25 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 32768
SF           161.9755930 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```





Compound (R_p)-28a
^{1}H NMR



Current Data Parameters
NAME OB 1588 pure2
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters

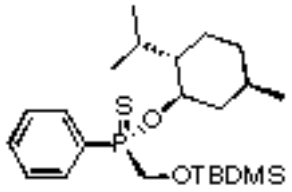
Date_	20151124
Time	11.04
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894465 sec
RG	12.96
DW	62.400 usec
DE	6.50 usec
TE	291.7 K
D1	1.00000000 sec
TD0	1

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

SFO1	400.1324710 MHz
NUC1	1H
P1	10.00 usec
PLW1	25.00300026 W

F2 - Processing parameters

SI	65536
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



Compound (R_p)-28a
^{13}C NMR

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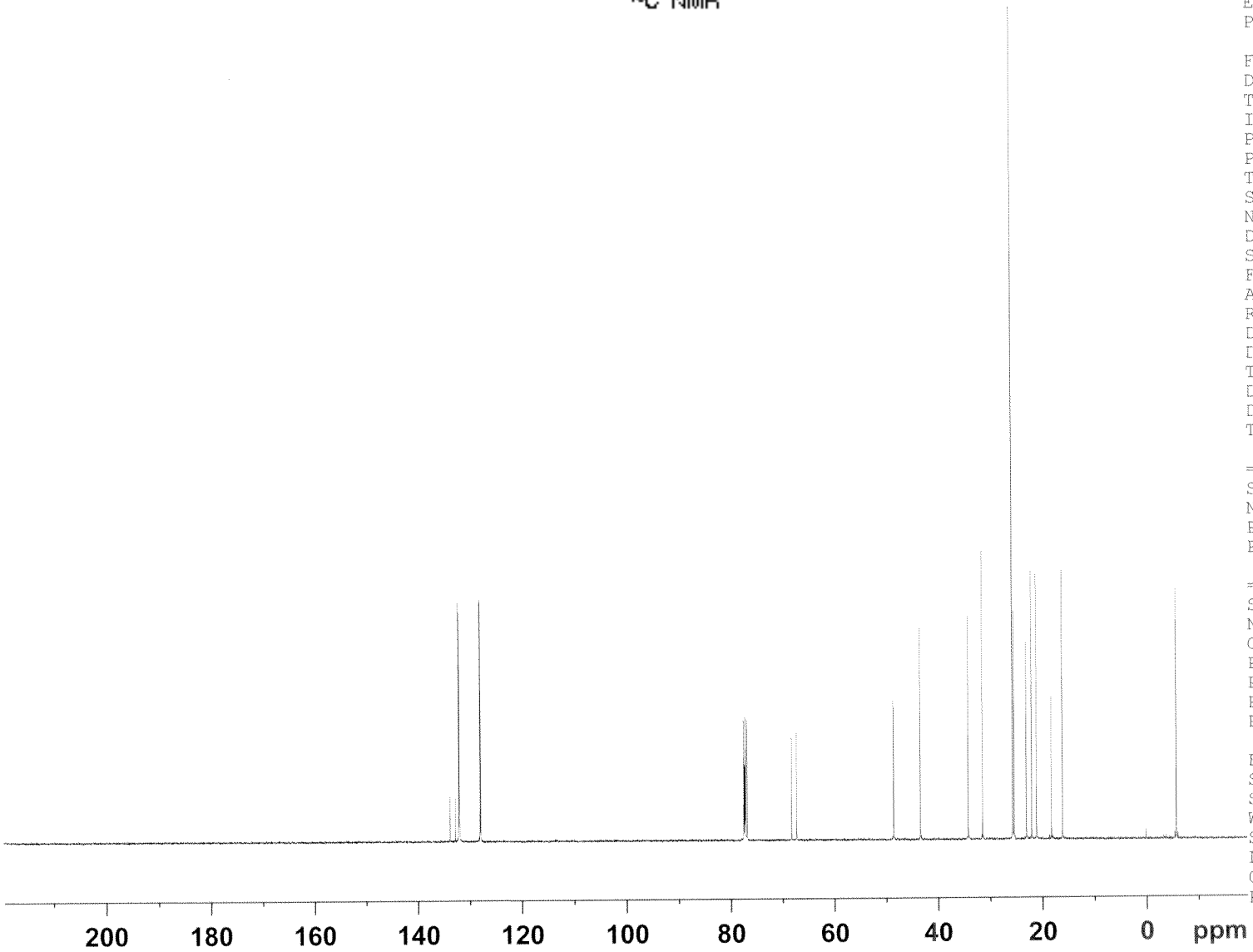
Current Data Parameters
NAME OB 1588 pure2
EXPNO 4
PROCNO 1

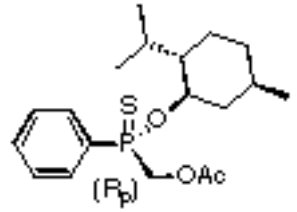
F2 - Acquisition Parameters
Date_ 20151124
Time 11.26
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 327
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 203.57
DW 20.800 usec
DE 6.50 usec
TE 292.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

CHANNEL f1
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
PLW1 45.00000000 W

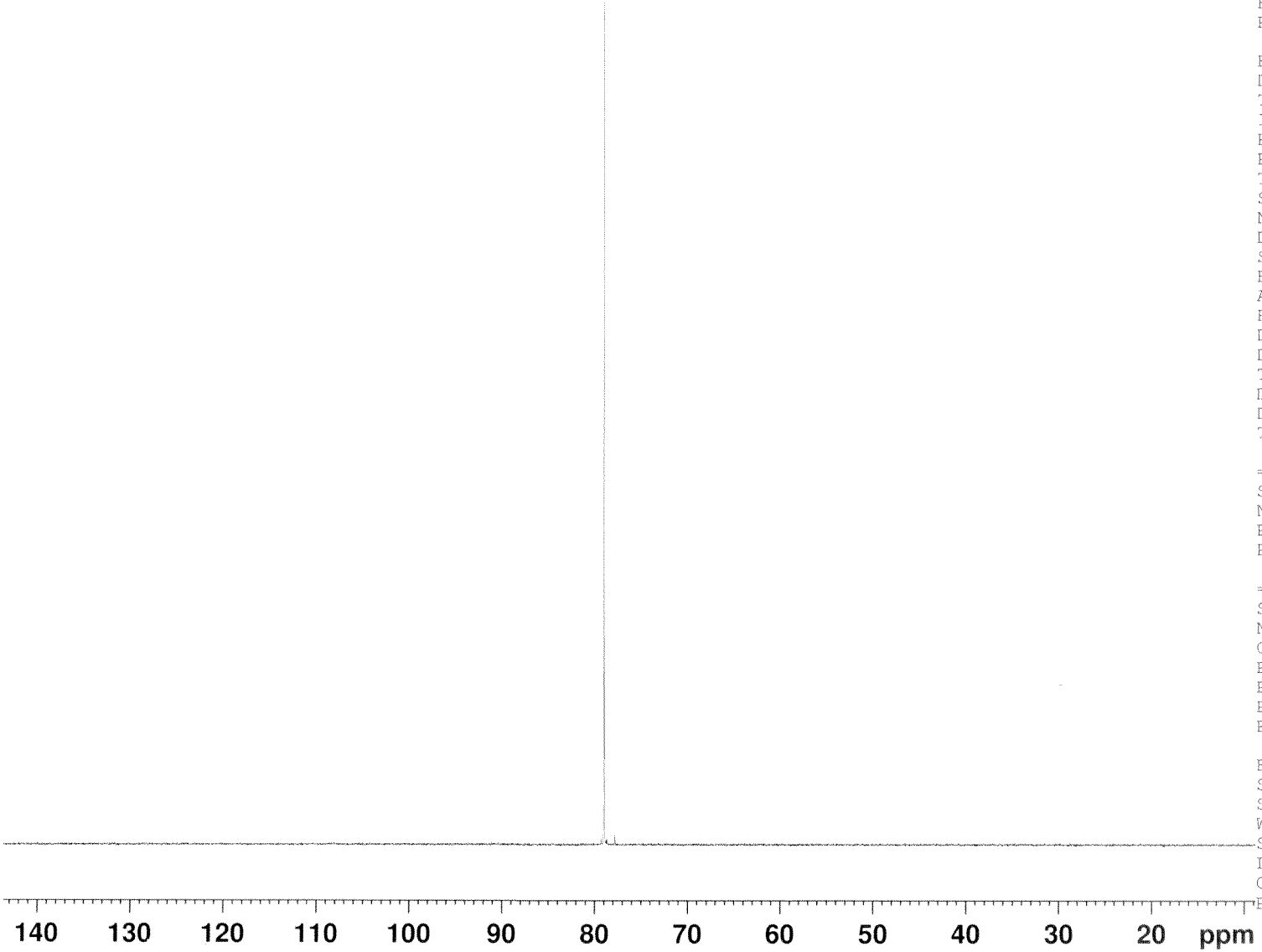
CHANNEL f2
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 10.00000000 W
PLW12 0.31604999 W
PLW13 0.25600001 W

F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

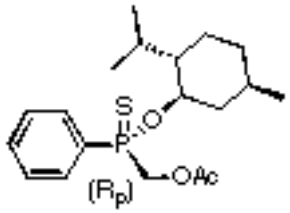




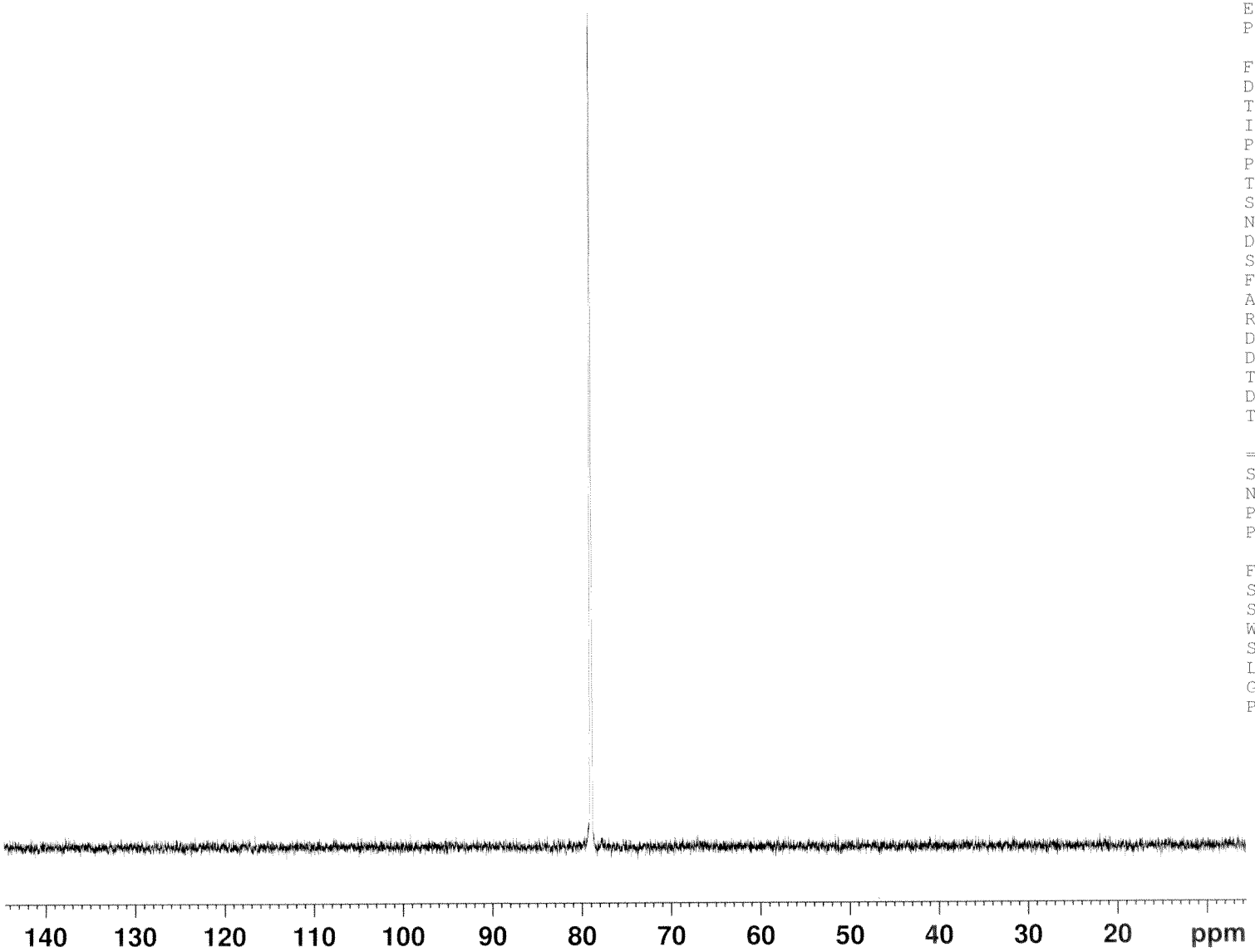
Compound (R_p)-28b
$^{31}P\{^1H\}$ NMR decoupled



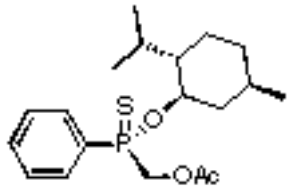

```
Current Data Parameters
NAME        OB 1595 pure
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140723
Time               19.43
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                 7.800 usec
DE                 6.50 usec
TE                295.3 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1        15.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                 32768
SF          161.9755930 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



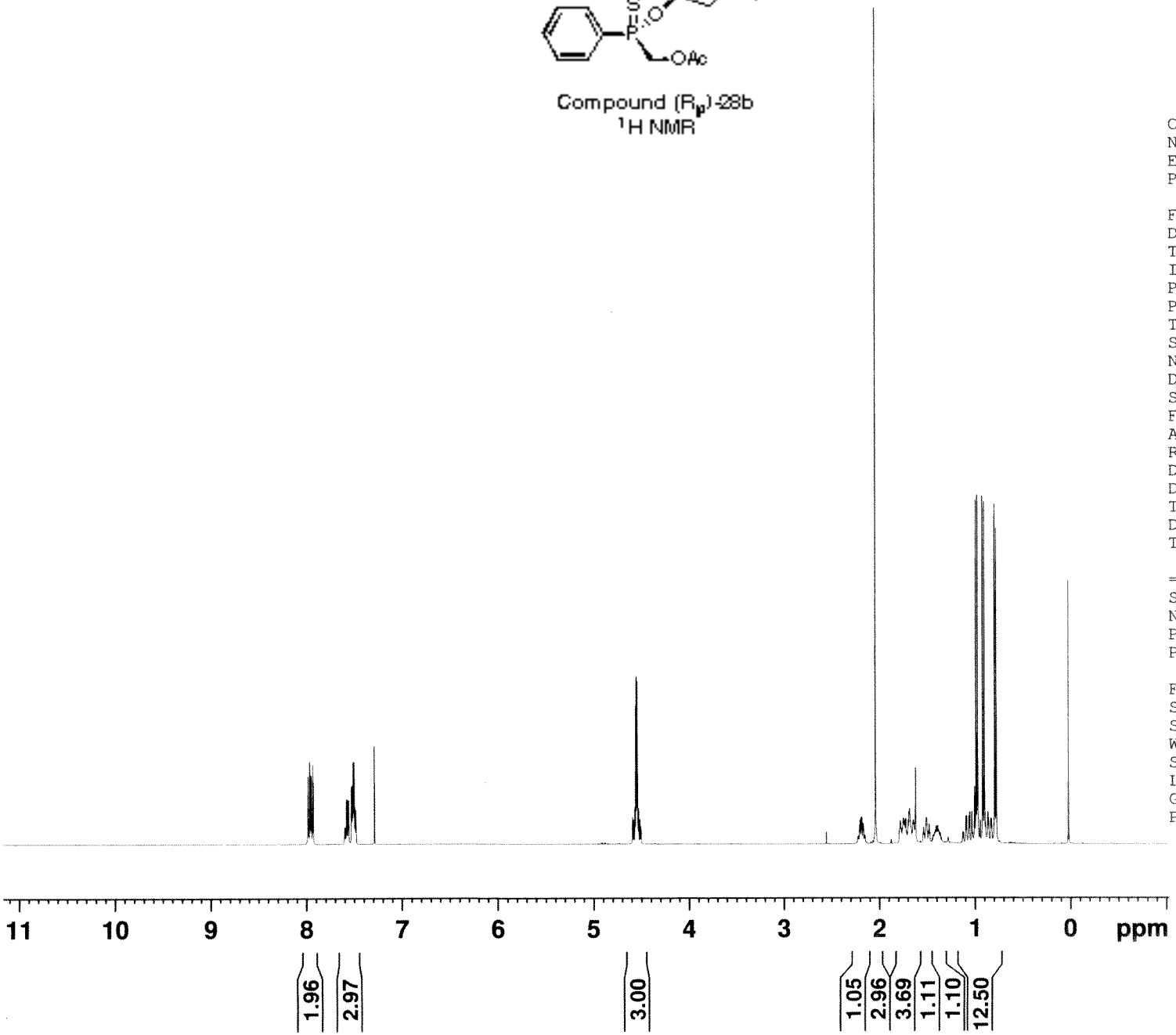
Compound (R_p)-28b
$^{31}P\{^1H\}$ NMR coupled



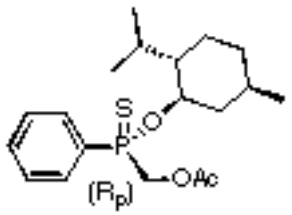

```
Current Data Parameters
NAME        OB 1595 pure
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140723
Time               19.46
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH            64102.563 Hz
FIDRES          0.978127 Hz
AQ              0.5111808 sec
RG                203.57
DW                 7.800 usec
DE                 6.50 usec
TE                294.9 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         161.9674942 MHz
NUC1                  31P
P1                 14.25 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 32768
SF           161.9755930 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



Compound (R_p)-28b
^{1}H NMR

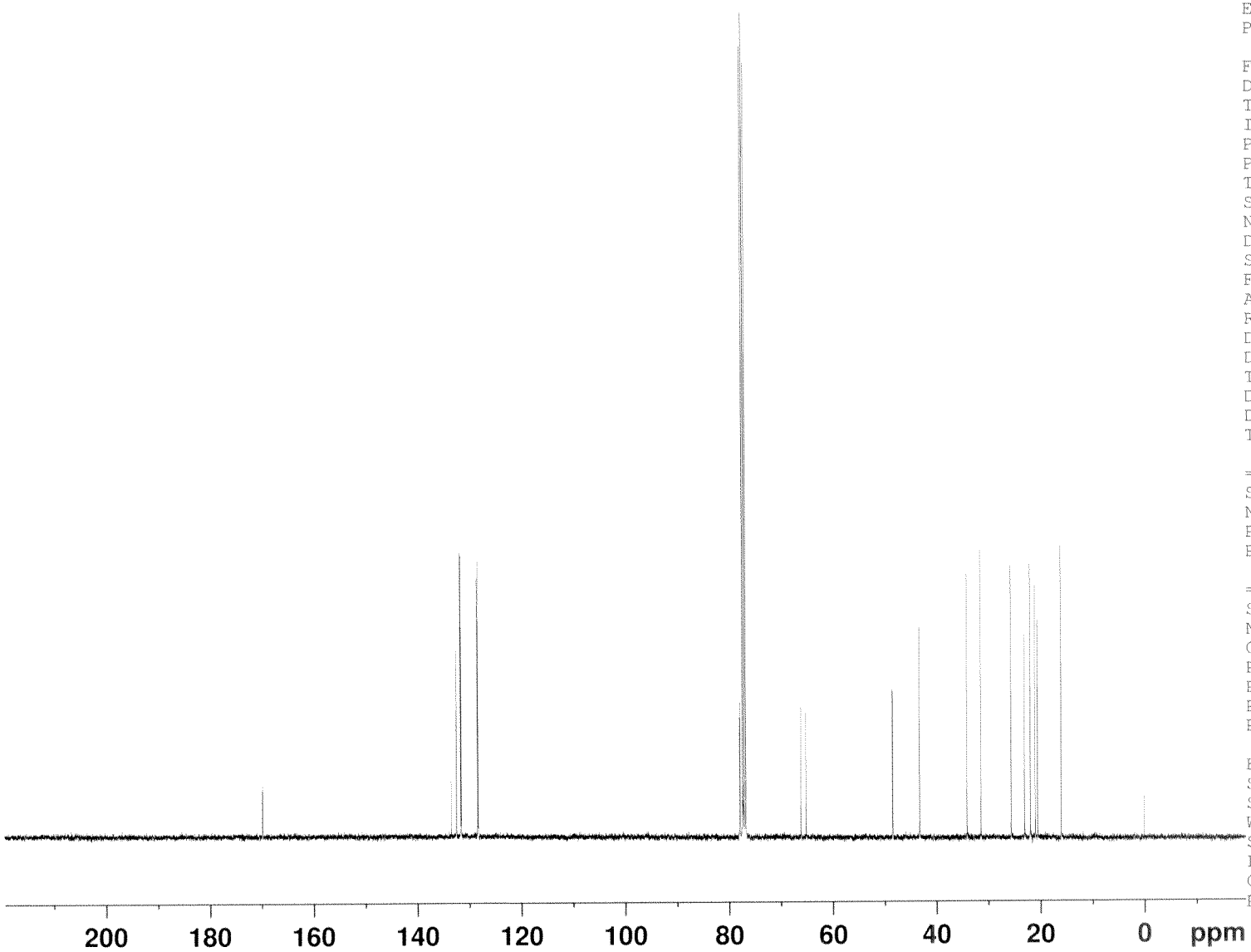



```
Current Data Parameters
NAME        OB 1595 pure
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140723
Time               19.50
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 81.67
DW                62.400 usec
DE                 6.50 usec
TE                294.9 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 10.00 usec
PLW1         25.00300026 W

F2 - Processing parameters
SI                 65536
SF           400.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

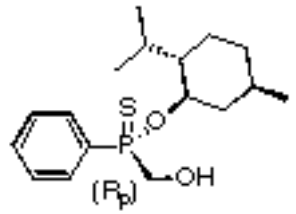


Compound (R_p)-28b

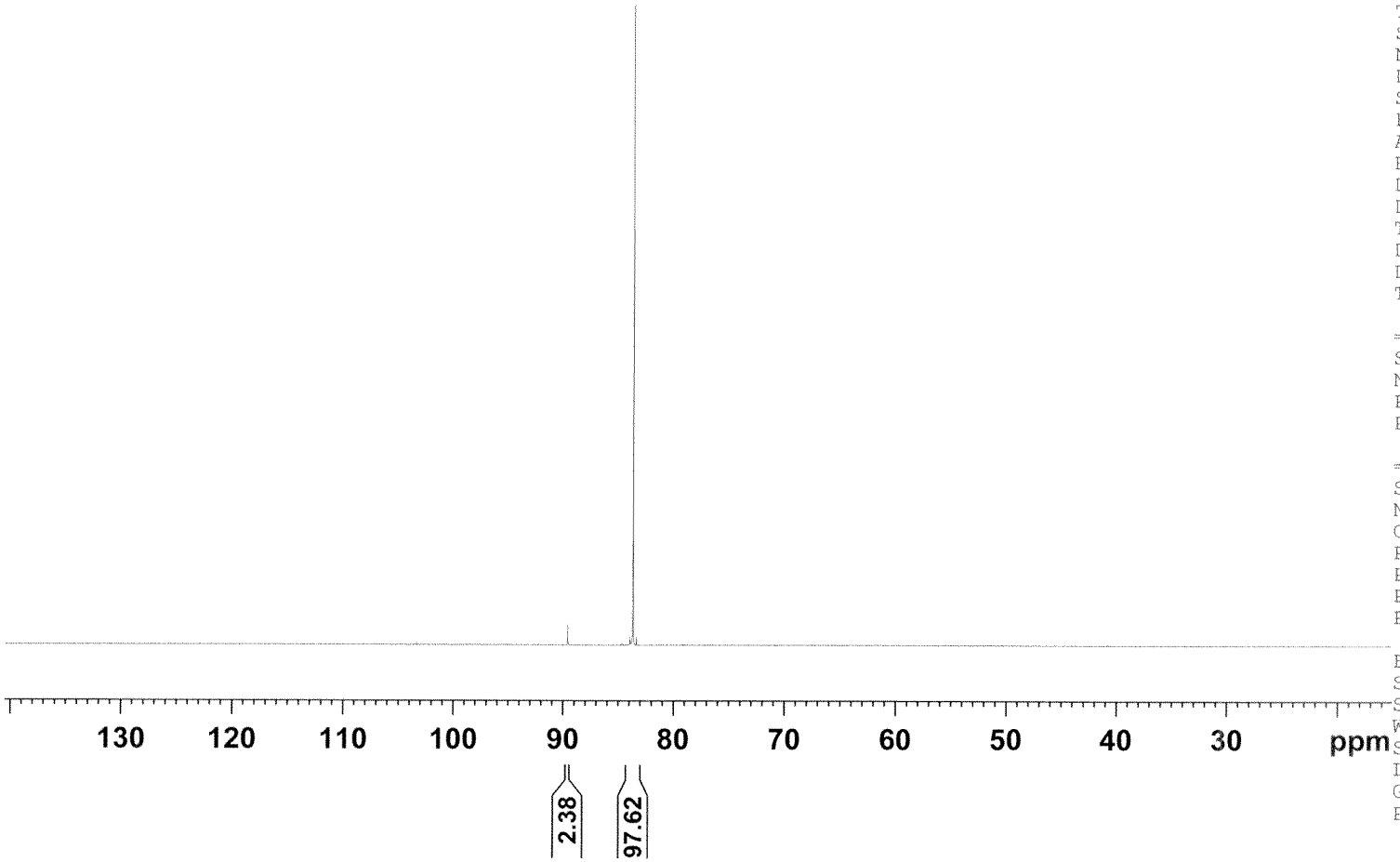
^{13}C NMR




```
Current Data Parameters
NAME        OB 1595 pure
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140723
Time               21.00
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                 1024
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                296.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         45.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



Compound (R_p)-29
$^{31}P\{^1H\}$ NMR decoupled



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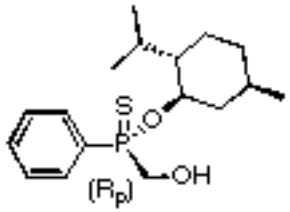
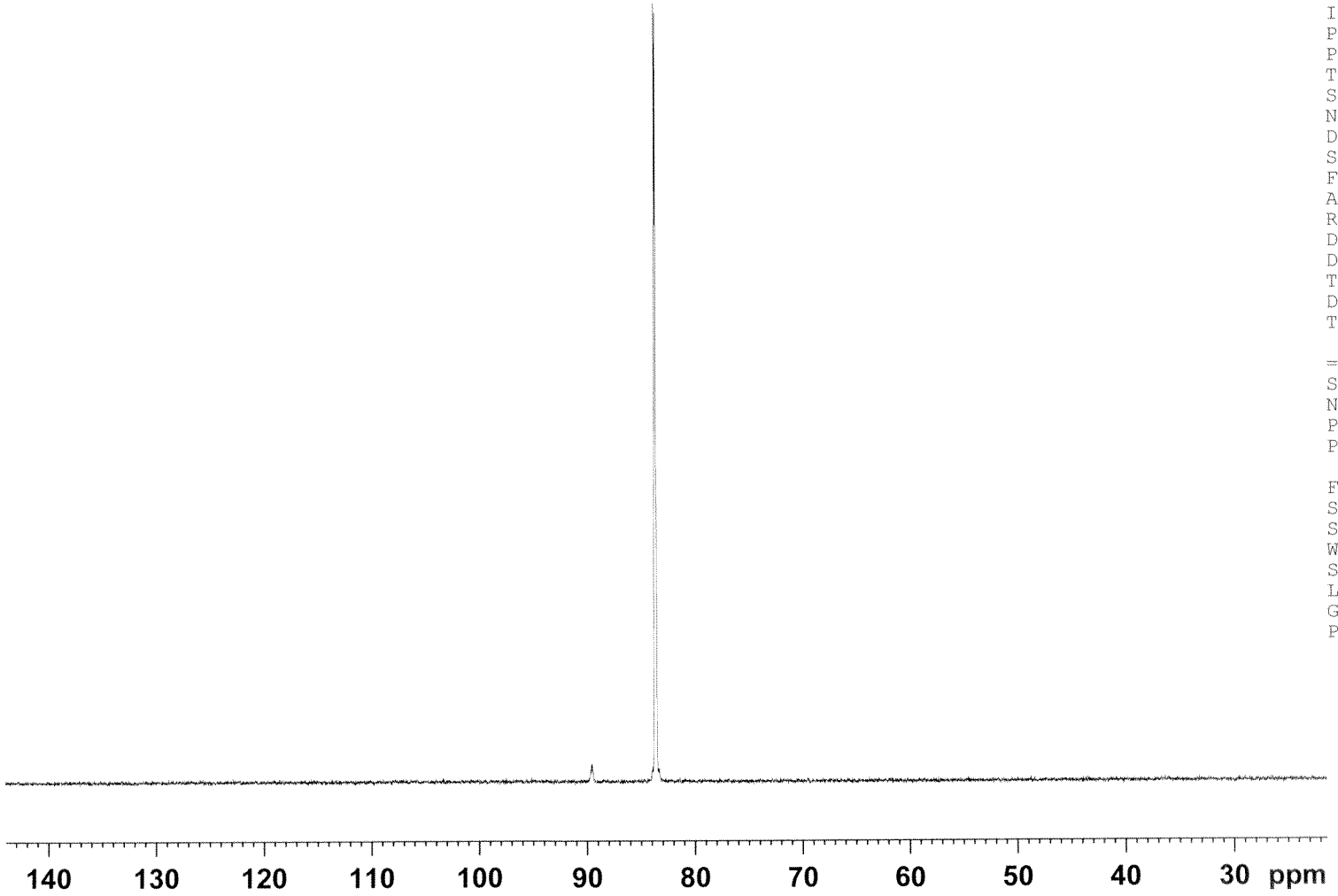
```
Current Data Parameters
NAME                  OB 1601
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20140724
Time                   9.50
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT             Acetone
NS                         16
DS                          4
SWH               64102.563 Hz
FIDRES             0.978127 Hz
AQ                 0.5111808 sec
RG                    203.57
DW                     7.800 usec
DE                     6.50 usec
TE                     295.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1           161.9674942 MHz
NUC1                    31P
P1                    14.25 usec
PLW1           15.00000000 W

======== CHANNEL f2 ========
SFO2           400.1316005 MHz
NUC2                    1H
CPDPRG[2           waltz16
PCPD2                 90.00 usec
PLW2           10.00000000 W
PLW12           0.31604999 W
PLW13           0.25600001 W

F2 - Processing parameters
SI                      32768
SF             161.9755930 MHz
WDW                        EM
SSB         0
LB                     1.00 Hz
GB          0
PC                     1.40
```



Compound (R_p)-29
$^{31}P\{^1H\}$ NMR coupled



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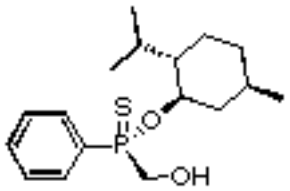
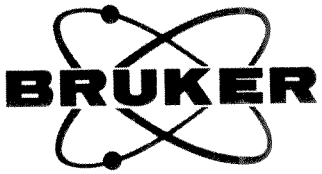
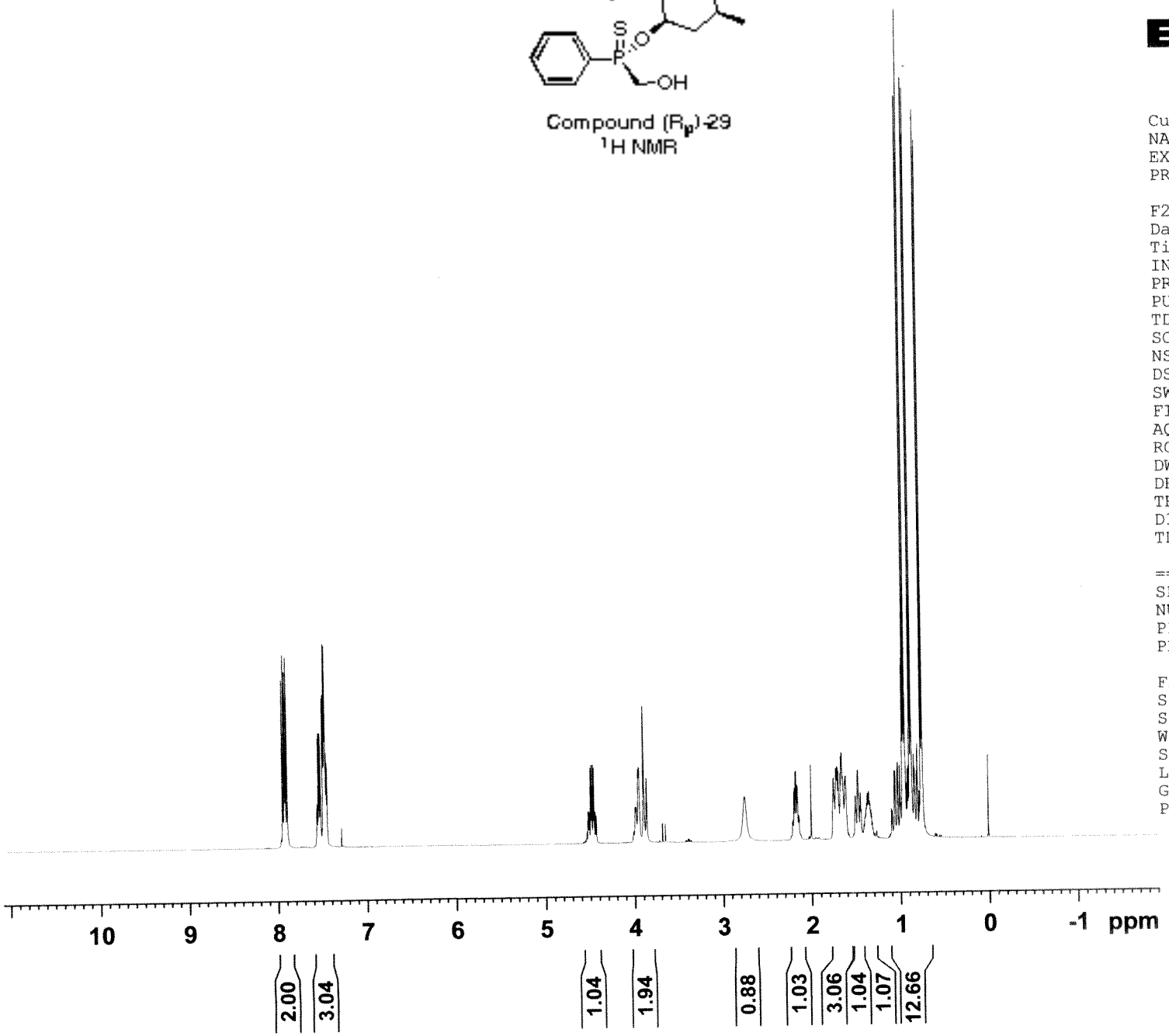
```
Current Data Parameters
NAME                  OB 1601
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20140724
Time                   9.52
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT           Acetone
NS                         32
DS                          4
SWH               64102.563 Hz
FIDRES             0.978127 Hz
AQ                0.5111808 sec
RG                   203.57
DW                    7.800 usec
DE                    6.50 usec
TE                   295.2 K
D1              2.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1           161.9674942 MHz
NUC1                    31P
P1                    14.25 usec
PLW1           15.00000000 W

F2 - Processing parameters
SI                      32768
SF             161.9755930 MHz
WDW                        EM
SSB       0
LB                     1.00 Hz
GB        0
PC                     1.40
```

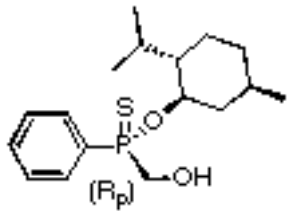


Compound (R_p)-29
^{1}H NMR



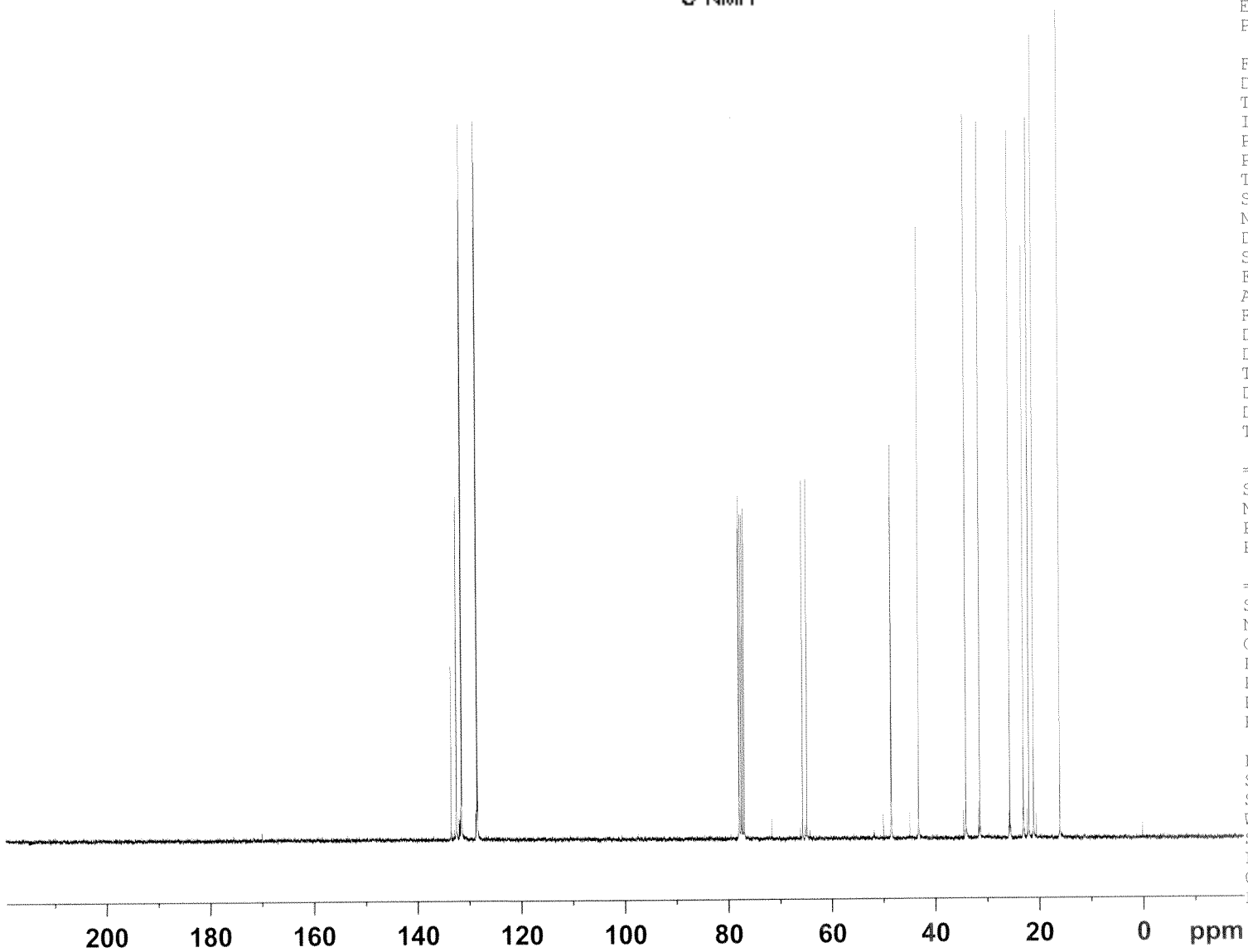

```
Current Data Parameters
NAME          OB1601 pure2
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_             20151124
Time                 17.18
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                   12.96
DW                 62.400 usec
DE                  6.50 usec
TE                  292.2 K
D1              1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                     1H
P1                  10.00 usec
PLW1          25.00300026 W

F2 - Processing parameters
SI                      65536
SF            400.1300000 MHz
WDW                     EM
SSB       0
LB                   0.30 Hz
GB        0
PC                    1.00
```



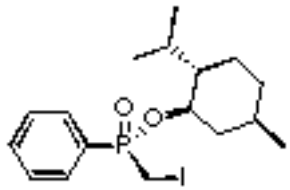


Compound (R_p)-29

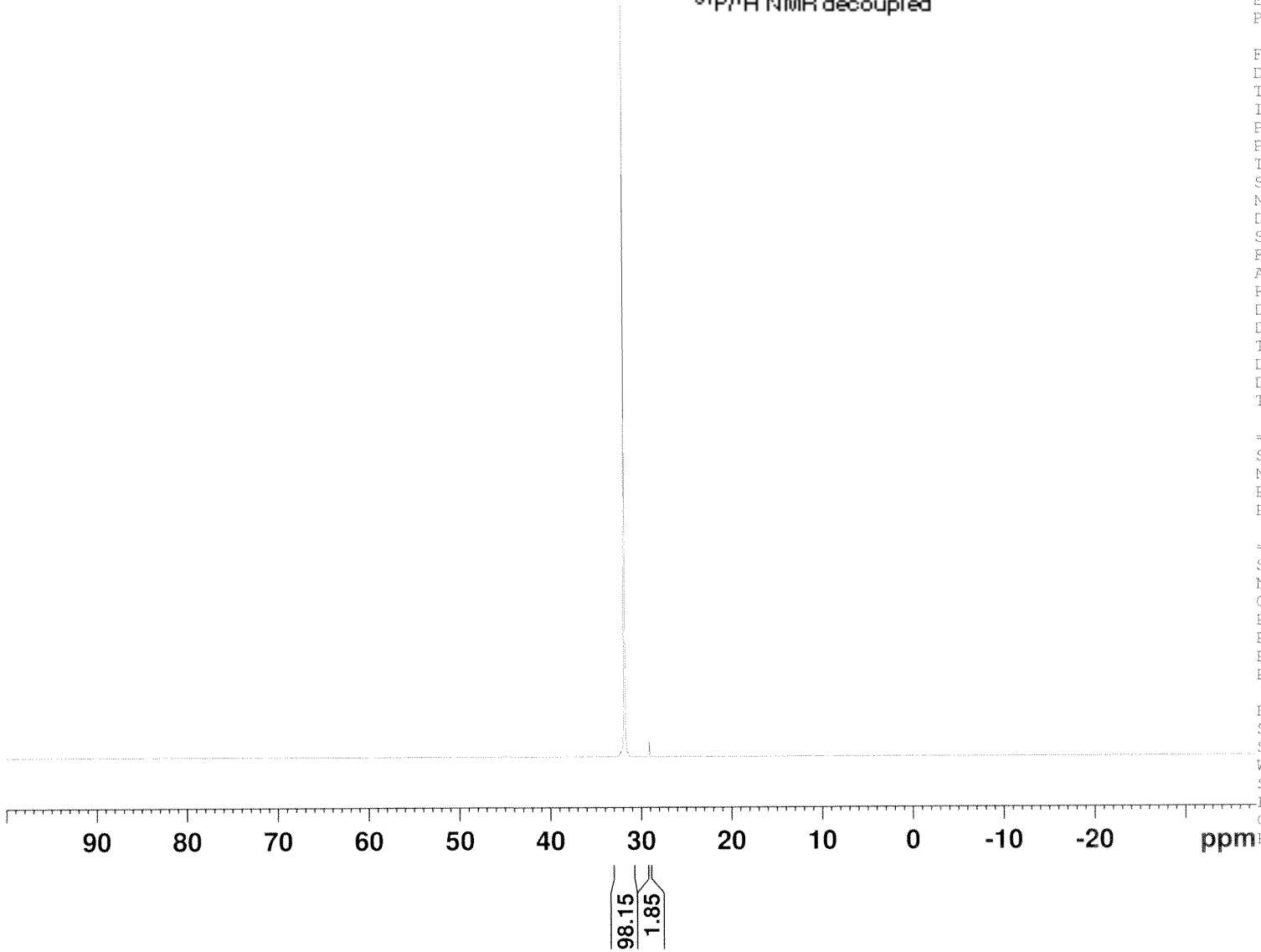
^{13}C NMR



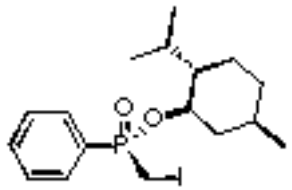

```
Current Data Parameters
NAME          OB1601 pure2
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_             20151124
Time                 17.35
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    242
DS                      4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                293.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         45.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                   32768
SF           100.6127685 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



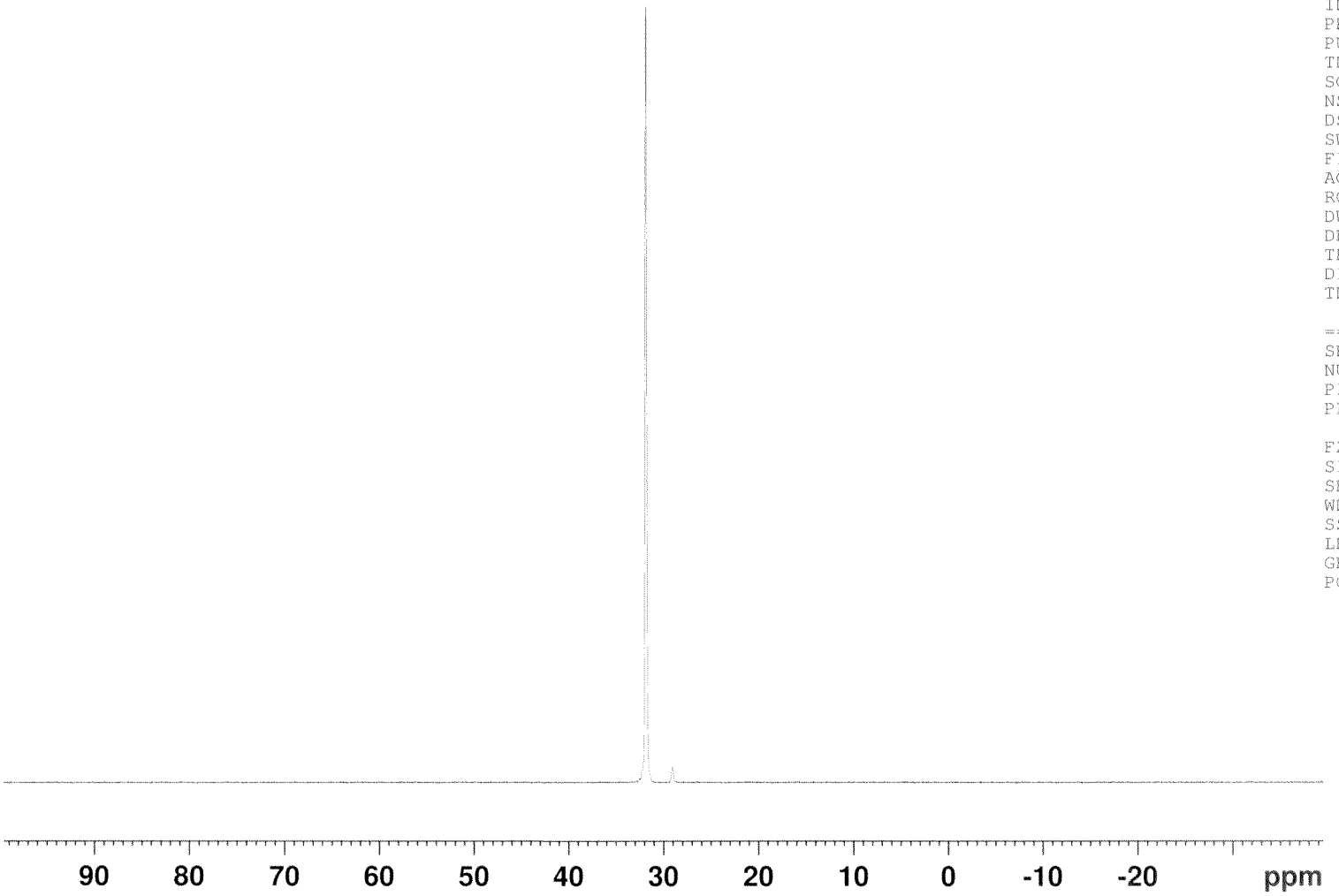
Compound (S_p)-30
$^{31}P/^{1}H$ NMR decoupled



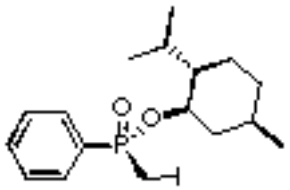

```
Current Data Parameters
NAME      OB 1662 after work-up
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_              20140815
Time                  17.51
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        4
SWH               64102.563 Hz
FIDRES             0.978127 Hz
AQ                0.5111808 sec
RG                   203.57
DW                    7.800 usec
DE                    6.50 usec
TE                   295.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1           161.9674942 MHz
NUC1                   31P
P1                   14.25 usec
PLW1           15.00000000 W

======== CHANNEL f2 ========
SFO2           400.1316005 MHz
NUC2                   1H
CPDPRG[2            waltz16
PCPD2                90.00 usec
PLW2           10.00000000 W
PLW12           0.31604999 W
PLW13           0.25600001 W

F2 - Processing parameters
SI                    32768
SF             161.9755930 MHz
WDW                      EM
SSB       0
LB                    1.00 Hz
GB        0
PC                    1.40
```



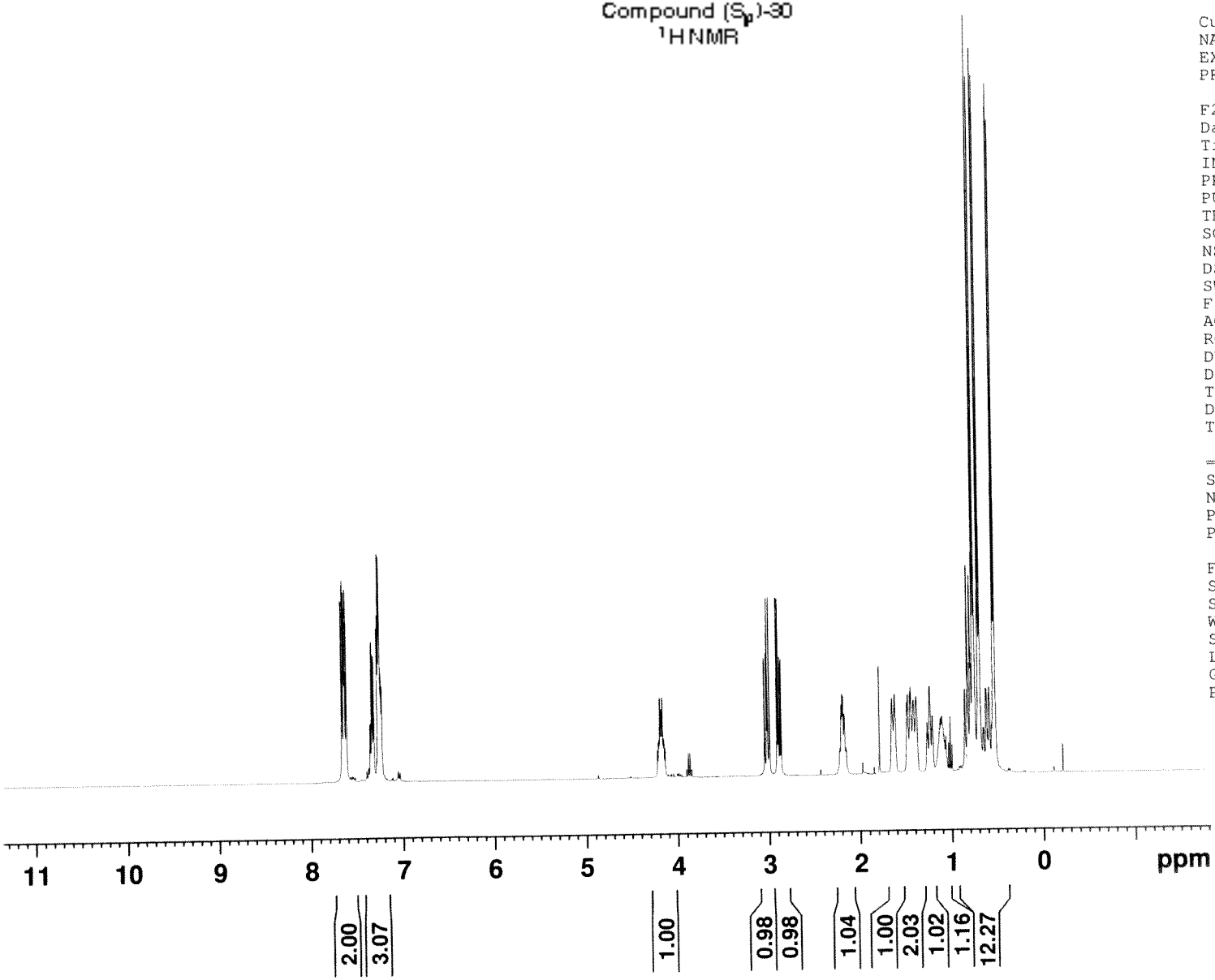
Compound (S_p)-30
$^{31}P/^{1}H$ NMR coupled



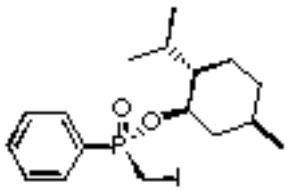

```
Current Data Parameters
NAME      OB 1662 after work-up
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_            20140815
Time                17.53
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     4
SWH             64102.563 Hz
FIDRES           0.978127 Hz
AQ              0.5111808 sec
RG                 203.57
DW                  7.800 usec
DE                  6.50 usec
TE                 294.9 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          161.9674942 MHz
NUC1                  31P
P1                  14.25 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                 32768
SF            161.9755930 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



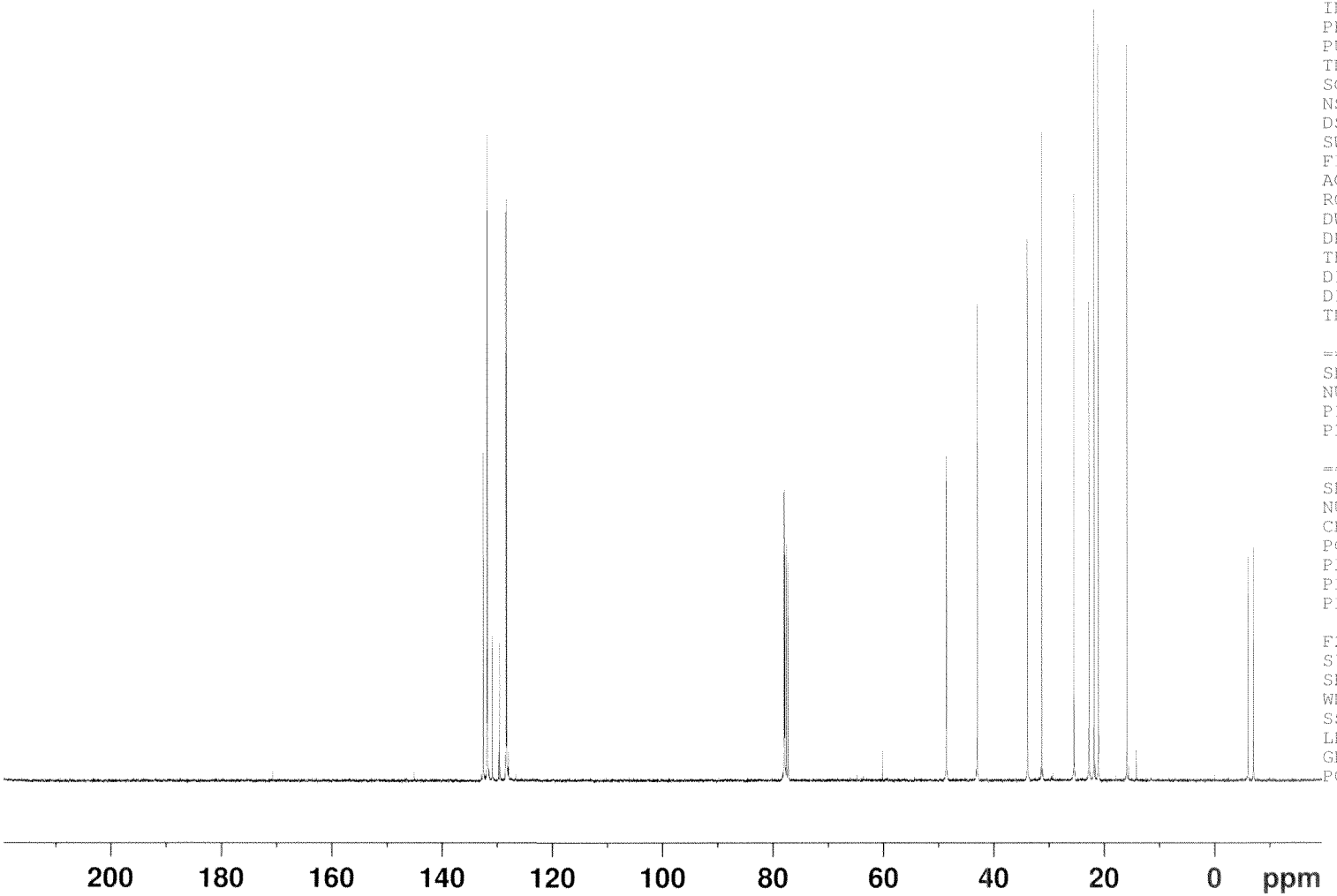
Compound (S_p)-30
^{1}H NMR




```
Current Data Parameters
NAME      OB 1662 after work-up
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_               20140815
Time                   17.57
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        9
DS                        2
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894465 sec
RG                      8.12
DW                    62.400 usec
DE                     6.50 usec
TE                    295.1 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            400.1324710 MHz
NUC1                      1H
P1                    10.00 usec
PLW1           25.00300026 W

F2 - Processing parameters
SI                        65536
SF              400.1300000 MHz
WDW                       EM
SSB          0
LB                     0.30 Hz
GB           0
PC                     1.00
```



Compound (S_p)-30
^{13}C NMR

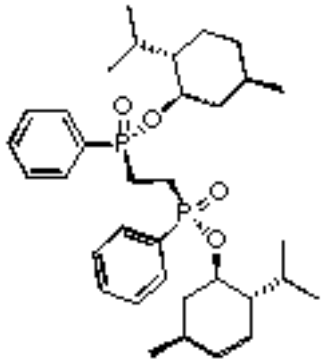



```
Current Data Parameters
NAME      OB 1662 after work-up
EXPNO                      4
PROCNO                     1

F2 - Acquisition Parameters
Date_            20140815
Time                 18.13
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    222
DS                      4
SWH            24038.461 Hz
FIDRES          0.366798 Hz
AQ             1.3631488 sec
RG                 203.57
DW                 20.800 usec
DE                  6.50 usec
TE                 296.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         45.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                    32768
SF           100.6127685 MHz
WDW                     EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```

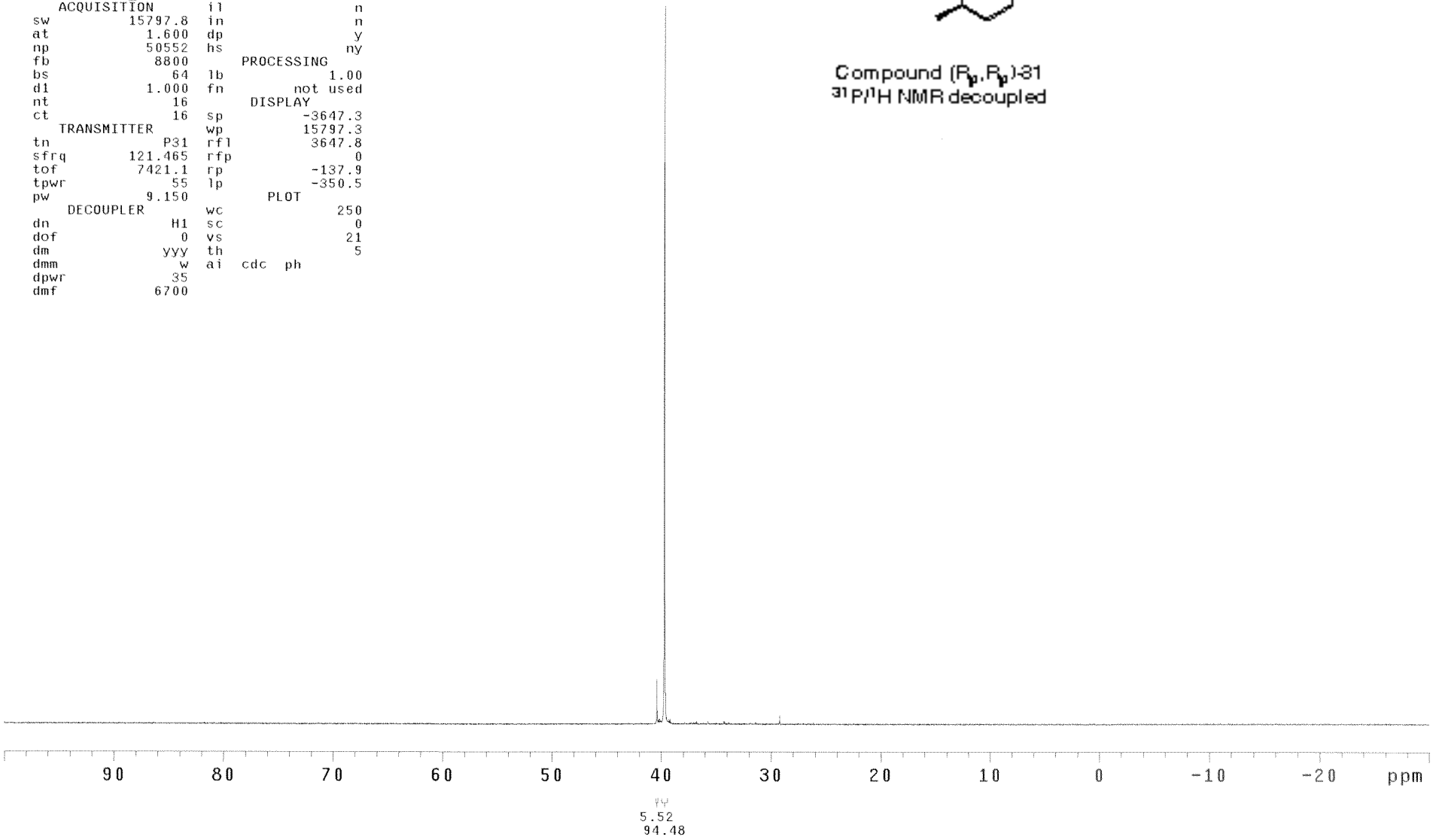
OB 927
pad=10 run with findz0 before acquisitio
n

exp1 Phosphorus

SAMPLE		SPECIAL	
date	Jan 31 2013	temp	not used
solvent	cdcl3	gain	14
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2013.01.20/s_2013~		pw90	18.300
0131_14/data/cdcl3~		alfa	10.000
_03.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	-137.9
tpwr	55	lp	-350.5
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	21
dm	yyy	th	5
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



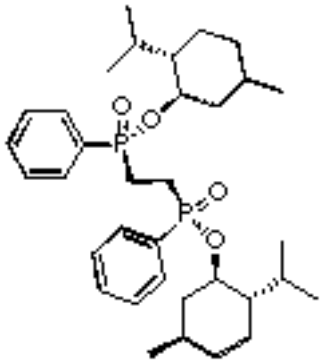
Compound (R_p,R_p)-31
$^{31}P\{^1H\}$ NMR decoupled



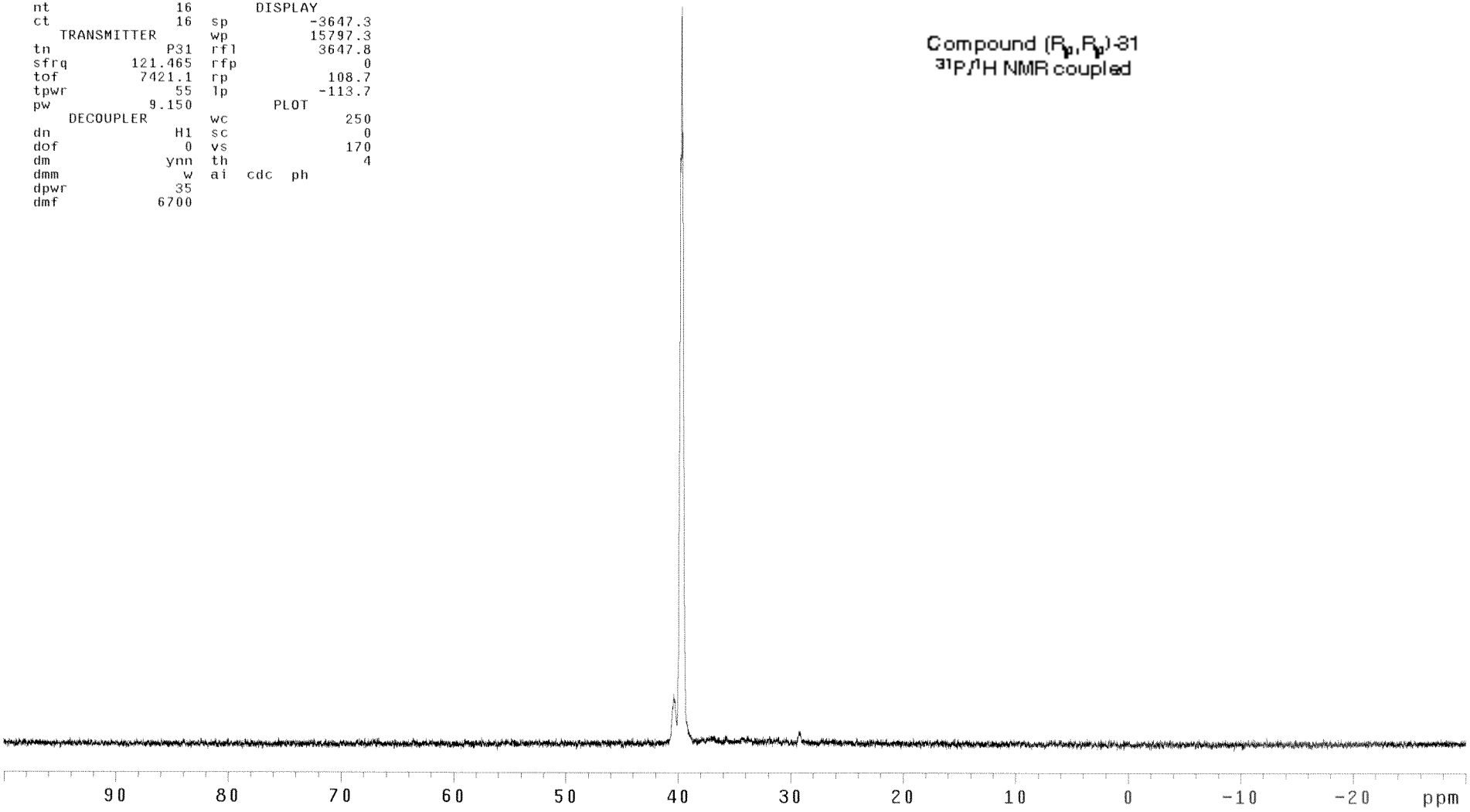
OB 927

exp1 Phosphorus

SAMPLE		SPECIAL	
date	Jan 31 2013	temp	not used
solvent	cdcl3	gain	14
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2013.01.20/s_2013~		pw90	18.300
0131_14/data/cdcl3~		alfa	10.000
_06.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	16	DISPLAY	
ct	16	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	108.7
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	170
dm	ynn	th	4
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



Compound (R_p,R_p)-31
$^{31}P\{^1H\}$ NMR coupled

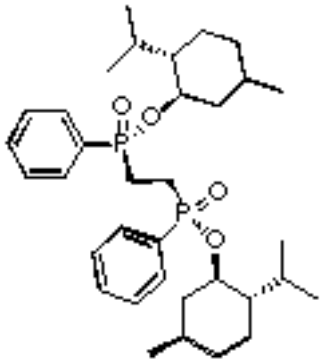


OB 927
pad=10 run with findz0 before acquisitio
n

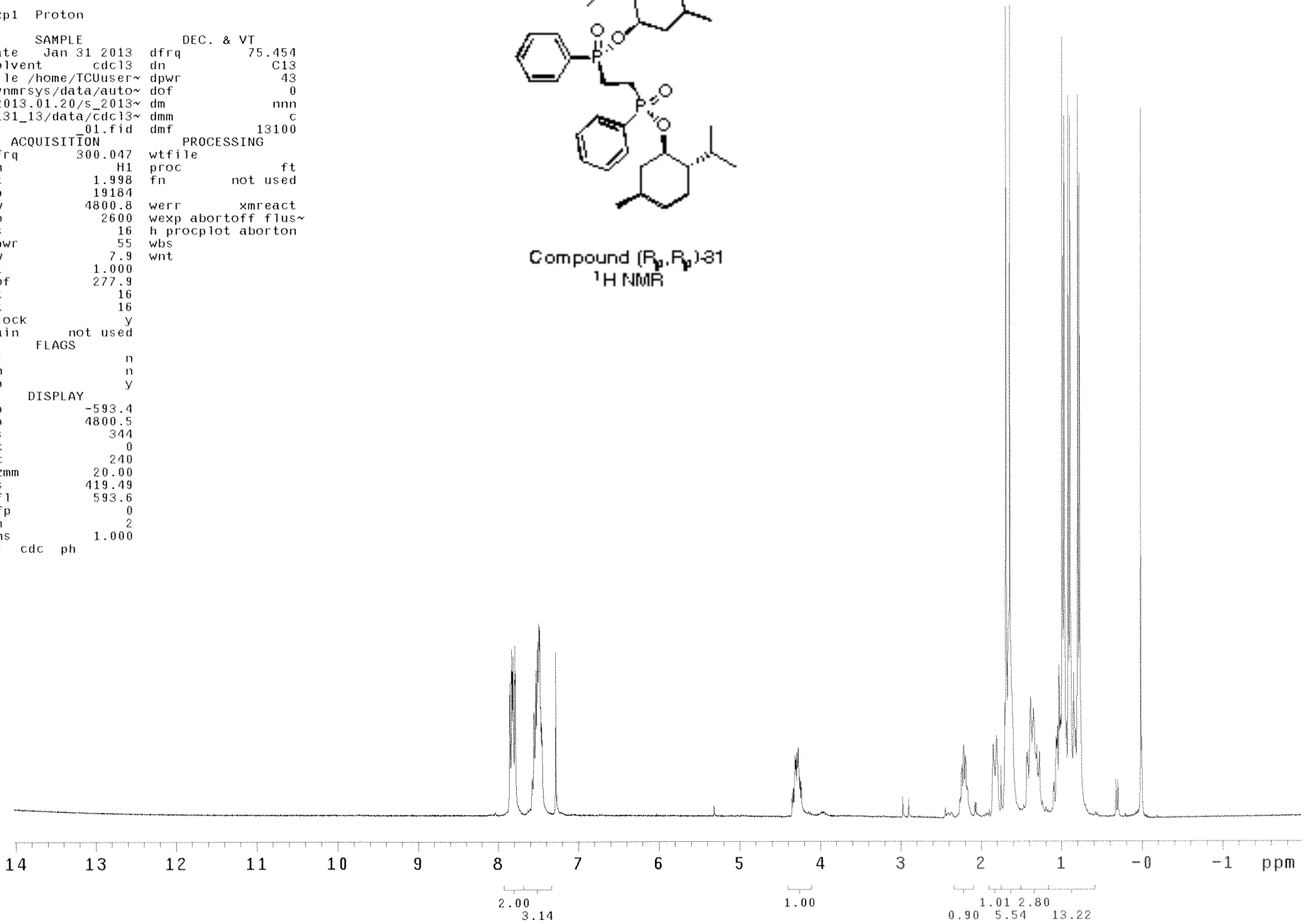
exp1 Proton

SAMPLE		DEC. & VT	
date	Jan 31 2013	dfrq	75.454
solvent	cdcl3	dn	C13
file	/home/TCUuser~	dpwr	43
	/vnmrsys/data/auto~	dof	0
	_2013.01.20/s_2013~	dm	nnn
	0131_13/data/cdcl3~	dmm	c
	_01.fid	dmf	13100
ACQUISITION		PROCESSING	
sfrq	300.047	wtfile	
tn	H1	proc	ft
at	1.998	fn	not used
np	19184		
sw	4800.8	werr	xmreact
fb	2600	wexp	abortoff flus~
bs	16	h procplot	aborton
tpwr	55	wbs	
pw	7.9	wnt	
d1	1.000		
tof	277.9		
nt	16		
ct	16		
alock	y		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-593.4		
wp	4800.5		
vs	344		
sc	0		
wc	240		
hzmm	20.00		
is	419.49		
rfl	593.6		
rfp	0		
th	2		
ins	1.000		

ai cdc ph



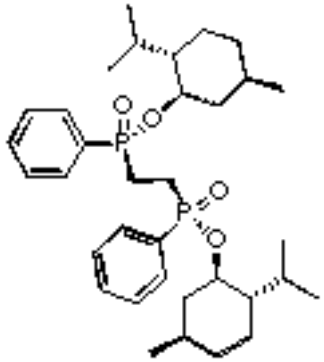
Compound (R_p,R_p)-31
^{1}H NMR



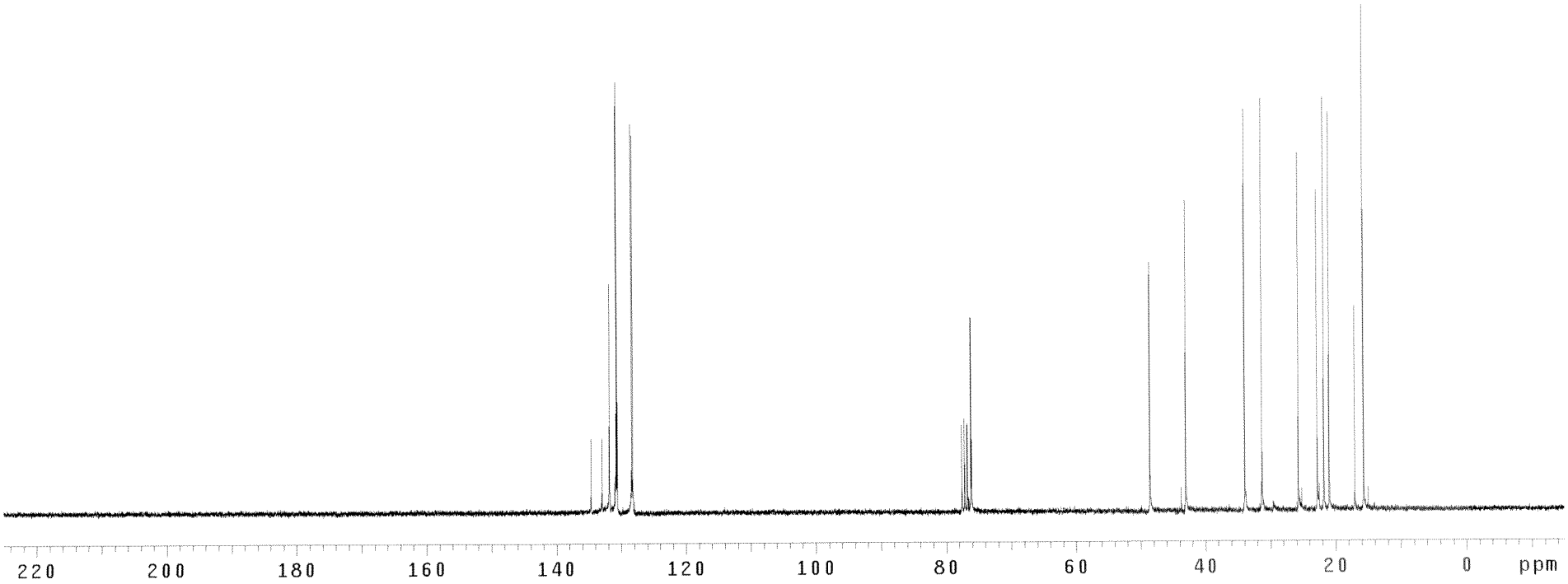
OB 927

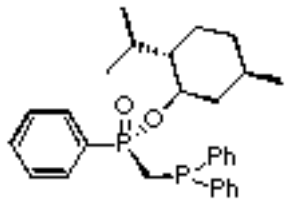
exp1 Carbon

SAMPLE		SPECIAL	
date	Jan 31 2013	temp	not used
solvent	cdcl3	gain	20
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2013.01.20/s_2013~		pw90	18.500
0131_14/data/cdcl3~		alfa	10.000
_07.fid		FLAGS	
ACQUISITION		il	n
sw	18115.9	in	n
at	1.301	dp	y
np	47120	hs	nn
fb	10000	PROCESSING	
bs	64	lb	0.50
d1	2.000	fn	not used
nt	800	DISPLAY	
ct	800	sp	-1135.5
TRANSMITTER		wp	18115.4
tn	C13	rfl	1136.1
sfrq	75.454	rfp	0
tof	766.0	rp	23.6
tpwr	58	lp	-181.3
pw	9.250	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	228
dm	yyy	th	6
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		

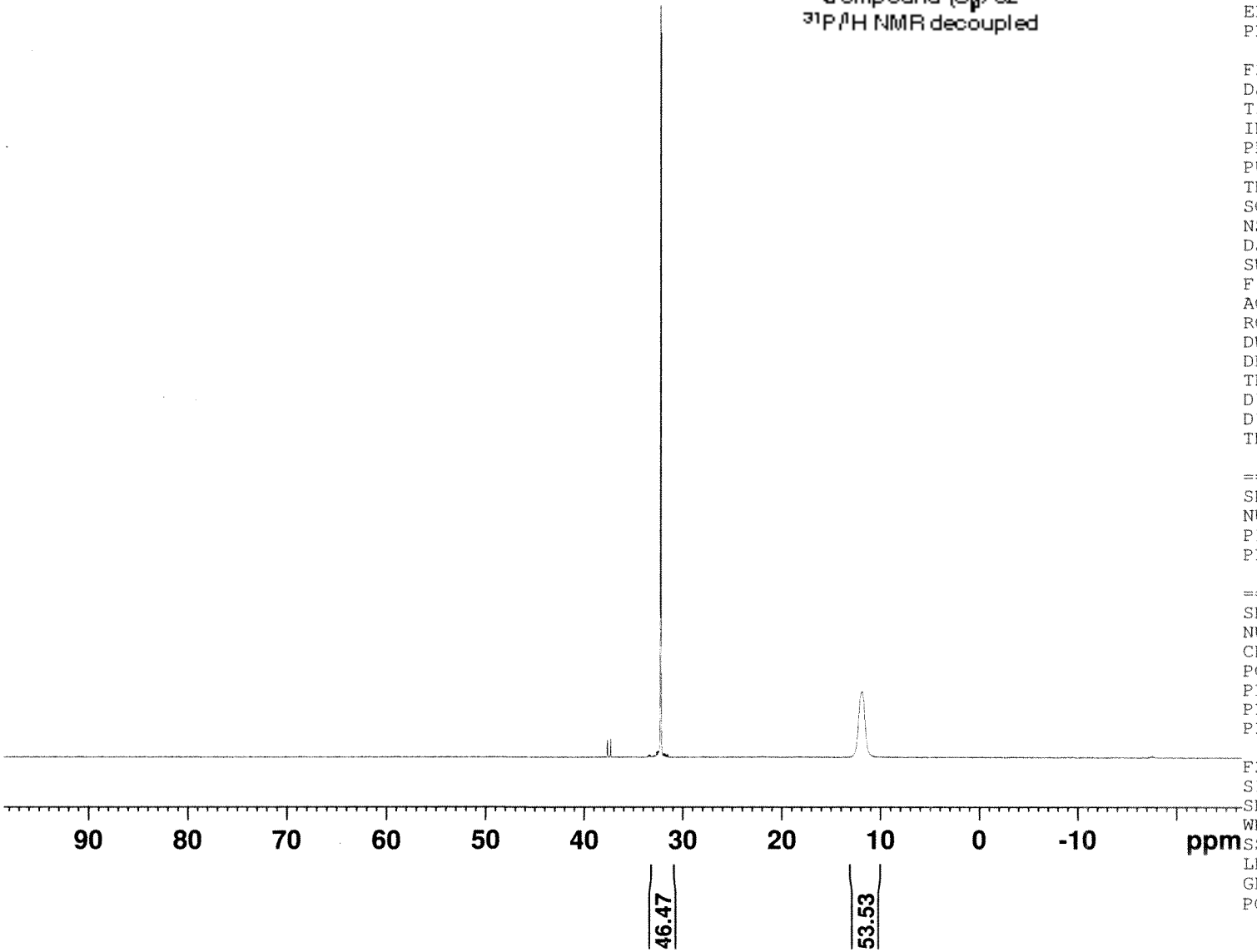


Compound (R_p,R_p)-31
^{13}C NMR

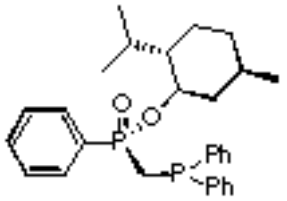




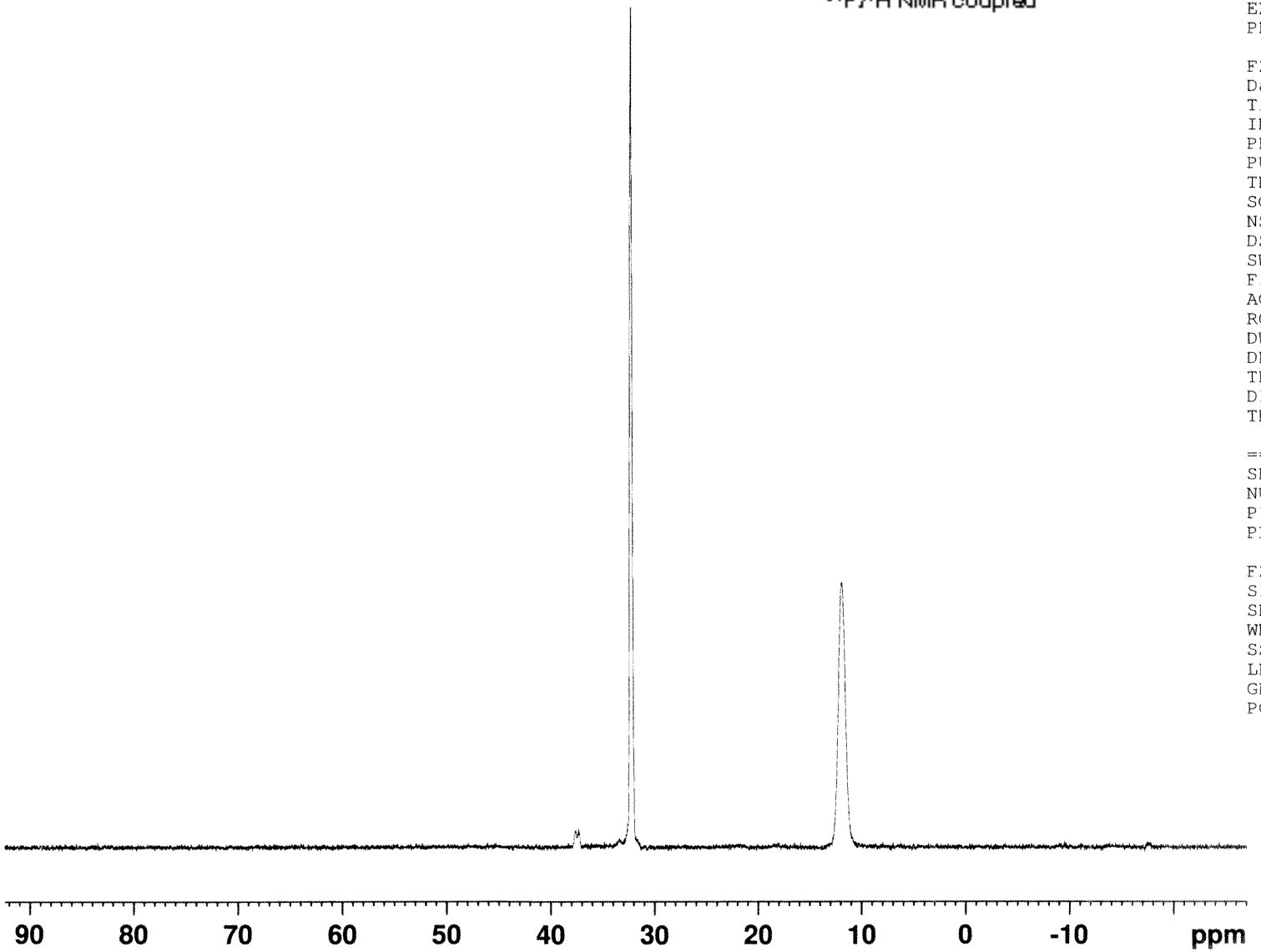
Compound (S_p)-32
$^{31}P\{^1H\}$ NMR decoupled



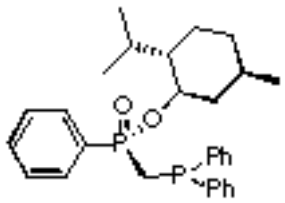

```
Current Data Parameters
NAME      OB 1719f2 pure
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140919
Time               18.02
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                 7.800 usec
DE                 6.50 usec
TE                295.7 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         161.9674942 MHz
NUC1                 31P
P1                14.25 usec
PLW1         15.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                 1H
CPDPRG[2          waltz16
PCPD2             90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                   32768
SF           161.9755930 MHz
WDW                     EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



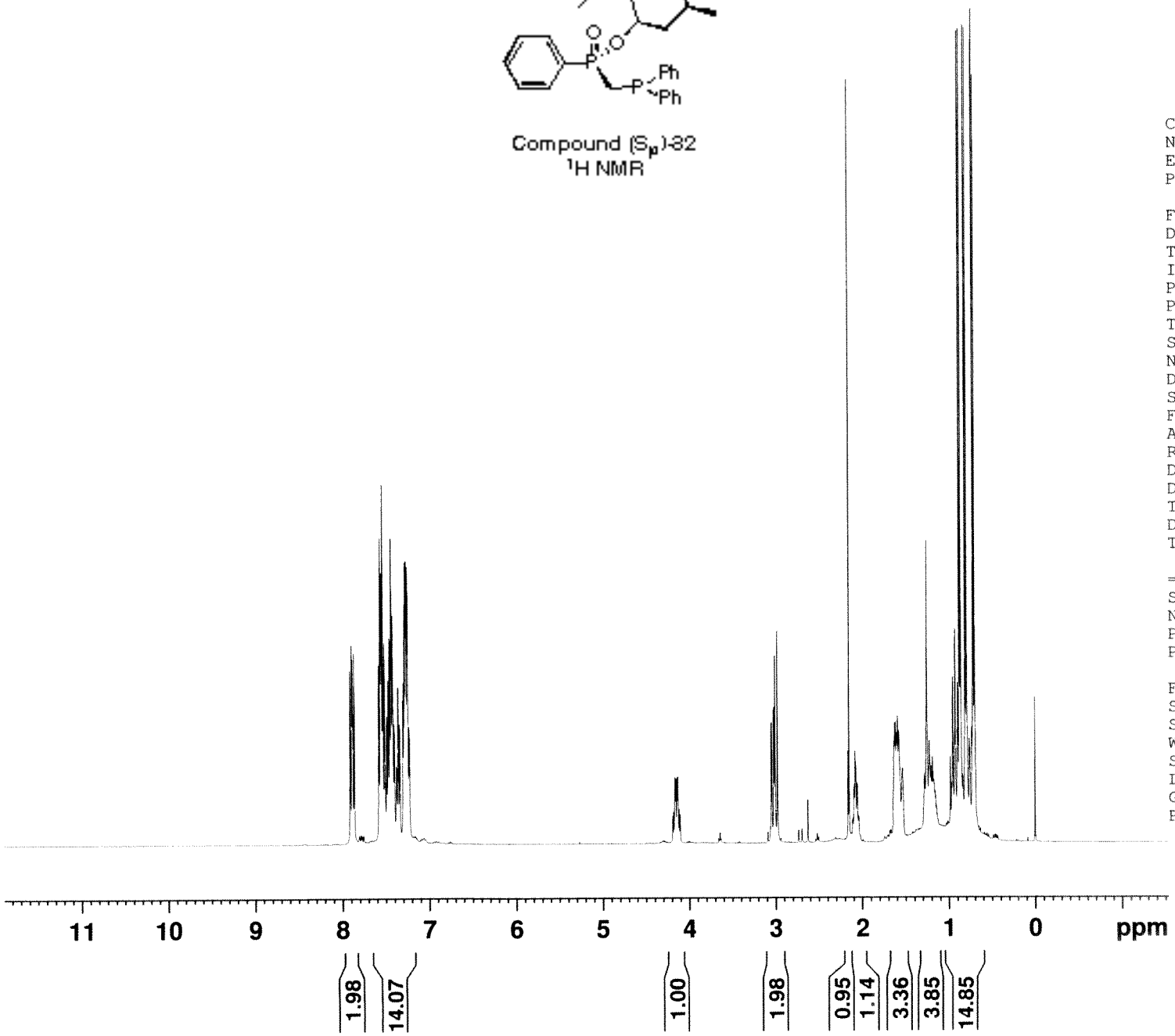
Compound (S_p)-32
$^{31}P\{^1H\}$ NMR coupled



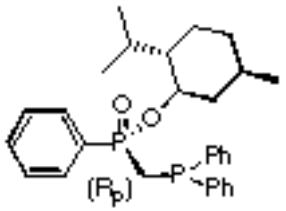

```
Current Data Parameters
NAME      OB 1719f2 pure
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140919
Time               18.04
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE               295.2 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1        15.00000000 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



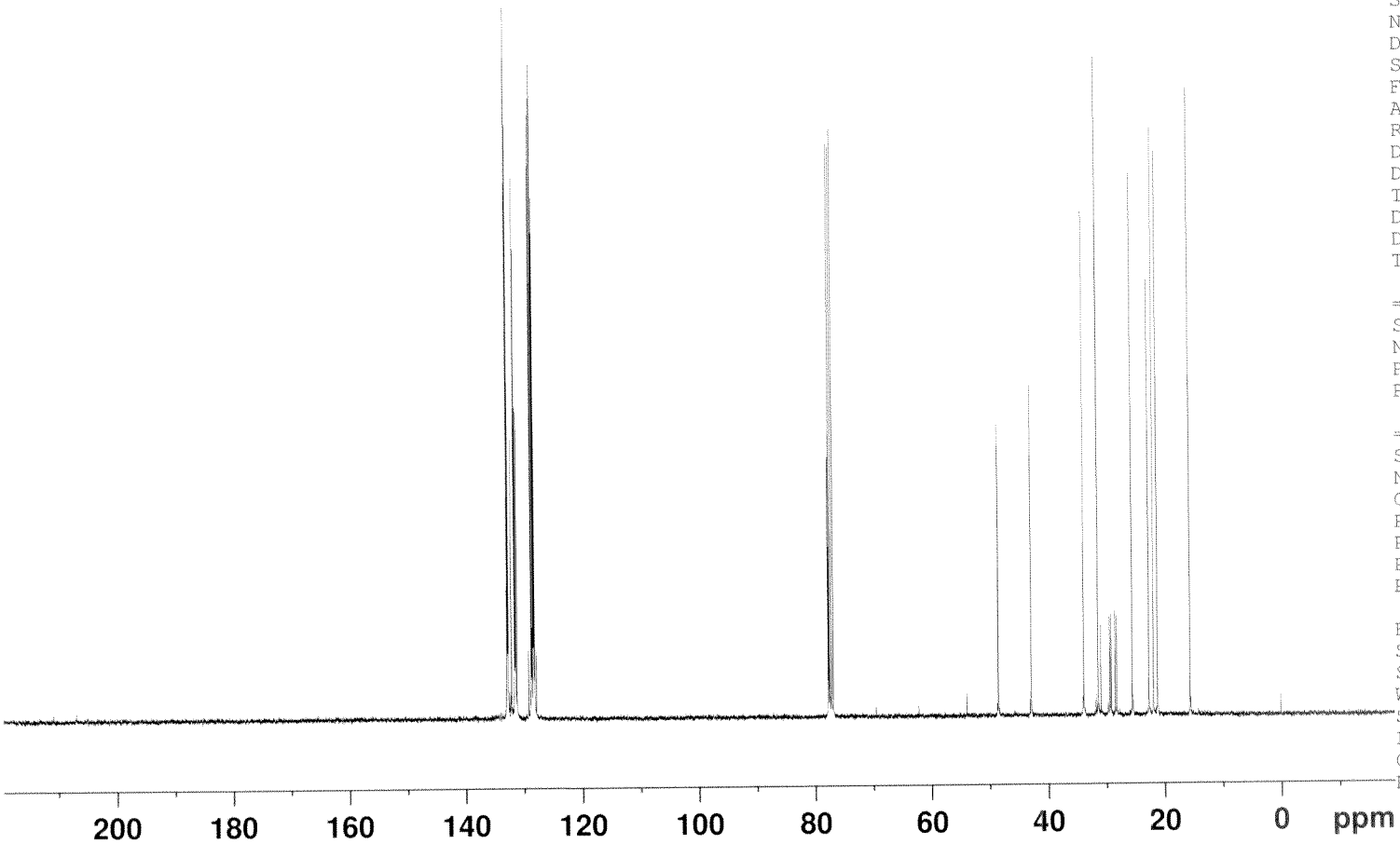
Compound (S_p)-32
^{1}H NMR



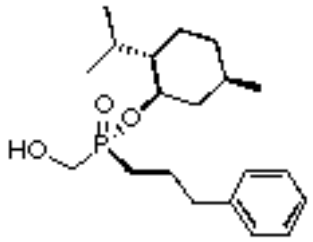

```
Current Data Parameters
NAME      OB 1719f2 pure
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140919
Time               18.07
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    12
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 16.39
DW                62.400 usec
DE                 6.50 usec
TE                295.2 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                 10.00 usec
PLW1         25.00300026 W

F2 - Processing parameters
SI                 65536
SF           400.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



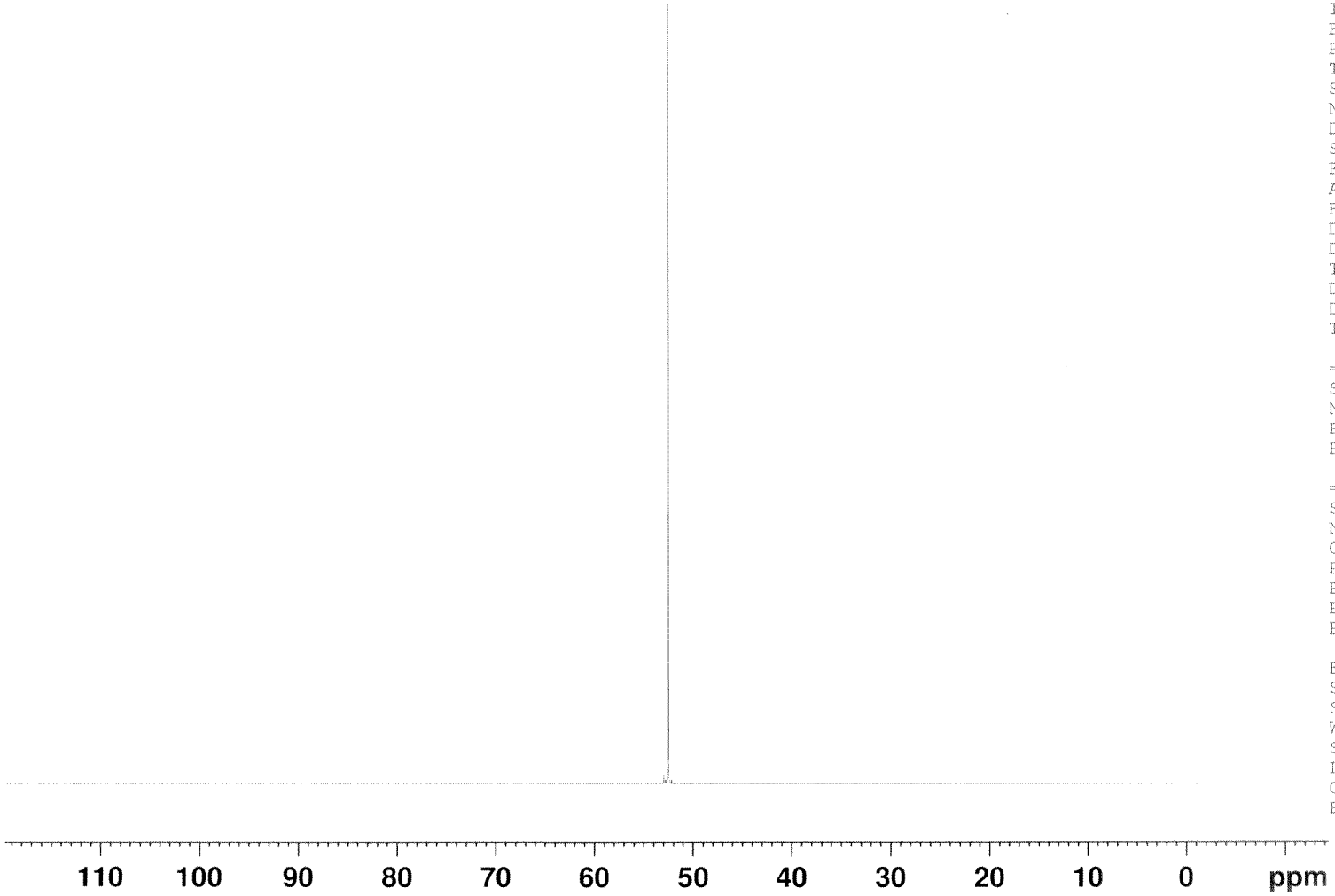
Compound (S_p)-32
^{13}C NMR



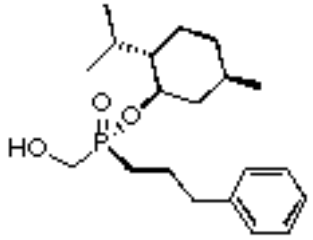

```
Current Data Parameters
NAME      OB 1719f2 pure
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140919
Time               18.56
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD              65536
SOLVENT           CDCl3
NS                  817
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                296.2 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1         45.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                 32768
SF          100.6127685 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



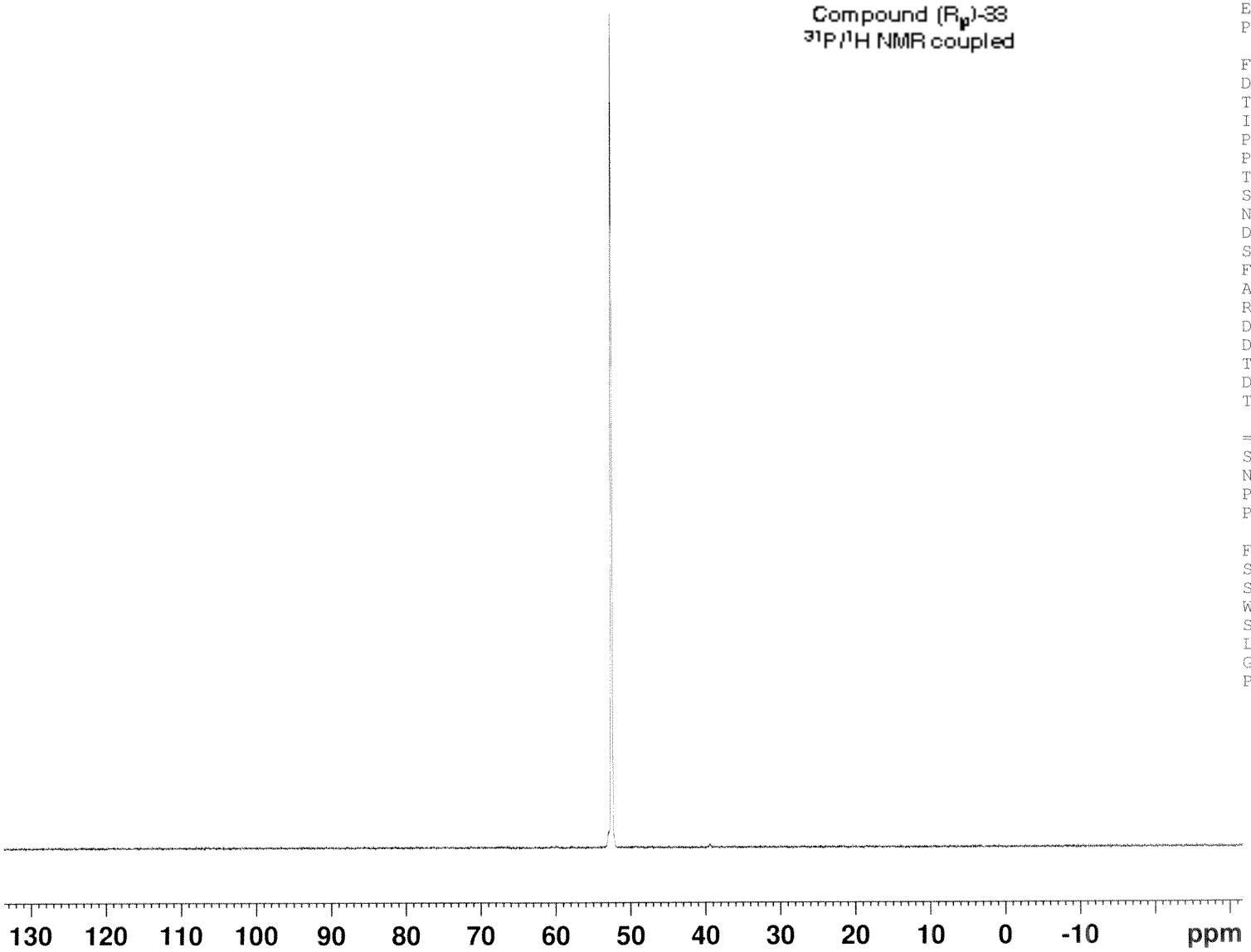
Compound (R_p)-33
$^{31}P/^{1}H$ NMR decoupled



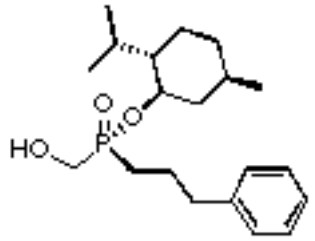

```
Current Data Parameters
NAME      OB 2092 after column
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150626
Time              17.17
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE                294.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

========== CHANNEL f1 =========
SFO1        161.9674942 MHz
NUC1               31P
P1               14.25 usec
PLW1        15.00000000 W

========== CHANNEL f2 =========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



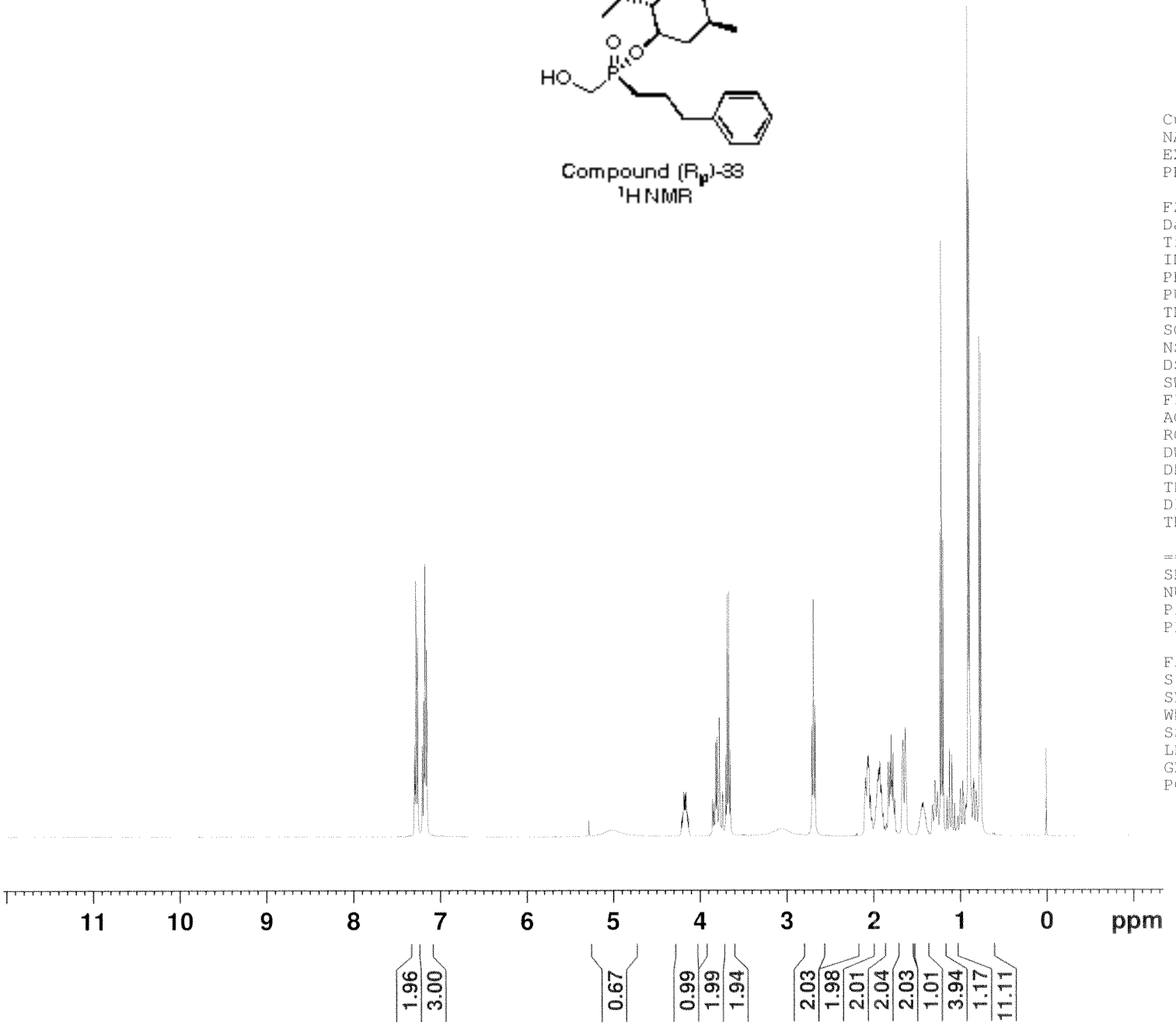
Compound (R_p)-33
$^{31}P/^{1}H$ NMR coupled



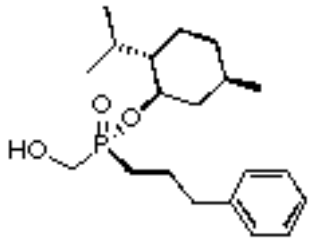

```
Current Data Parameters
NAME      OB 2092 after column
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150626
Time              17.19
INSTRUM           spect
PROBHD    5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE                294.1 K
D1           2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1               31P
P1               14.25 usec
PLW1        15.00000000 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



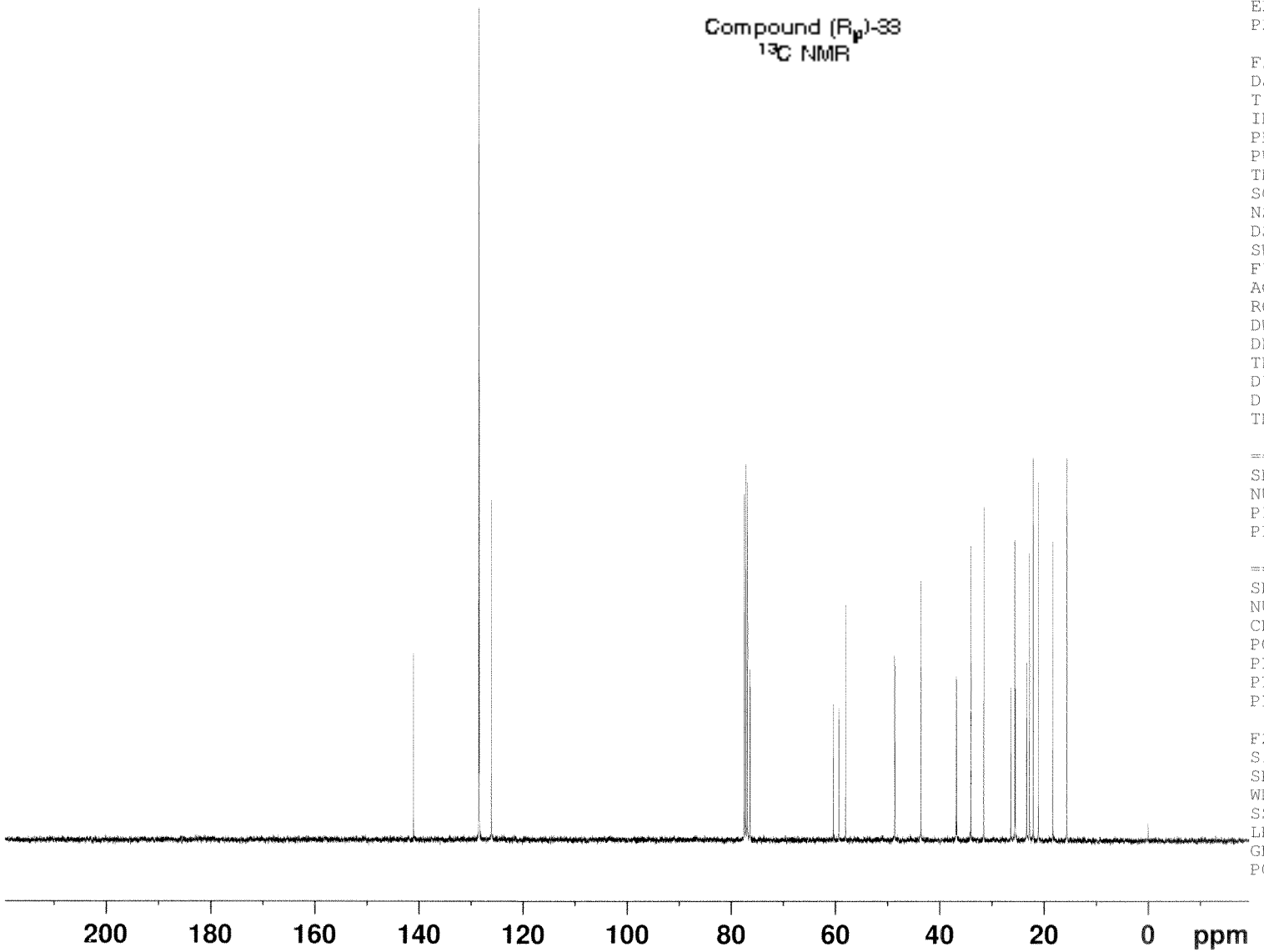
Compound (R_p)-33
^{1}H NMR



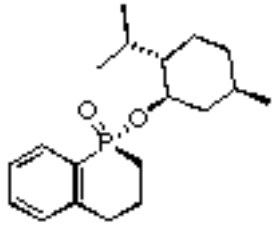

```
Current Data Parameters
NAME      OB 2092 after column
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150626
Time              17.20
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD                65536
SOLVENT           CDCl3
NS                   8
DS                   2
SWH            8012.820 Hz
FIDRES        0.122266 Hz
AQ            4.0894465 sec
RG               13.94
DW               62.400 usec
DE                6.50 usec
TE                294.0 K
D1           1.00000000 sec
TD0                  1

========== CHANNEL f1 ==========
SFO1         400.1324710 MHz
NUC1                1H
P1                10.00 usec
PLW1         25.00300026 W

F2 - Processing parameters
SI                65536
SF           400.1300000 MHz
WDW                 EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



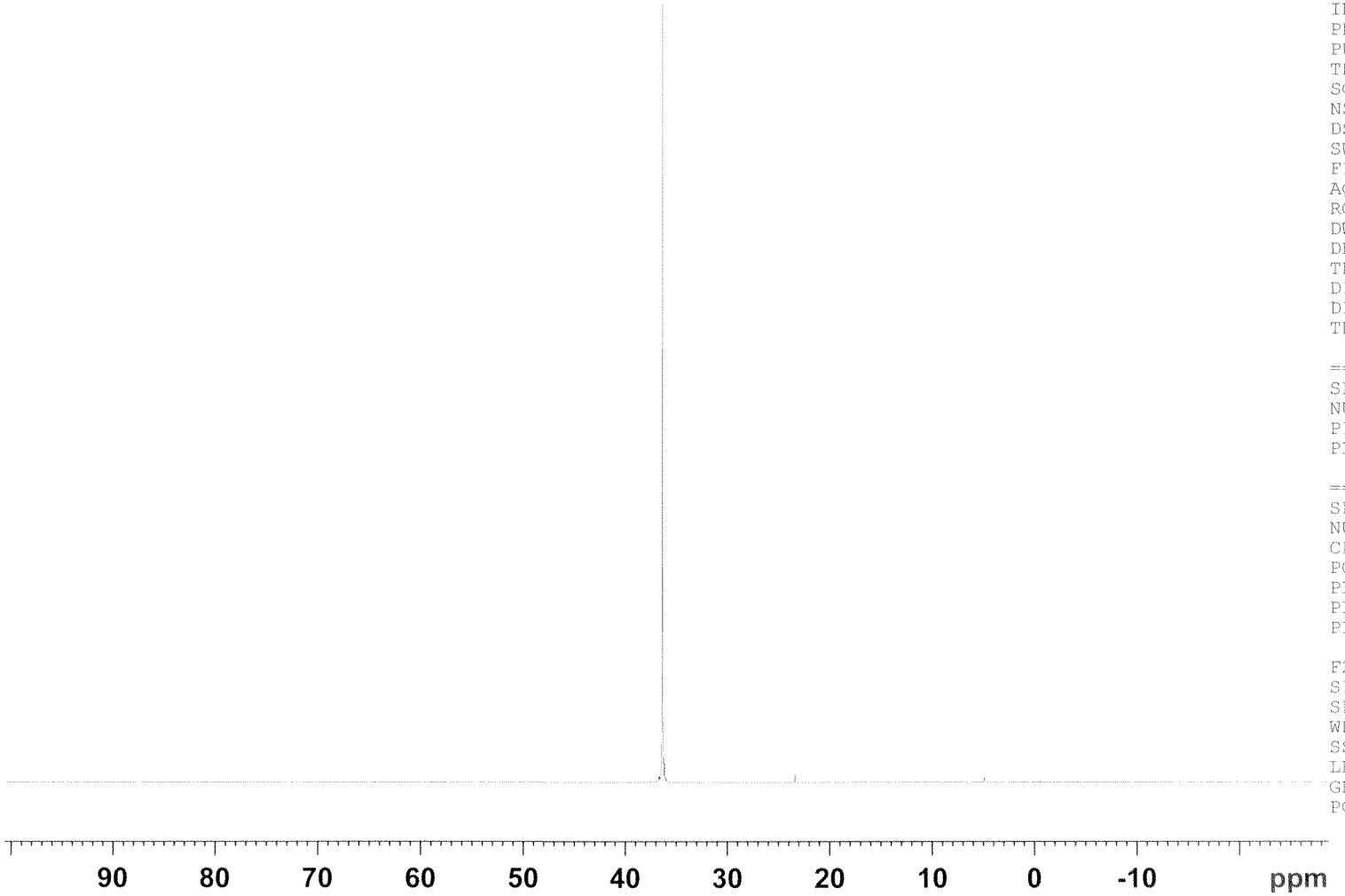
Compound (R_p)-33
^{13}C NMR



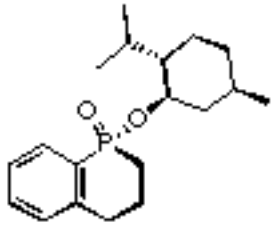

```
Current Data Parameters
NAME      OB 2092 after column
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150626
Time              17.30
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  124
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                294.8 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1         45.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                32768
SF           100.6127685 MHz
WDW                 EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



Compound (R_p)-34
$^{31}P\{^1H\}$ NMR decoupled



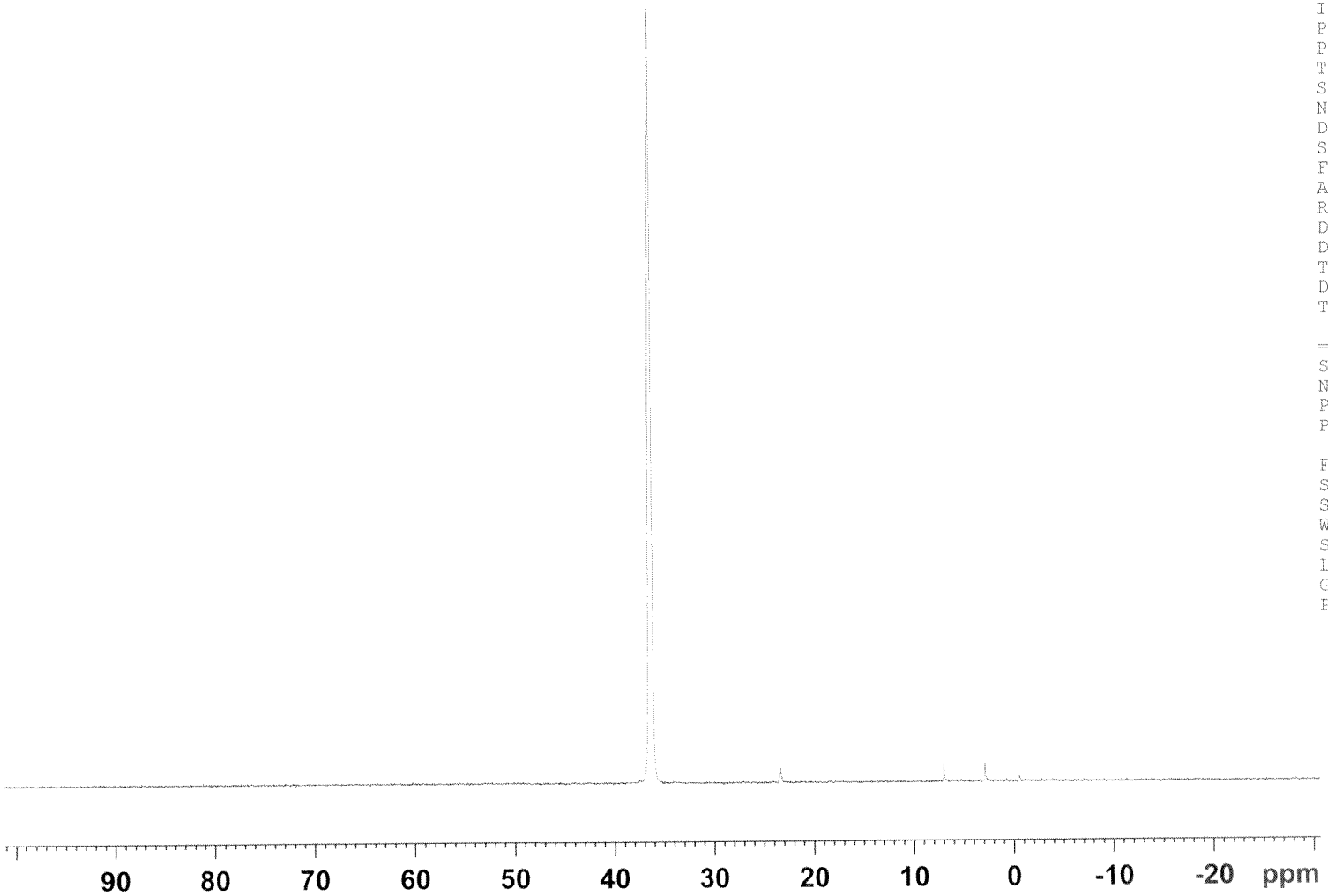

```
Current Data Parameters
NAME      OB 2097 after column
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20151201
Time               11.29
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     4
SWH            64102.563 Hz
FIDRES          0.978127 Hz
AQ              0.5111808 sec
RG                203.57
DW                 7.800 usec
DE                 6.50 usec
TE                291.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1         15.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2              90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                 32768
SF           161.9755930 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

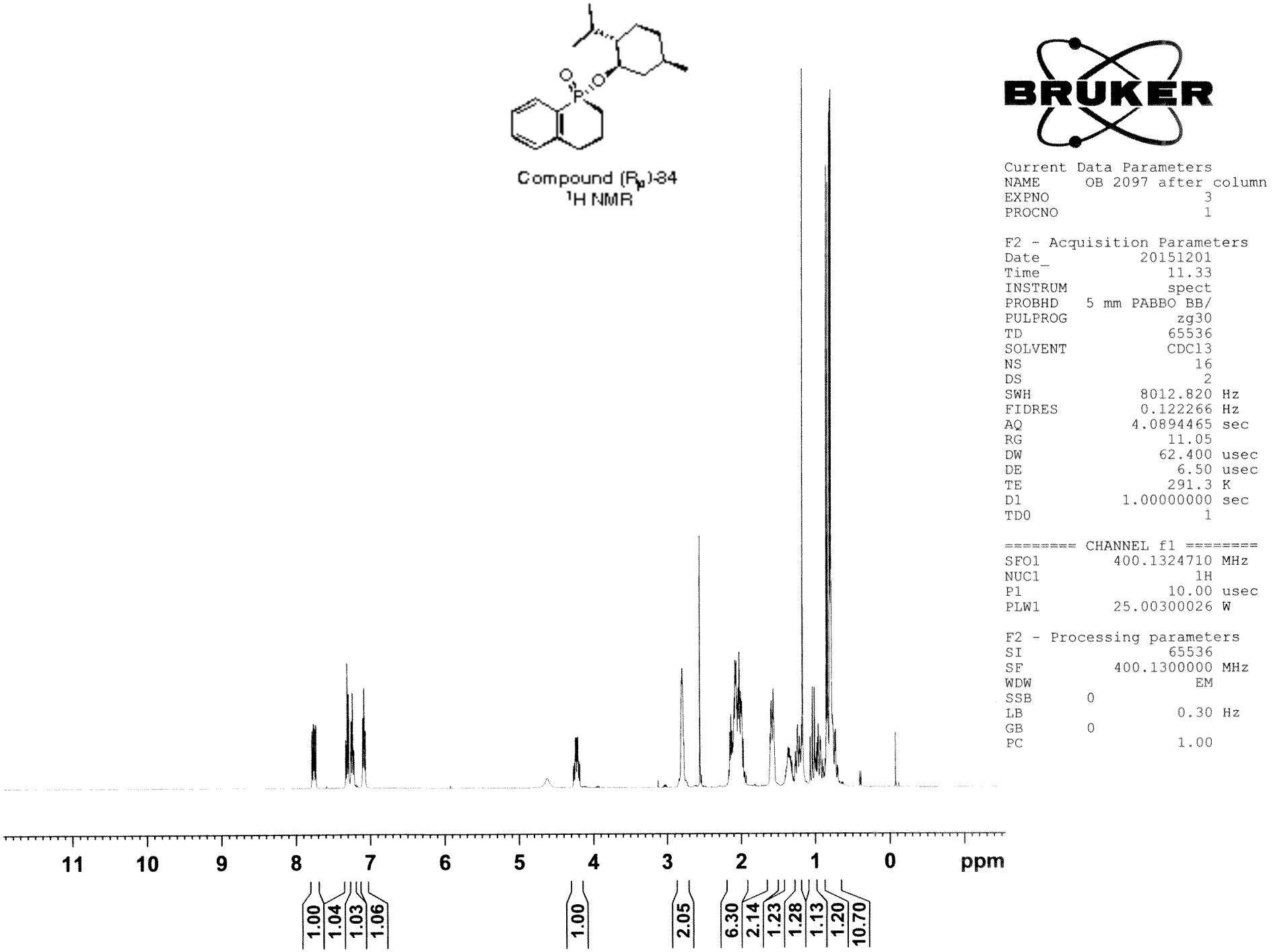


Compound (R_p)-34
$^{31}P\{^1H\}$ NMR coupled

BRUKER

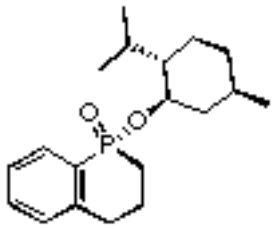
```
Current Data Parameters
NAME      OB 2097 after column
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_             20151201
Time                 11.31
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     32
DS                      4
SWH              64102.563 Hz
FIDRES           0.978127 Hz
AQ               0.5111808 sec
RG                  203.57
DW                 7.800 usec
DE                 6.50 usec
TE                 291.3 K
D1              2.00000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          161.9674942 MHz
NUC1                   31P
P1                 14.25 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                     32768
SF            161.9755930 MHz
WDW                    EM
SSB       0
LB                  2.00 Hz
GB        0
PC                  1.40
```



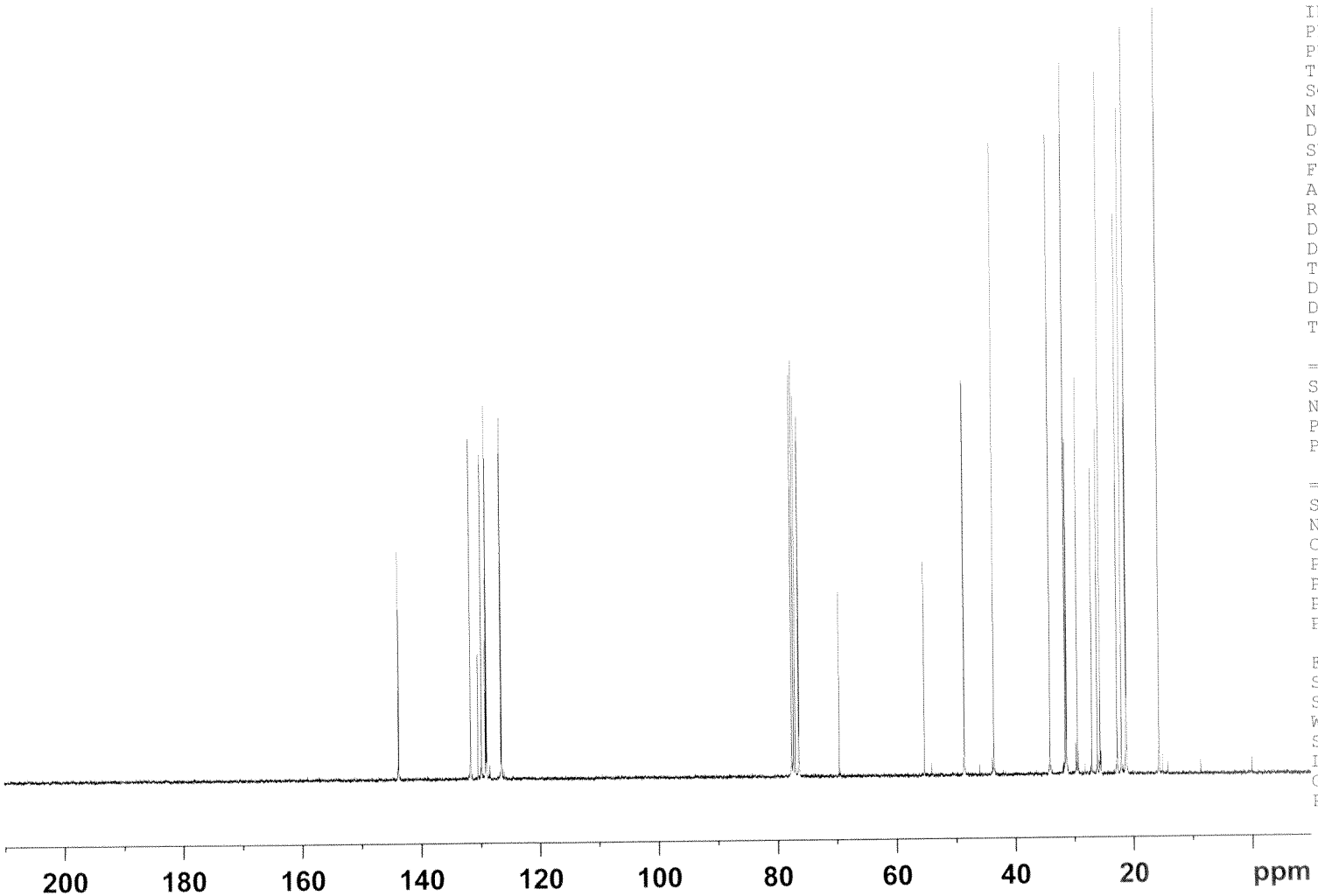


BRUKER

```
Current Data Parameters
NAME      OB 2097 after column
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_           20151201
Time                11.33
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  11.05
DW                62.400 usec
DE                 6.50 usec
TE                291.3 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                  1H
P1                 10.00 usec
PLW1          25.00300026 W

F2 - Processing parameters
SI                 65536
SF            400.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



Compound (R_p)-34
^{13}C NMR



BRUKER

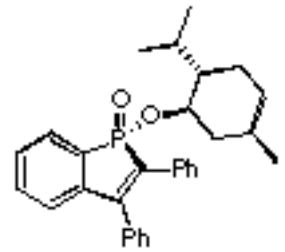
Current Data Parameters
NAME OB 2097 after column
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151201
Time 12.00
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 410
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 203.57
DW 20.800 usec
DE 6.50 usec
TE 292.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

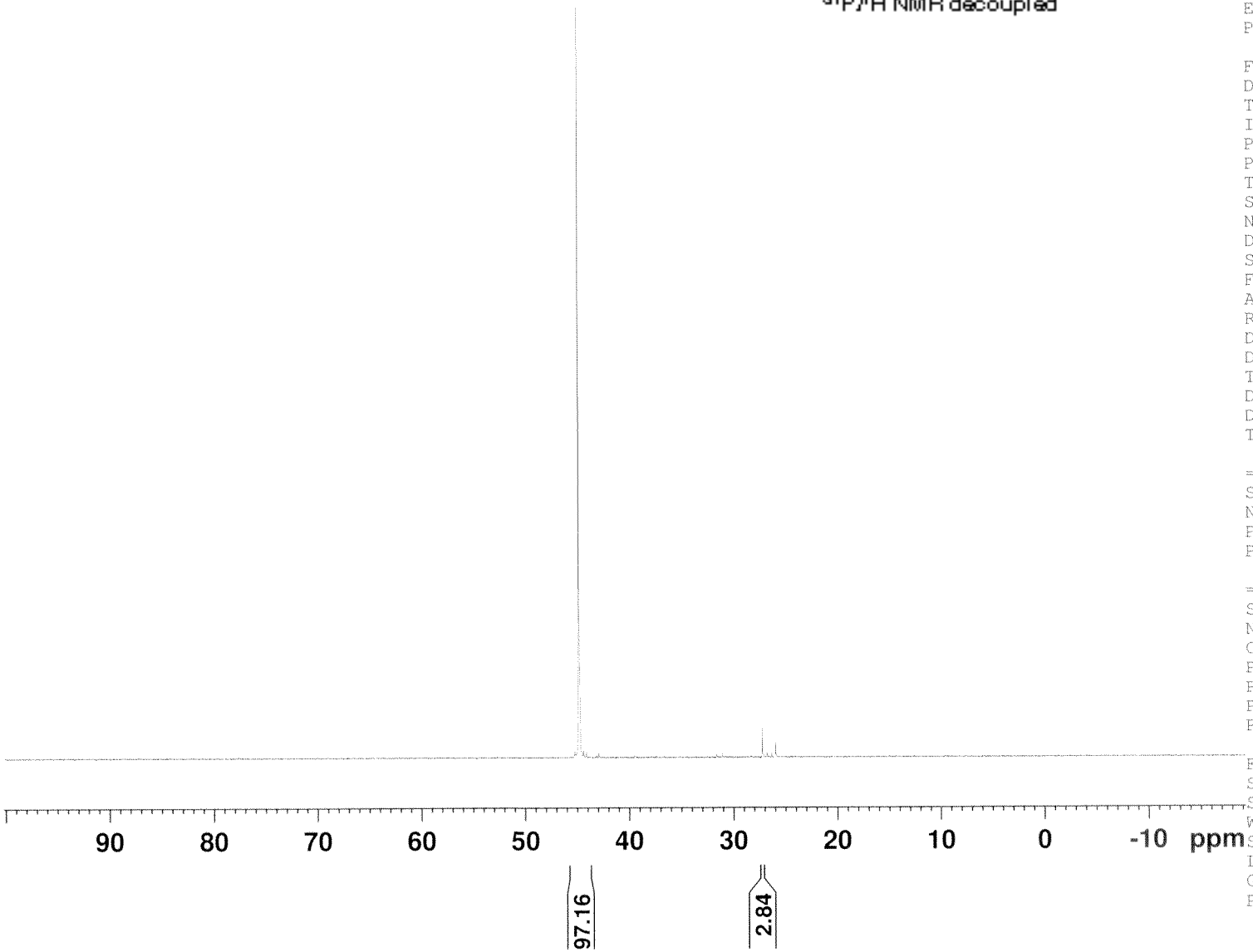
======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 10.00 usec
PLW1 45.00000000 W

======== CHANNEL f2 ========
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 10.00000000 W
PLW12 0.31604999 W
PLW13 0.25600001 W

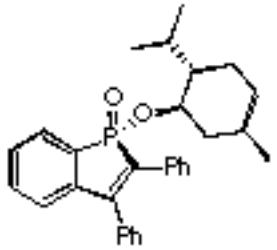
F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



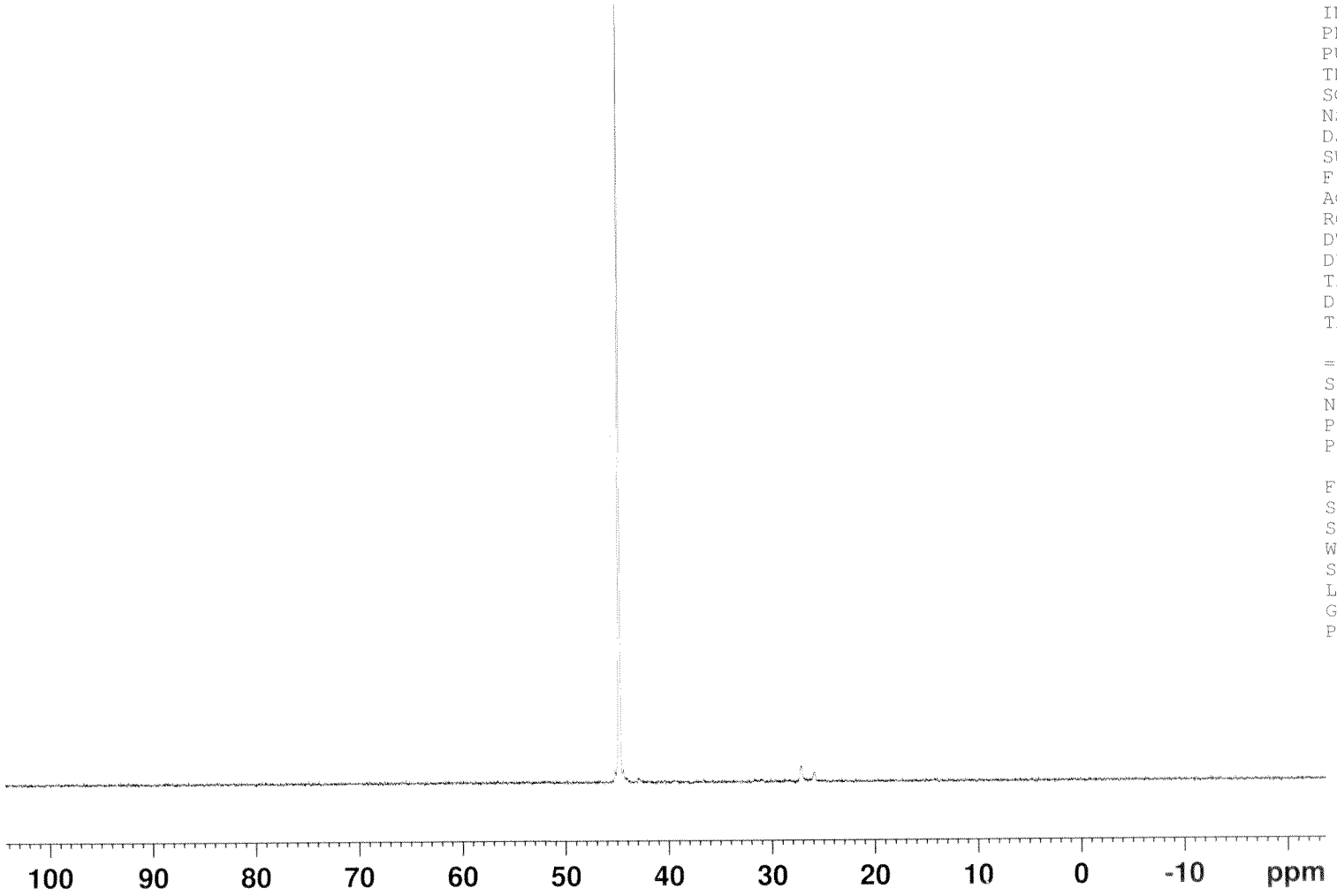
Compound (S_p)-35
$^{31}P\{^1H\}$ NMR decoupled



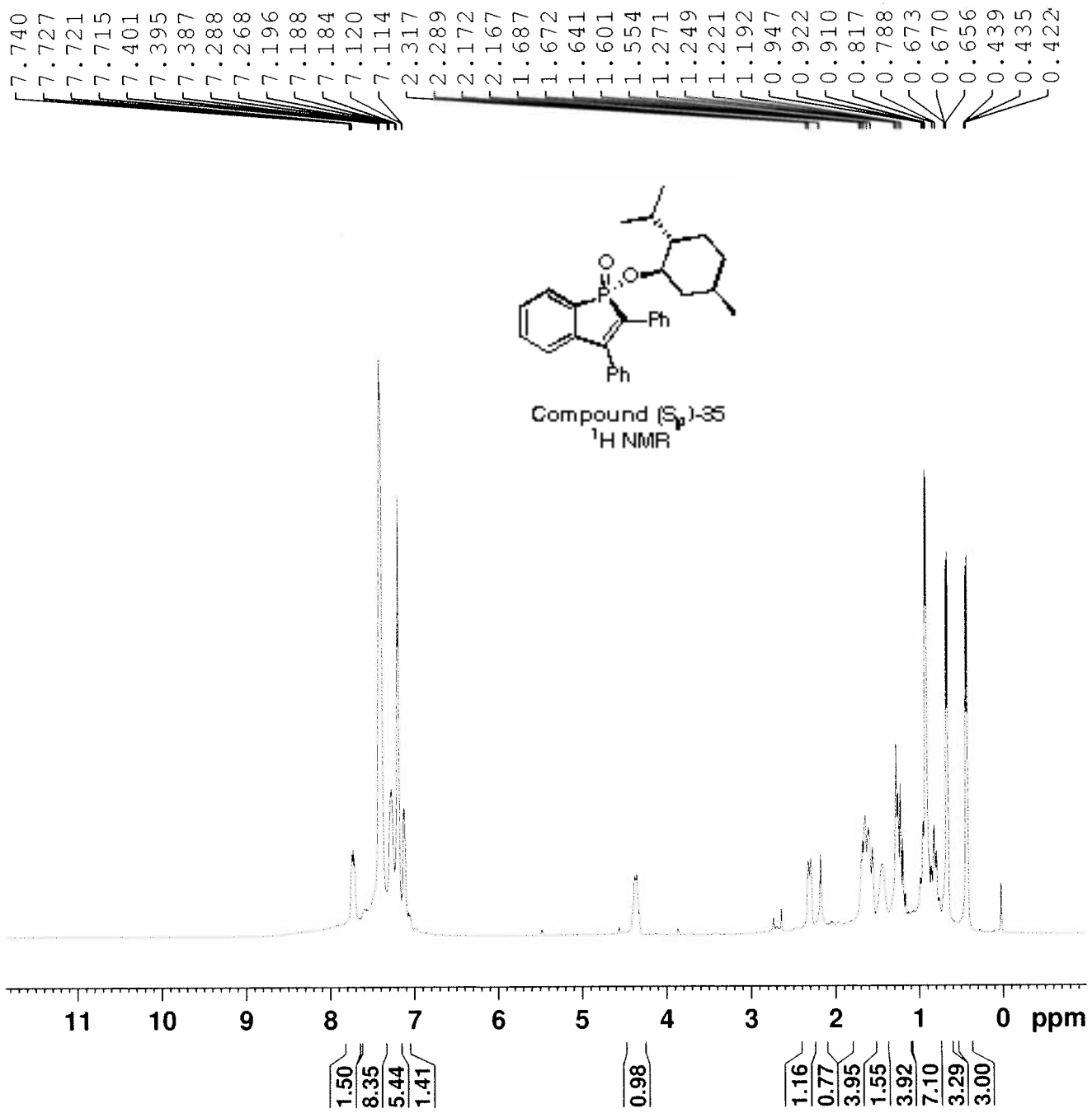

```
Current Data Parameters
NAME        OB 1481 pure
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140514
Time                9.36
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE               297.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1        15.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



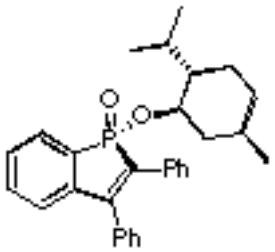
Compound (S_p)-35
$^{31}P\{^1H\}$ NMR coupled



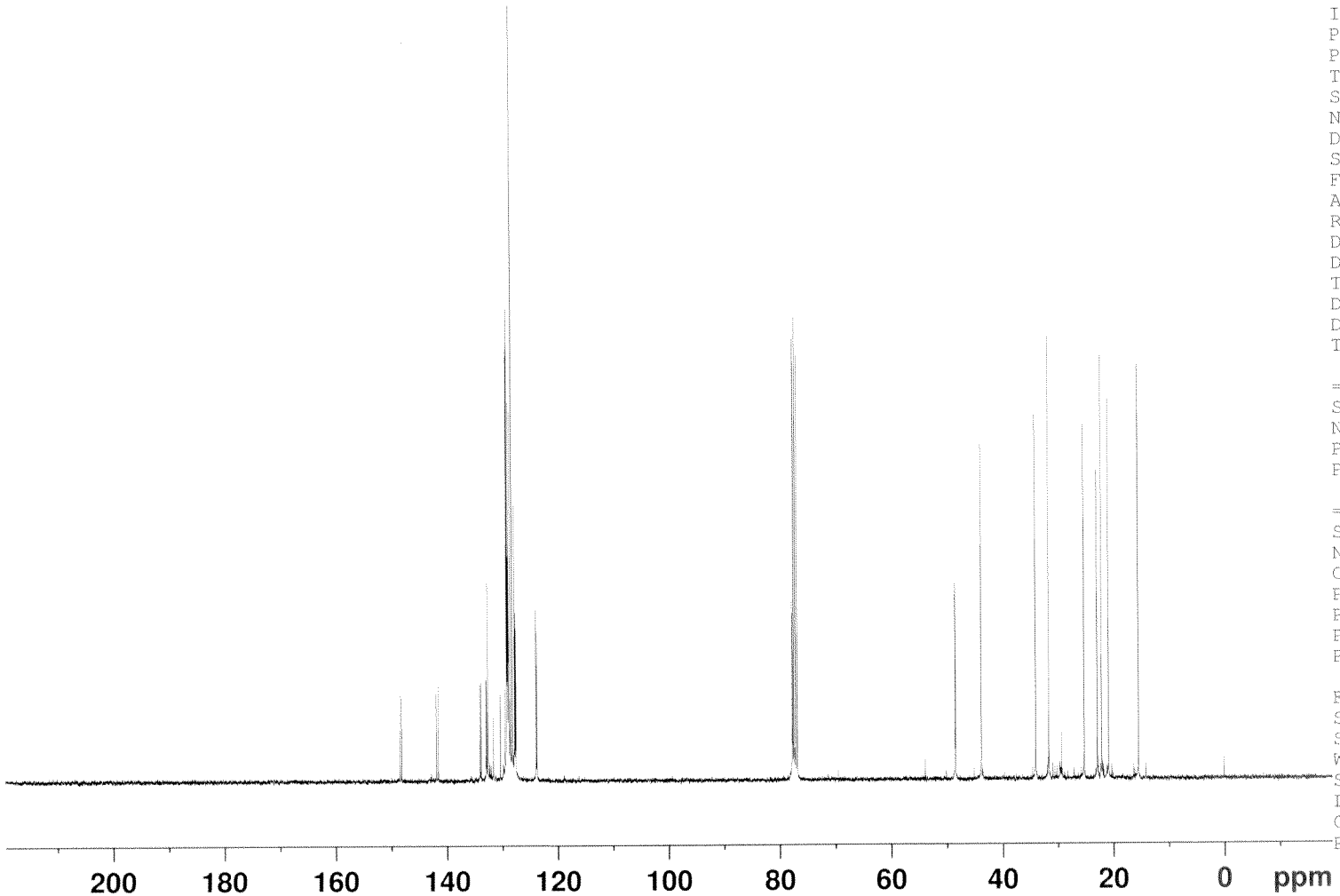

```
Current Data Parameters
NAME        OB 1481 pure
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140514
Time                9.38
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDC13
NS                    26
DS                     4
SWH            64102.563 Hz
FIDRES          0.978127 Hz
AQ              0.5111808 sec
RG                 203.57
DW                  7.800 usec
DE                  6.50 usec
TE                 296.4 K
D1            2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         161.9674942 MHz
NUC1                 31P
P1                 14.25 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 32768
SF           161.9755930 MHz
WDW                   EM
SSB       0
LB                  1.00 Hz
GB        0
PC                  1.40
```



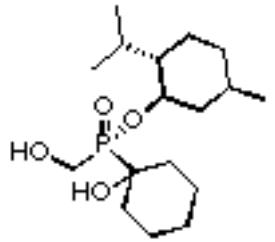

```
Current Data Parameters
NAME        OB 1481 pure
EXPNO                  3
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140514
Time                9.42
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 18.98
DW                62.400 usec
DE                 6.50 usec
TE                296.0 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                   1H
P1                10.00 usec
PLW1         25.00300026 W

F2 - Processing parameters
SI                 65536
SF           400.1300000 MHz
WDW                   EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```



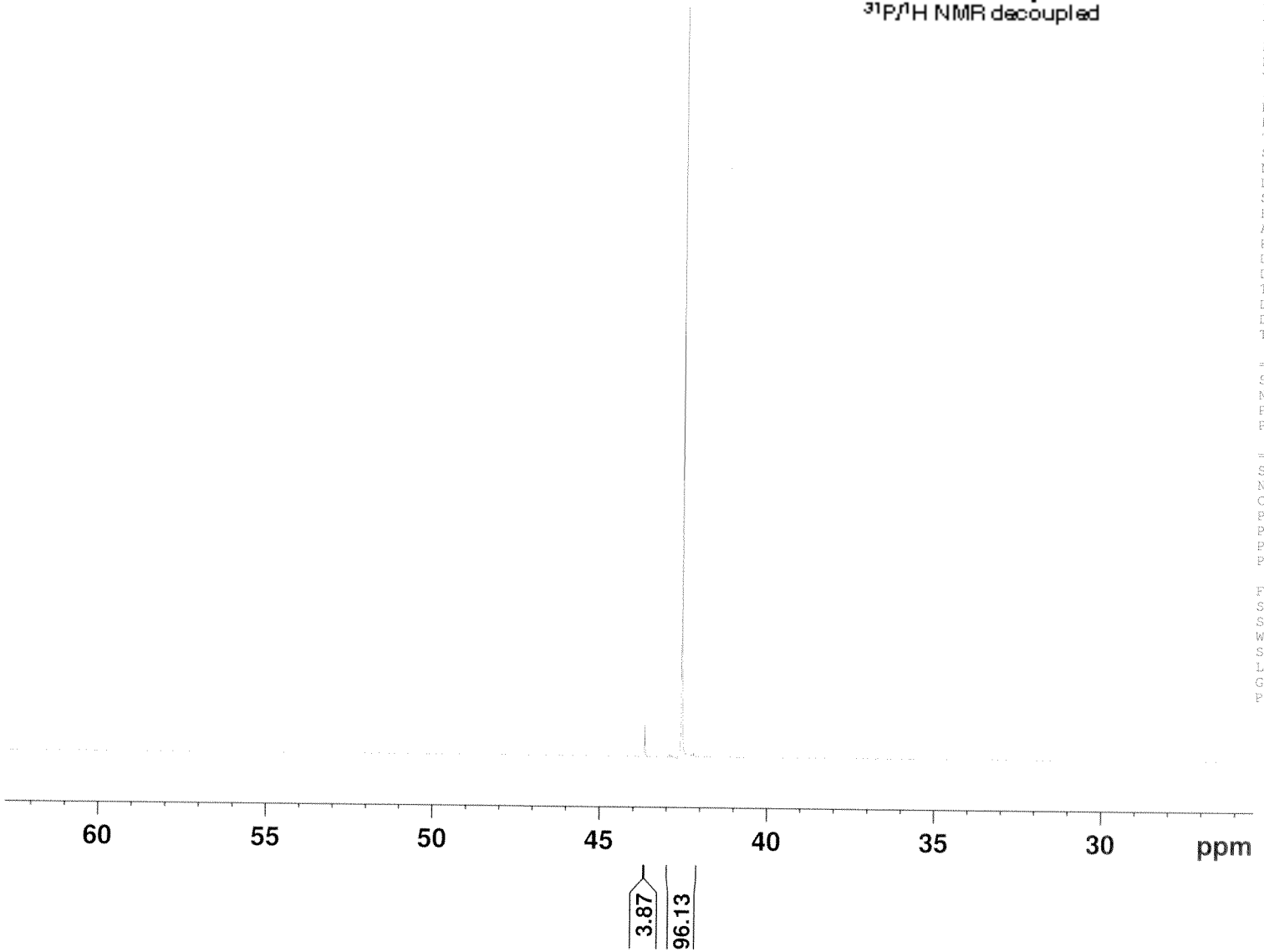
Compound (S_p)-35
^{13}C NMR



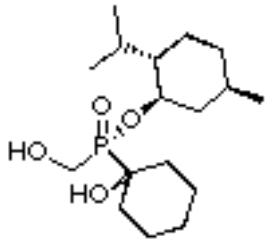

```
Current Data Parameters
NAME        OB 1481 pure
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20140514
Time               18.24
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  817
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                296.7 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

========== CHANNEL f1 ==========
SFO1         100.6228293 MHz
NUC1                  13C
P1                 10.00 usec
PLW1         45.00000000 W

========== CHANNEL f2 ==========
SFO2         400.1316005 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2              90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                 32768
SF           100.6127690 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



Compound (S_p)-37
$^{31}P\{^1H\}$ NMR decoupled

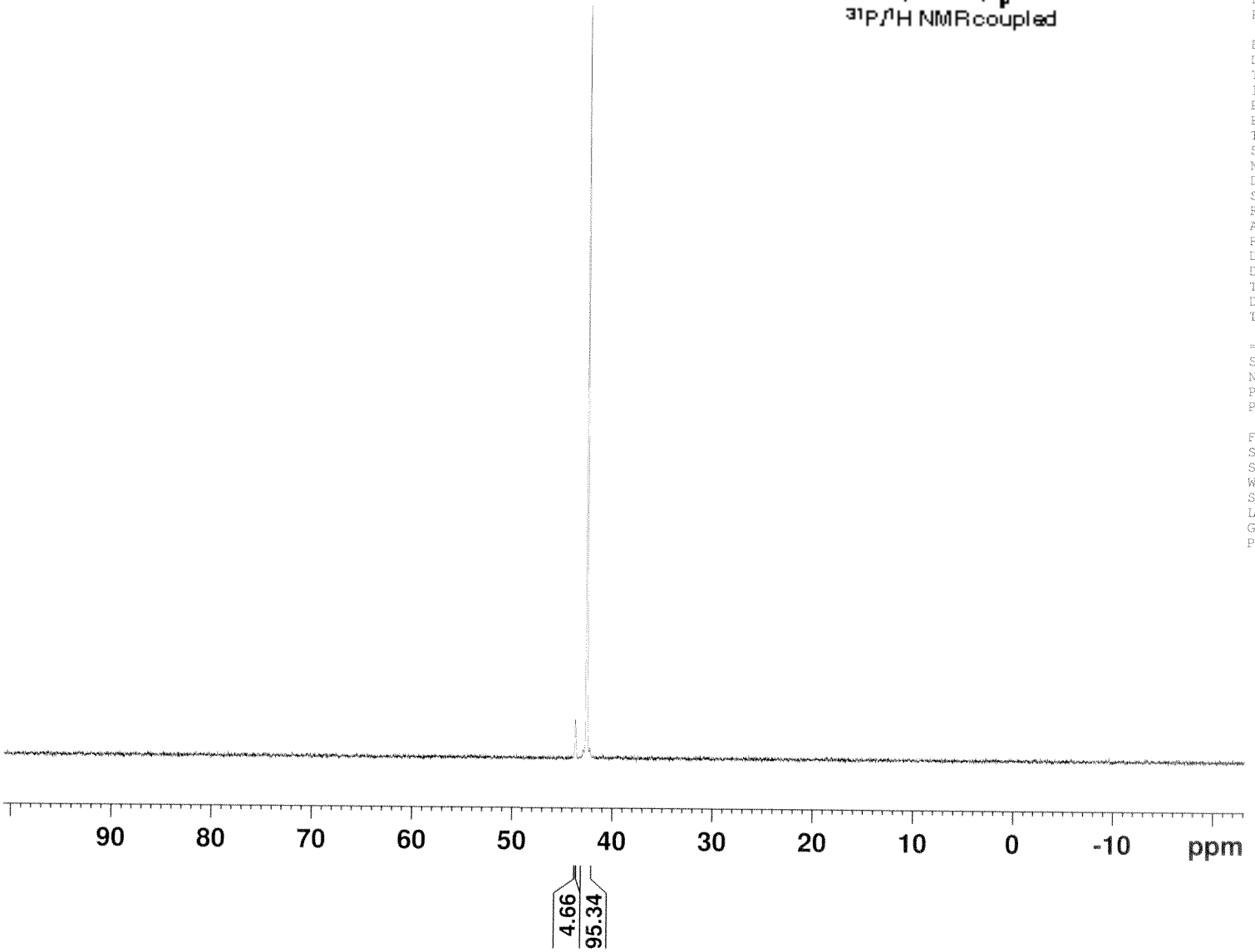



```
Current Data Parameters
NAME      OB 2105 1st crystallization
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_           20150716
Time              15.36
INSTRUM           spect
PROBHD    5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE               295.6 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1        15.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

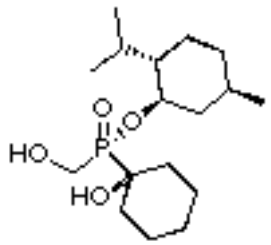


Compound (S_p)-37

$^{31}P/^{1}H$ NMR coupled

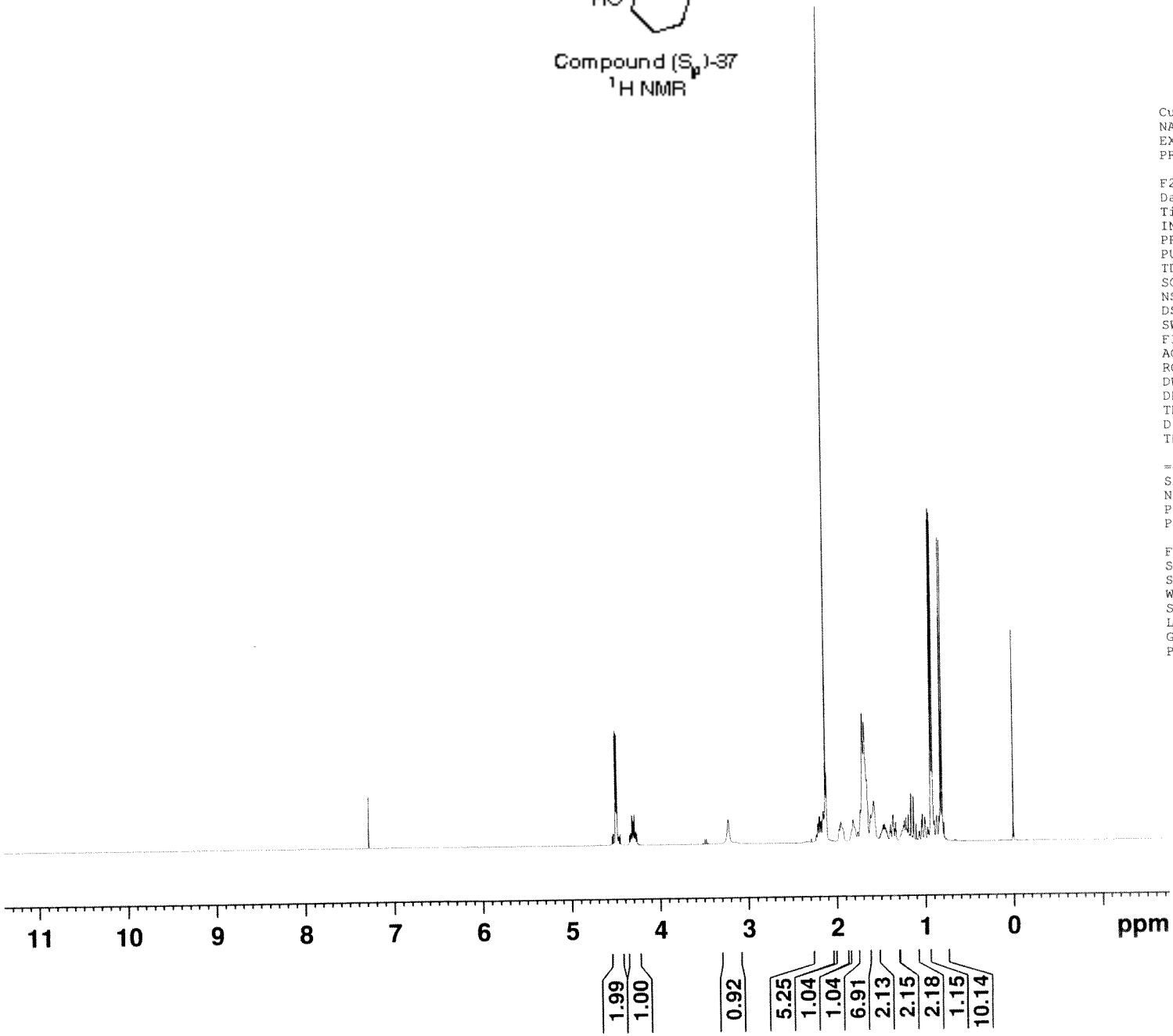


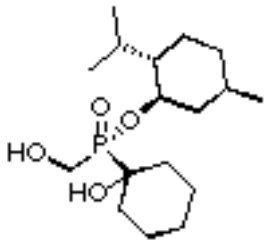

```
Current Data Parameters
NAME      OB 2105 1st crystallization
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20150716
Time              15.38
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  32
DS                   4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE               295.2 K
D1            2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1               31P
P1               14.25 usec
PLW1        15.00000000 W

F2 - Processing parameters
SI               32768
SF          161.9755930 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



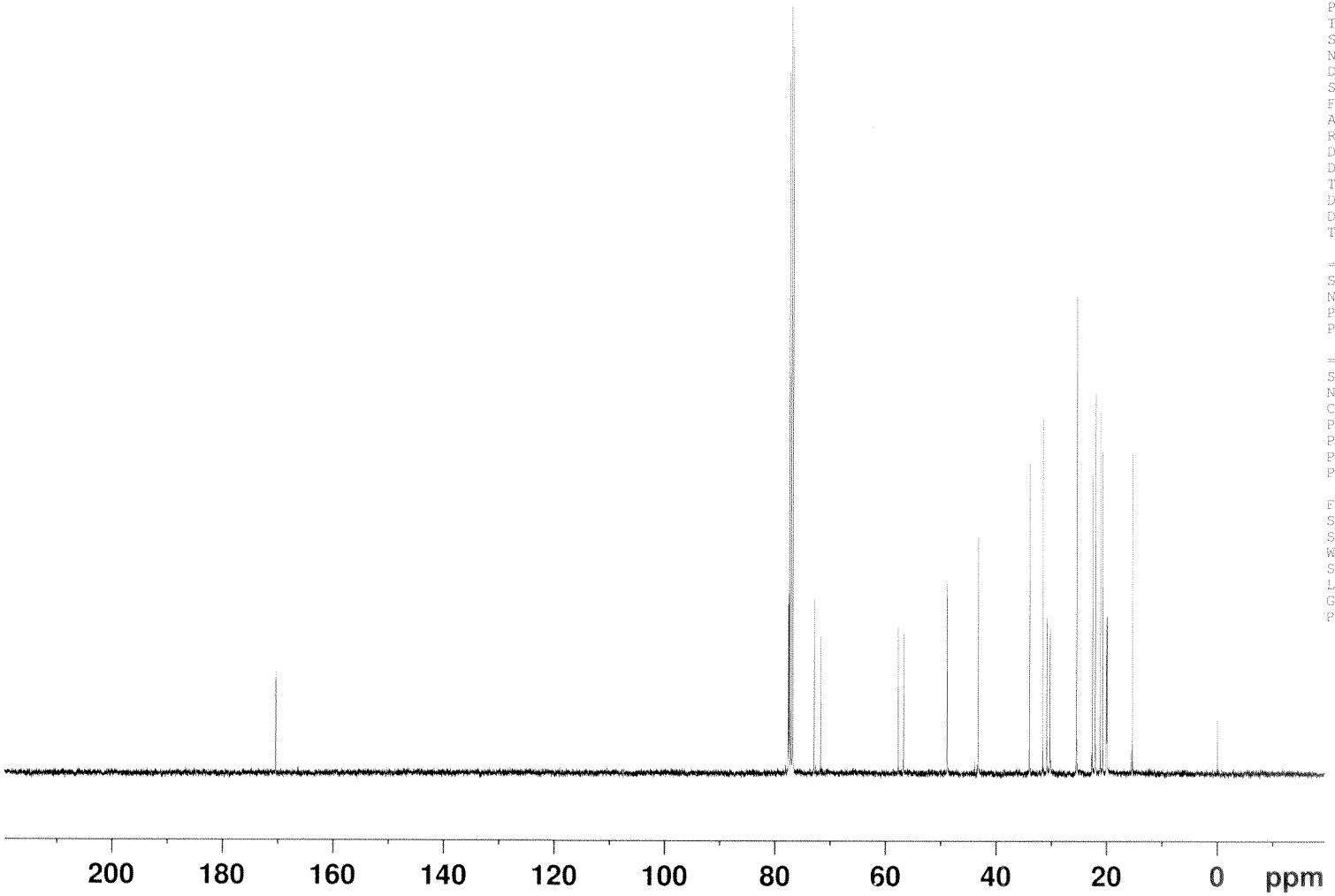
Compound (S_p)-37
^{1}H NMR

```
Current Data Parameters
NAME      OB 2105 1st crystallization
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20150716
Time              15.40
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                   9
DS                   2
SWH           8012.820 Hz
FIDRES           0.122266 Hz
AQ            4.0894465 sec
RG                32.38
DW               62.400 usec
DE                6.50 usec
TE              295.2 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        400.1324710 MHz
NUC1               1H
P1               10.00 usec
PLW1        25.00300026 W

F2 - Processing parameters
SI               65536
SF          400.1300000 MHz
WDW                EM
SSB       0
LB               0.30 Hz
GB        0
PC               1.00
```

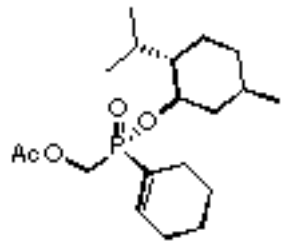




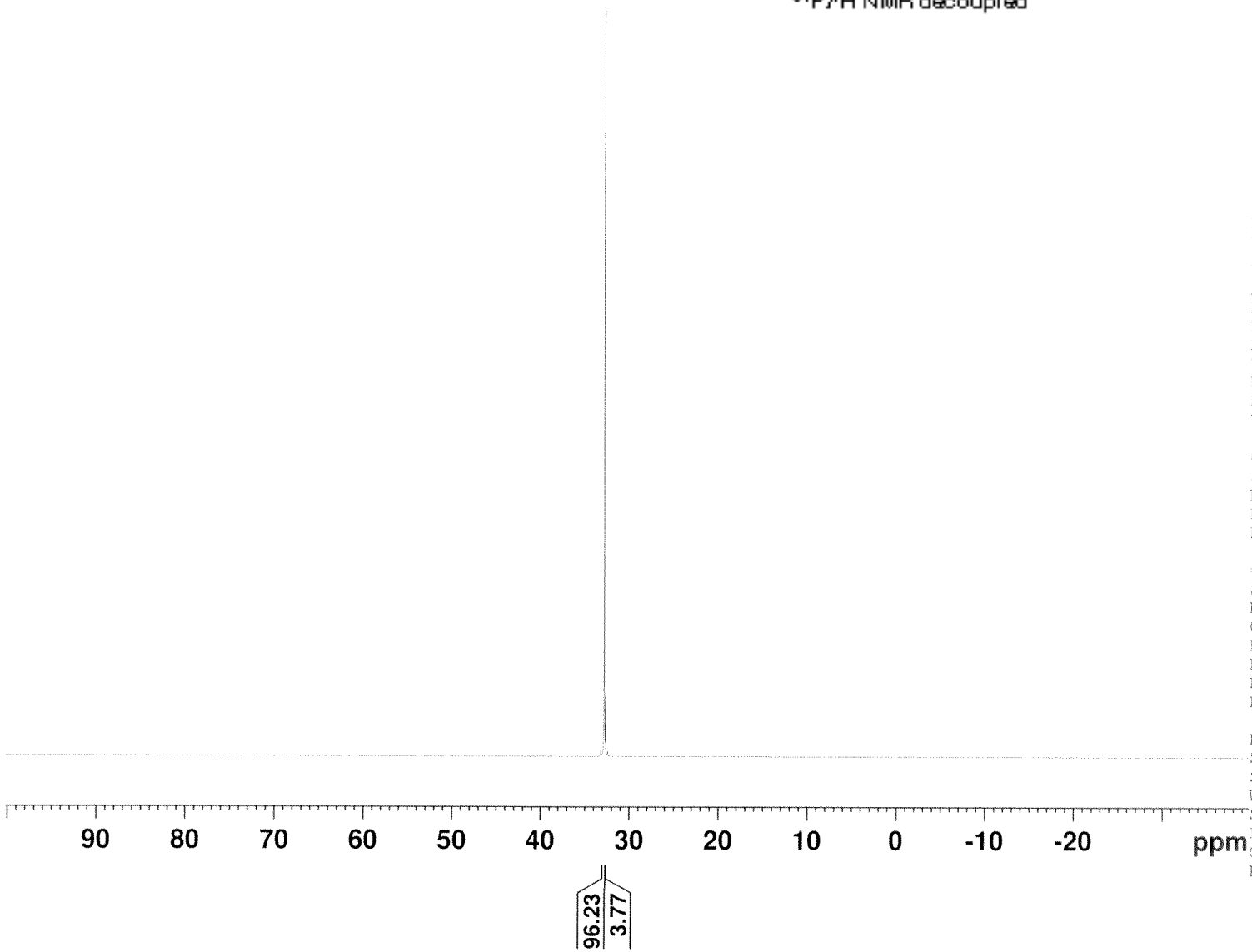
Compound (S_p)-37
^{13}C NMR



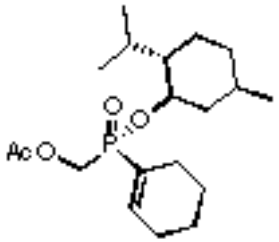

```
Current Data Parameters
NAME      OB 2105 1st crystallization
EXPNO                 4
PROCNO                1

F2 - Acquisition Parameters
Date_          20150716
Time              17.20
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  608
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                295.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1         45.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                32768
SF          100.6127685 MHz
WDW                EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



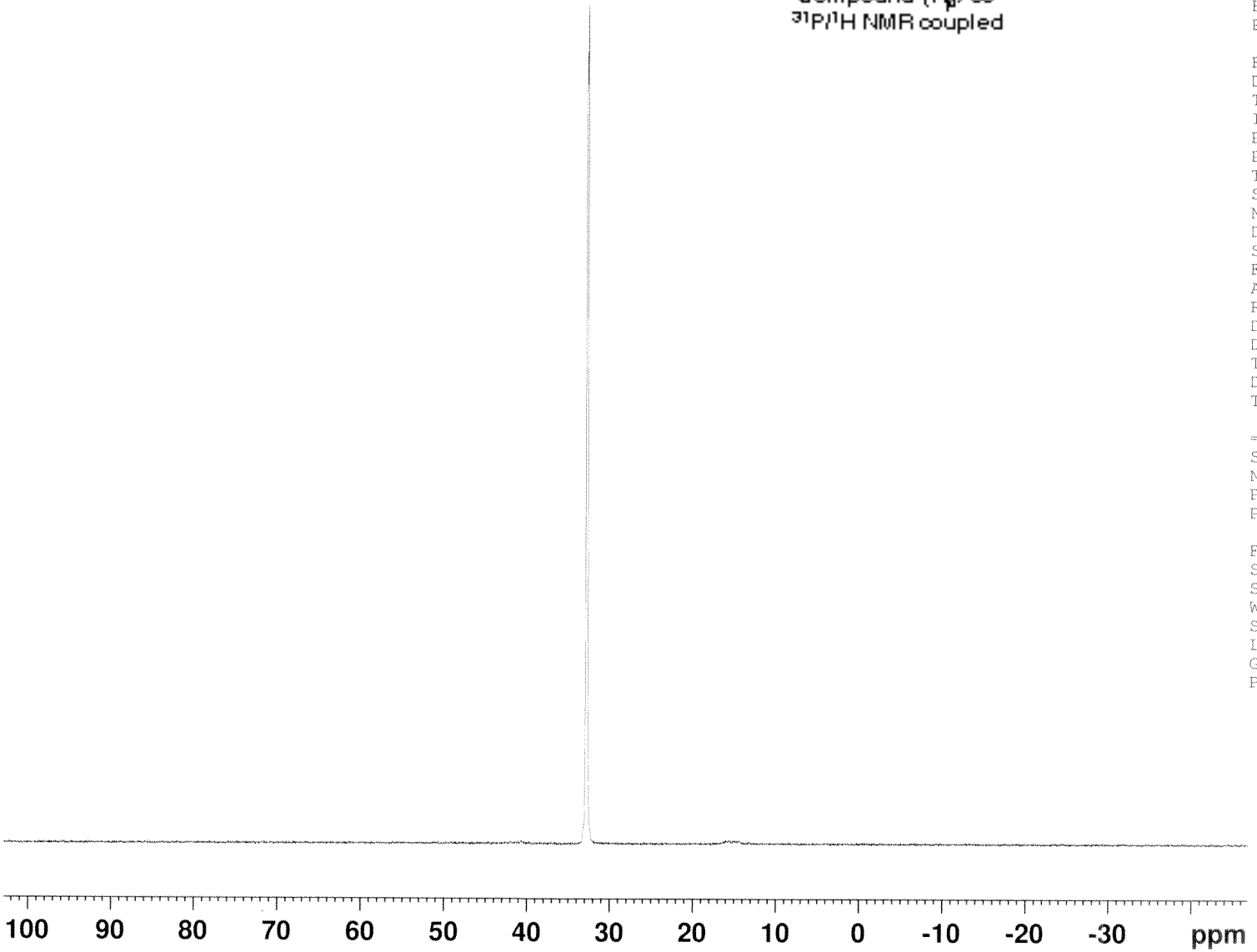
Compound (R_p)-38
$^{31}P\{^1H\}$ NMR decoupled



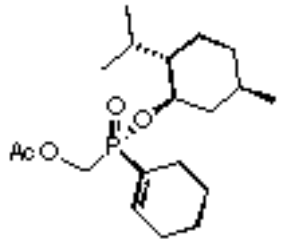

```
Current Data Parameters
NAME      OB 2096 after column
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150701
Time               10.05
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE                294.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1        15.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



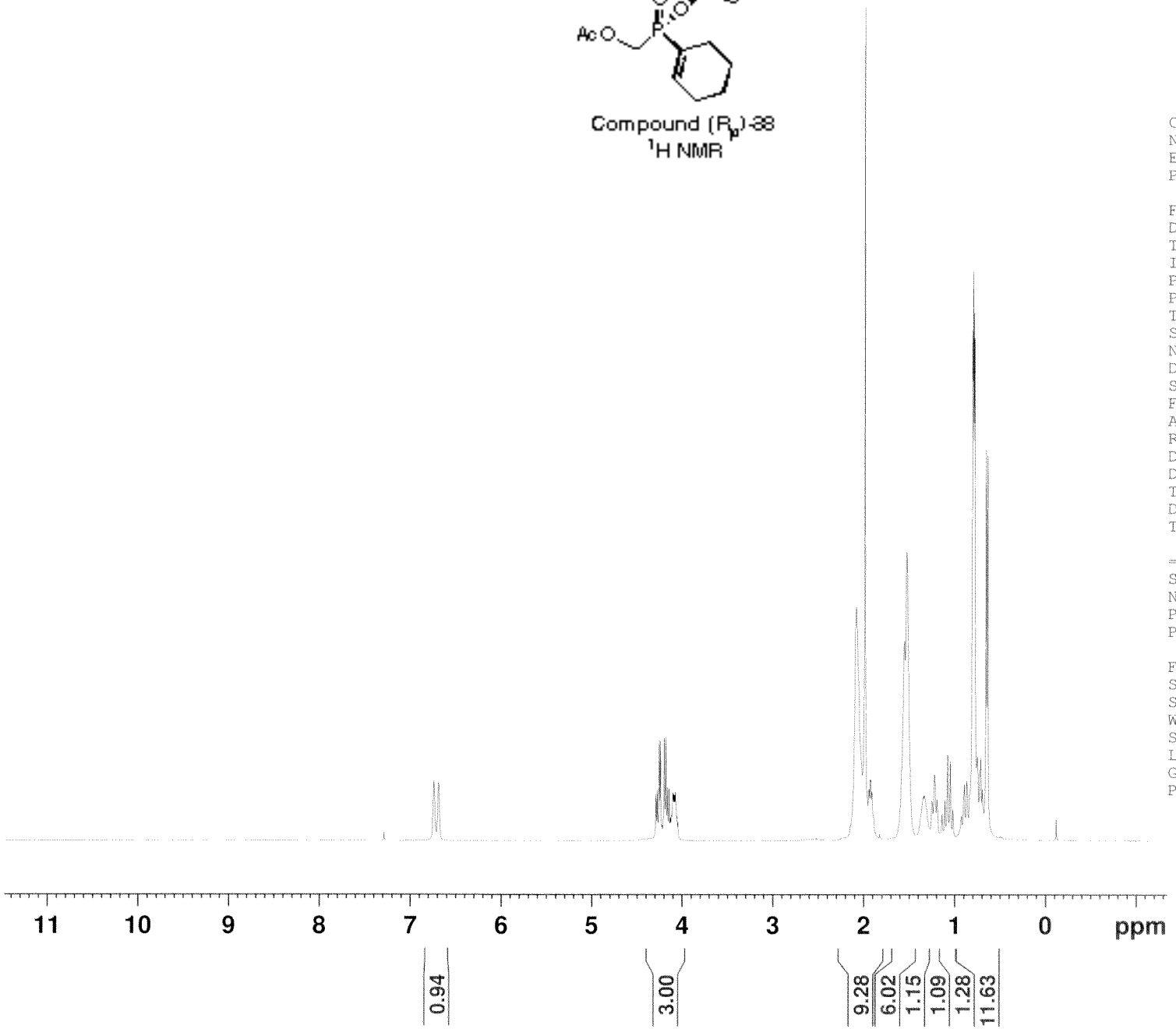
Compound (R_p)-38
$^{31}P\{^{1}H\}$ NMR coupled



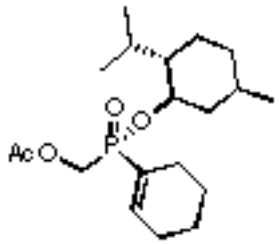

```
Current Data Parameters
NAME      OB 2096 after column
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150701
Time               10.07
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH            64102.563 Hz
FIDRES          0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                 7.800 usec
DE                 6.50 usec
TE                294.4 K
D1           2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1         15.00000000 W

F2 - Processing parameters
SI                 32768
SF           161.9755930 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



Compound (R_p)-38
^{1}H NMR

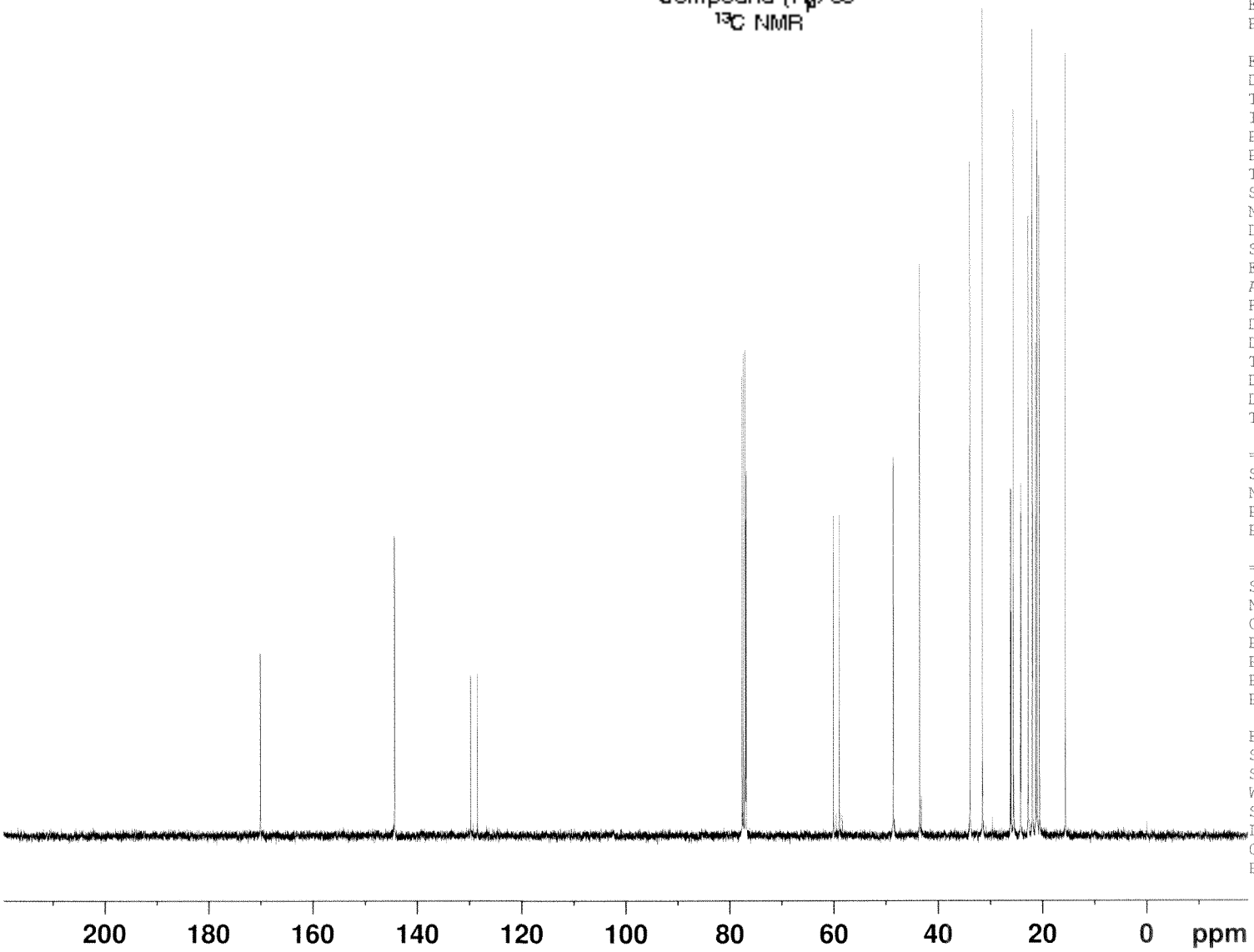



```
Current Data Parameters
NAME      OB 2096 after column
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150701
Time               10.08
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ             4.0894465 sec
RG                 11.05
DW                62.400 usec
DE                 6.50 usec
TE                294.3 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1                1H
P1                10.00 usec
PLW1        25.00300026 W

F2 - Processing parameters
SI                 65536
SF           400.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

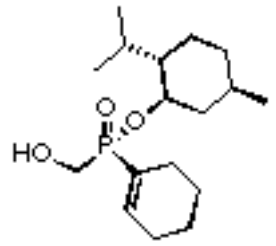


Compound (R_p)-38

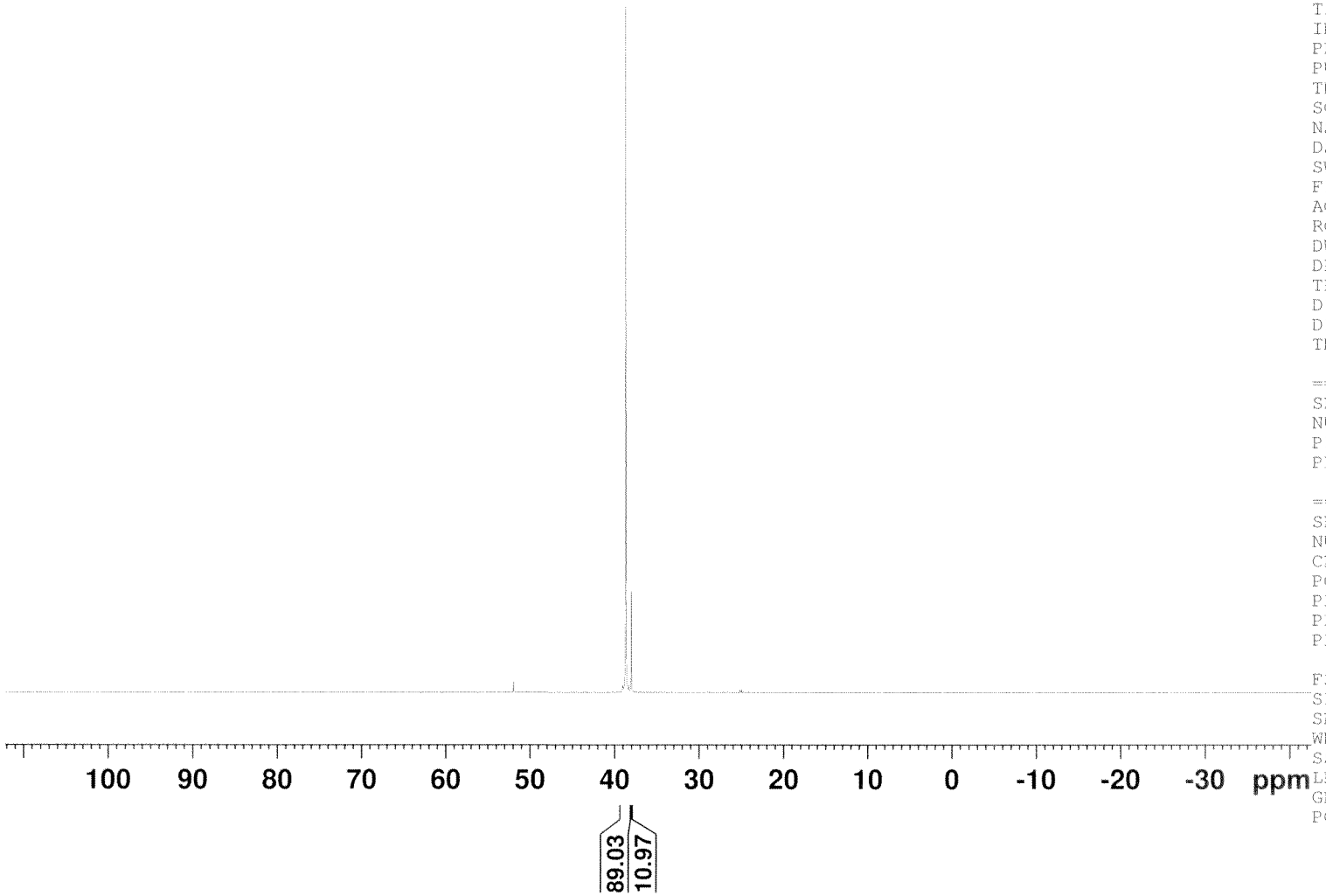
^{13}C NMR



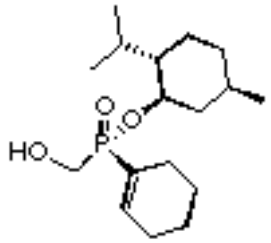

```
Current Data Parameters
NAME      OB 2096 after column
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150701
Time              10.21
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  101
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               203.57
DW               20.800 usec
DE                6.50 usec
TE               294.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

========== CHANNEL f1 =========
SFO1        100.6228293 MHz
NUC1               13C
P1               10.00 usec
PLW1        45.00000000 W

========== CHANNEL f2 =========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2        waltz16
PCPD2            90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                32768
SF          100.6127685 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



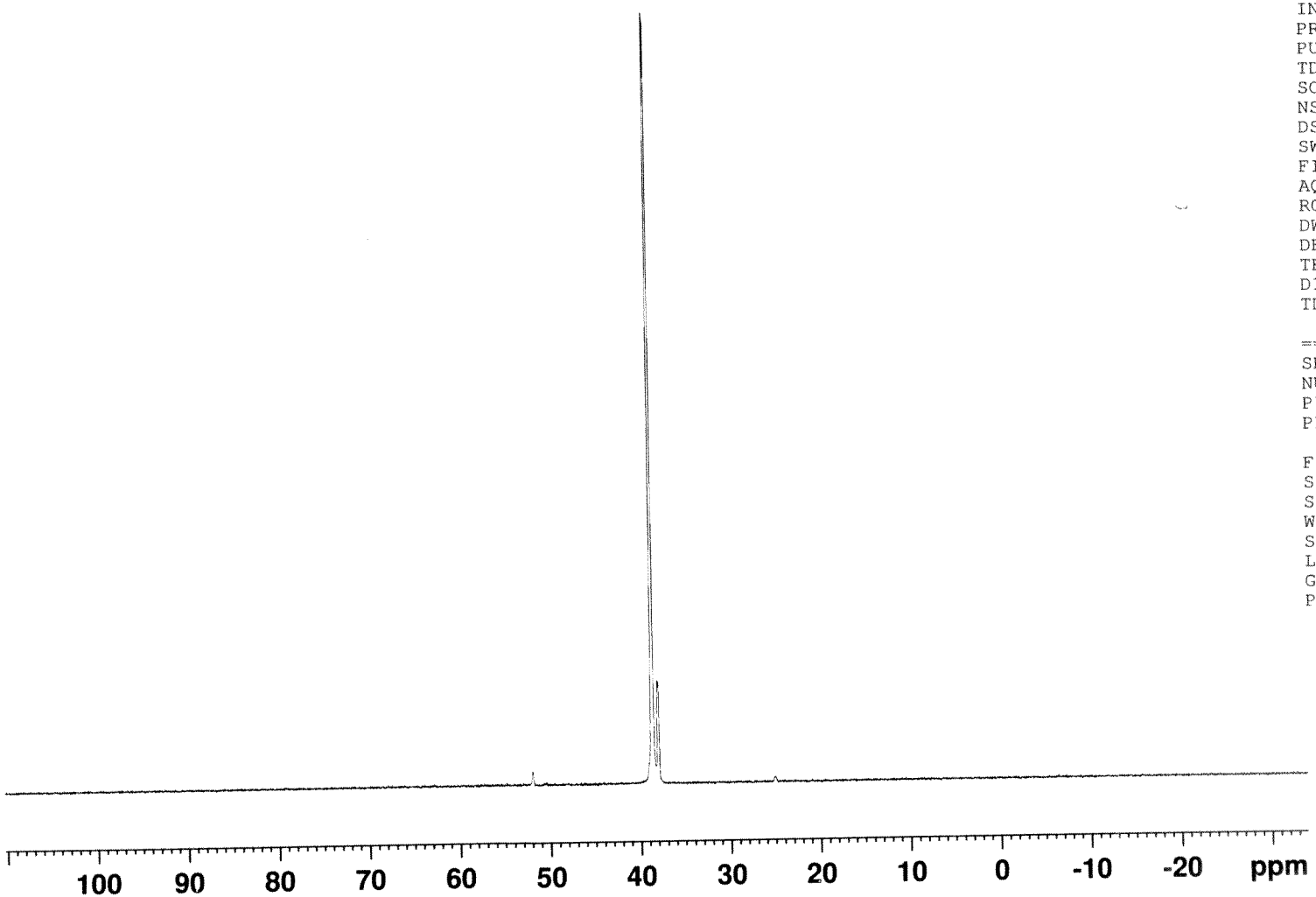
$^{31}P\{^{1}H\}$ NMR decoupled



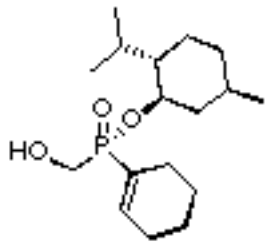

```
Current Data Parameters
NAME      OB 2114 after column
EXPNO                    5
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150715
Time               16.05
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE                295.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1        15.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



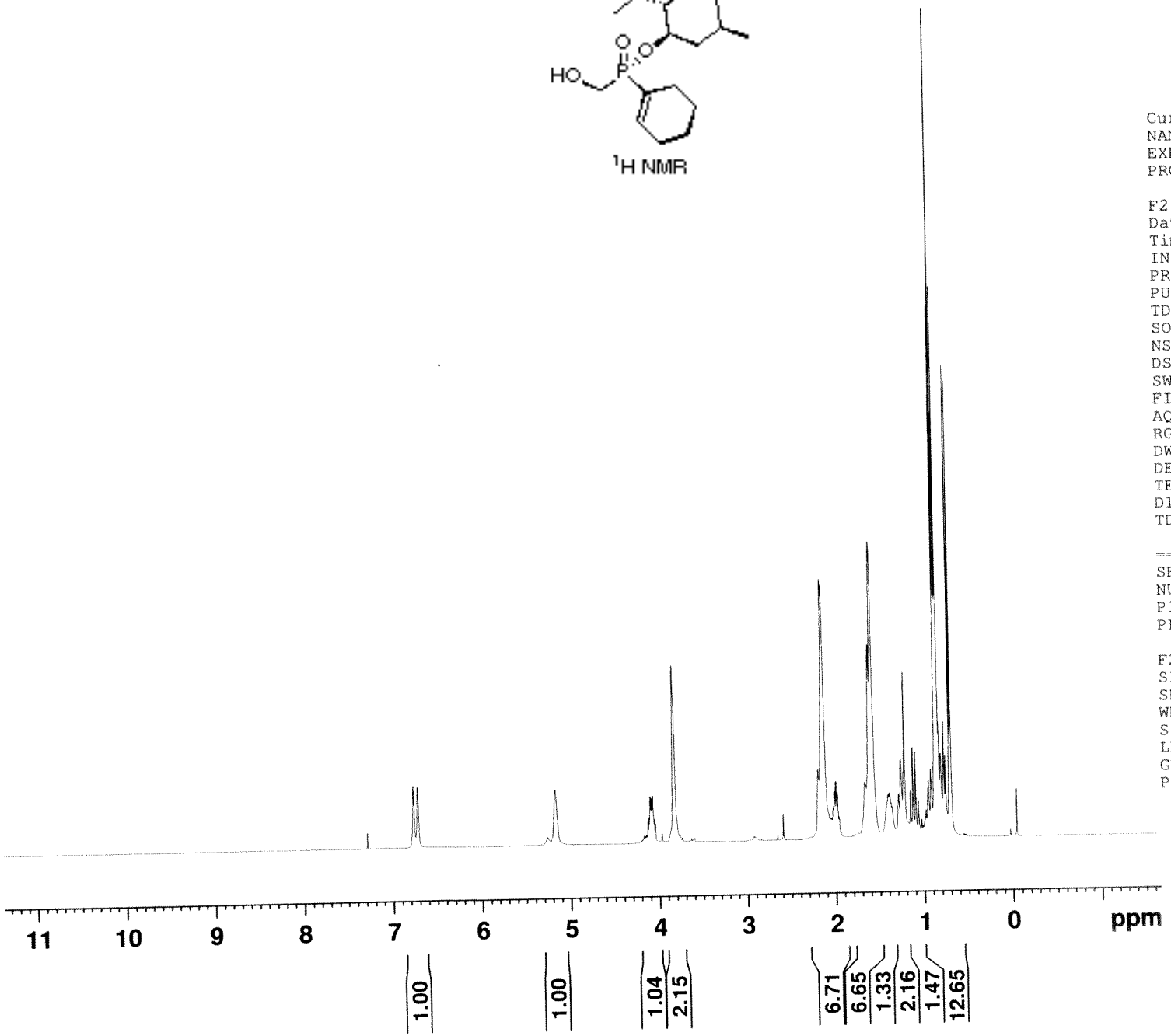
$^{31}P\{^{1}H\}$ NMR coupled




```
Current Data Parameters
NAME      OB 2114 after column
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150715
Time              16.07
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  32
DS                   4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                 7.800 usec
DE                 6.50 usec
TE                295.0 K
D1           2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1              31P
P1                14.25 usec
PLW1       15.00000000 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



^{1}H NMR



Current Data Parameters

NAME	OB 2114 after column
EXPNO	3
PROCNO	1

F2 - Acquisition Parameters

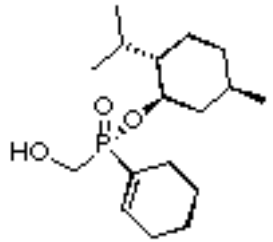
Date_	20150715
Time	16.09
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	10
DS	2
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894465 sec
RG	12.96
DW	62.400 usec
DE	6.50 usec
TE	295.0 K
D1	1.00000000 sec
TD0	1

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

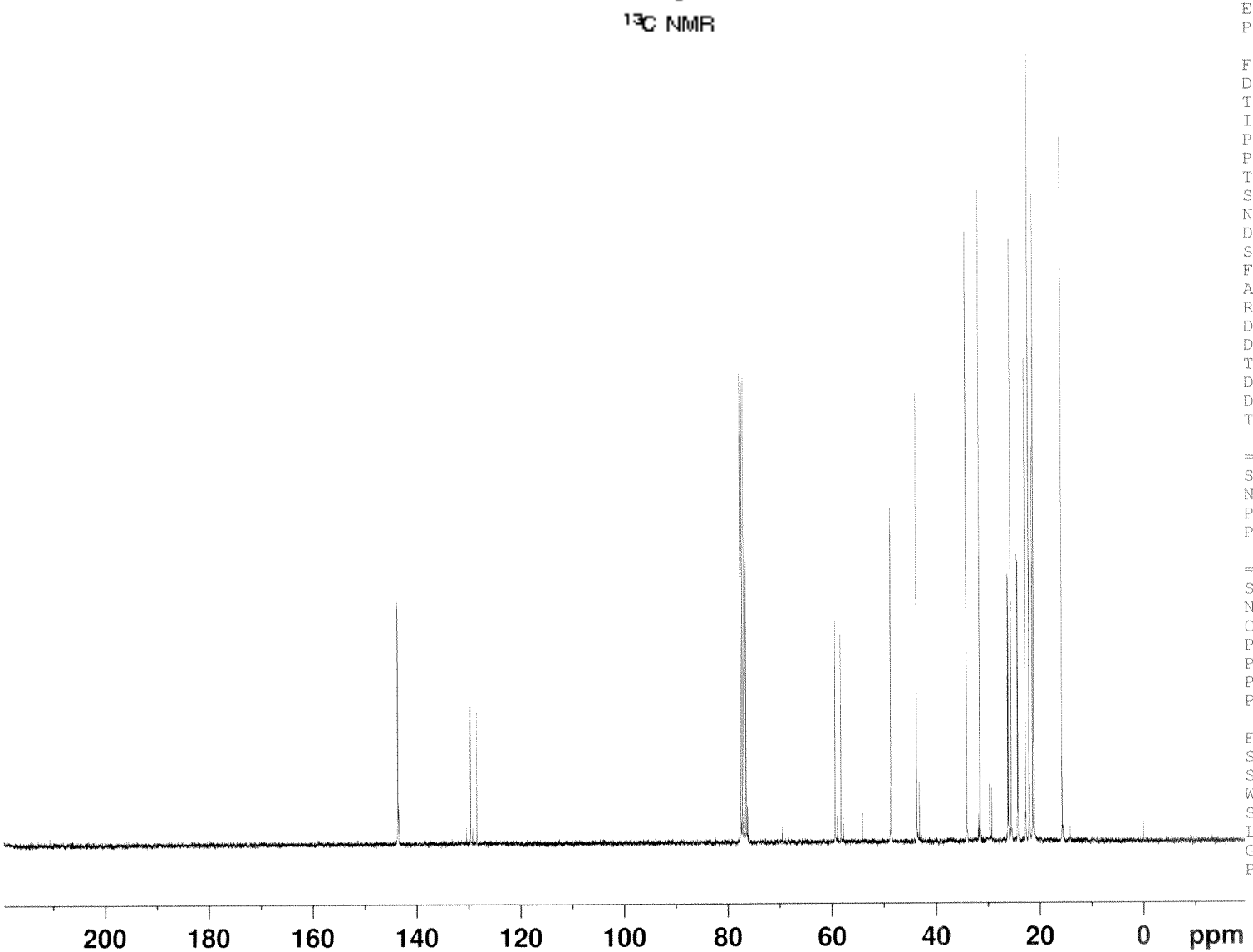
SFO1	400.1324710 MHz
NUC1	1H
P1	10.00 usec
PLW1	25.00300026 W

F2 - Processing parameters

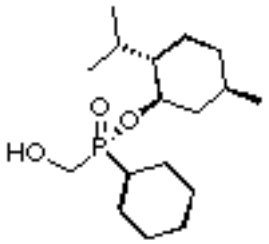
SI	65536
SF	400.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



^{13}C NMR

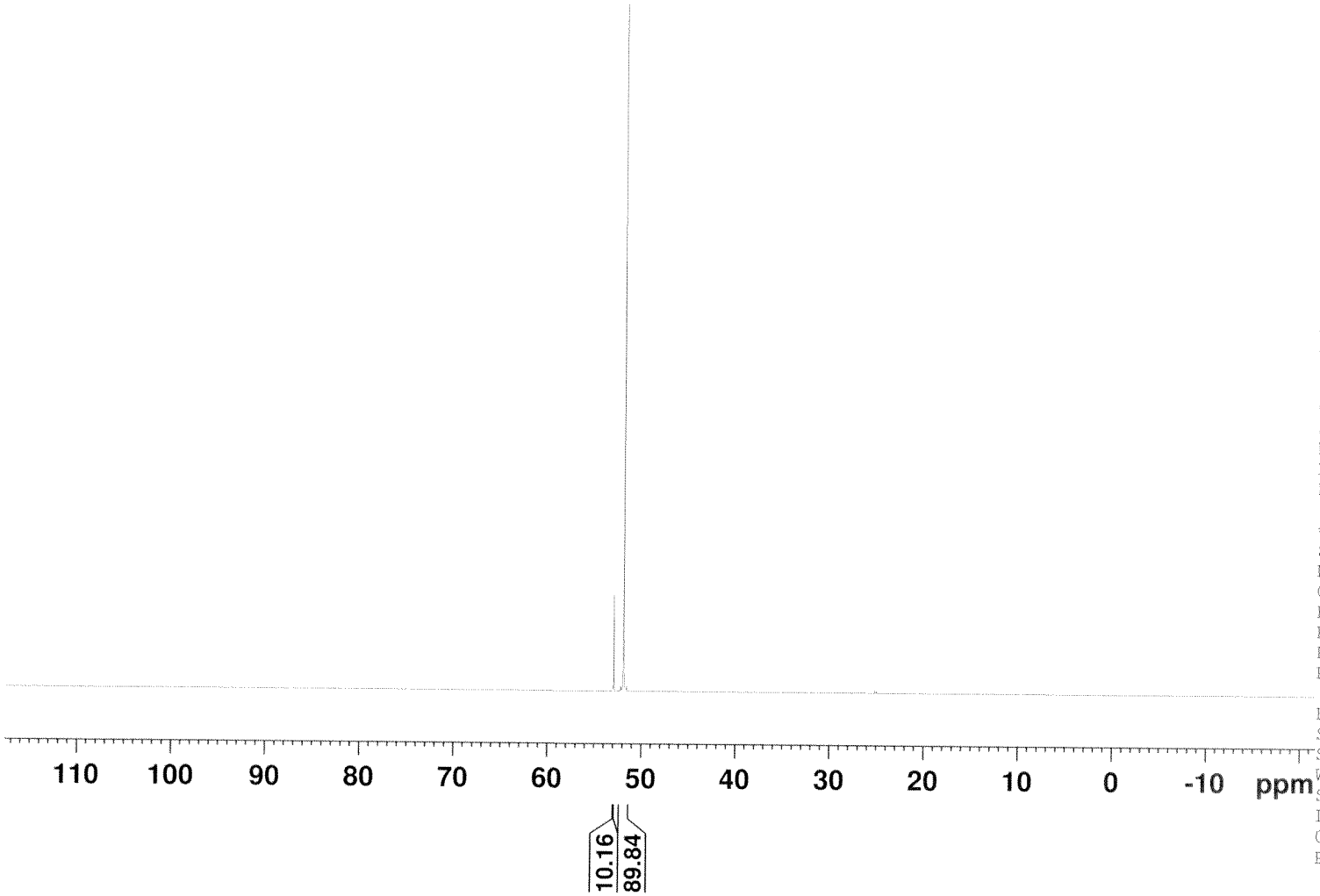



```
Current Data Parameters
NAME      OB 2114 after column
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_           20150715
Time               16.27
INSTRUM            spect
PROBHD    5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  275
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                295.8 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1         45.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                 32768
SF           100.6127685 MHz
WDW                  EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

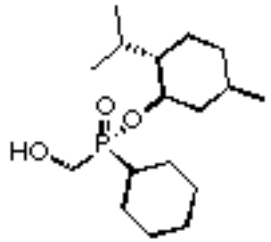


Compound (R_p)-39

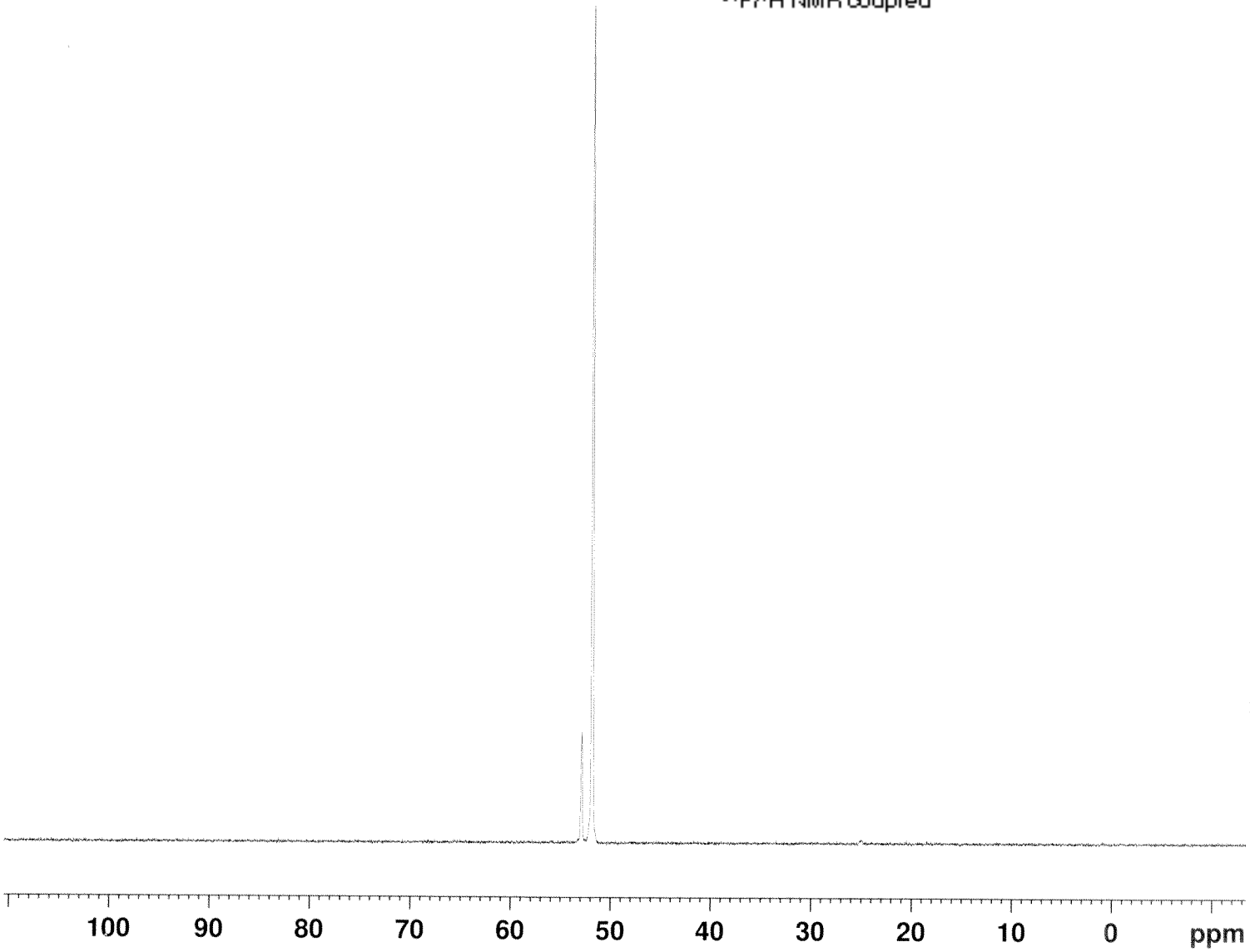
$^{31}P\{^1H\}$ NMR decoupled



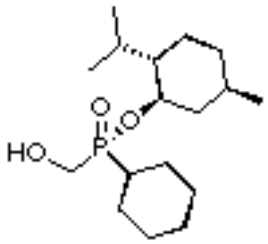

```
Current Data Parameters
NAME         OB 2115 dry
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150716
Time               17.30
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                 7.800 usec
DE                 6.50 usec
TE                295.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1               31P
P1               14.25 usec
PLW1        15.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2            90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                 32768
SF          161.9755930 MHz
WDW                EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



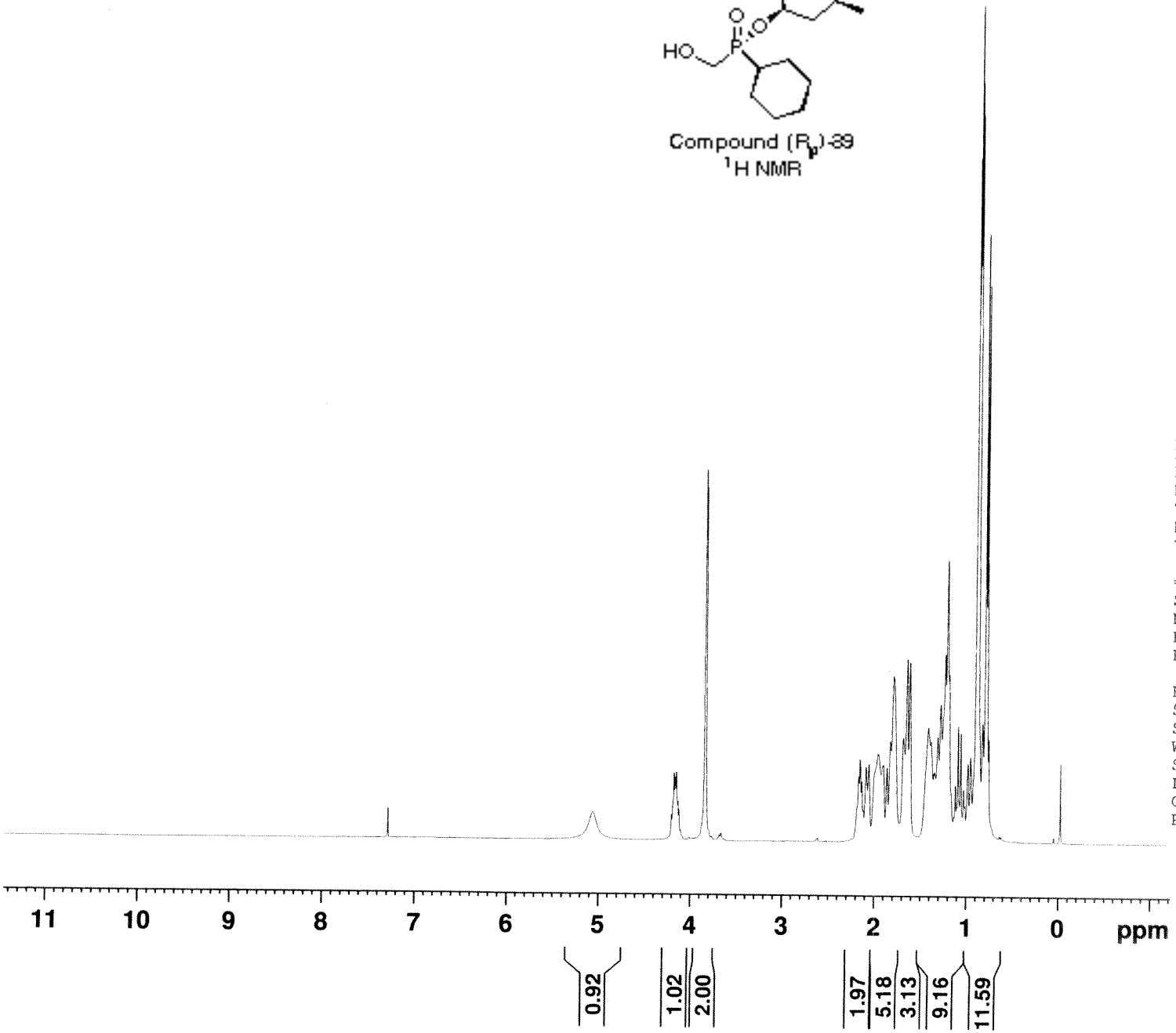
Compound (R_p)-39
$^{31}P\{^1H\}$ NMR coupled



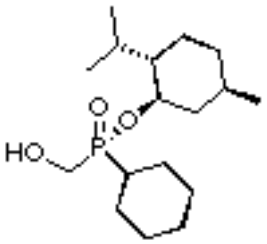

```
Current Data Parameters
NAME         OB 2115 dry
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150716
Time               17.33
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE                295.1 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1               31P
P1               14.25 usec
PLW1        15.00000000 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



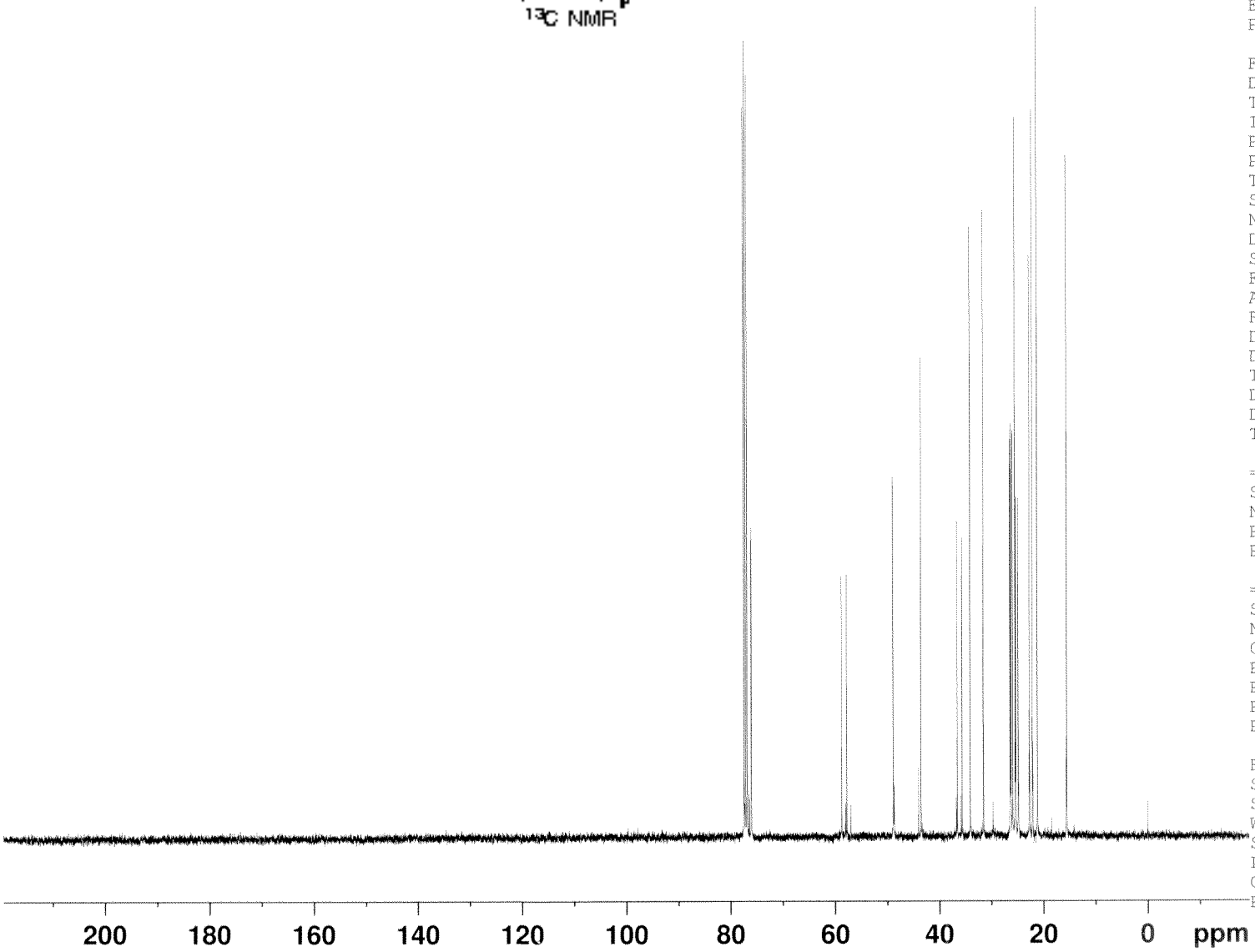
Compound (R_p)-39
^{1}H NMR



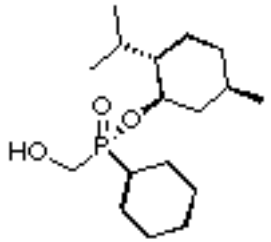

```
Current Data Parameters
NAME          OB 2115 dry
EXPNO                   3
PROCNO                  1

F2 - Acquisition Parameters
Date_            20150716
Time                17.34
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     9
DS                     2
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894465 sec
RG                 16.39
DW                62.400 usec
DE                 6.50 usec
TE                295.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                  1H
P1                10.00 usec
PLW1          25.00300026 W

F2 - Processing parameters
SI                 65536
SF            400.1300000 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```



Compound (R_p)-39
^{13}C NMR




```
Current Data Parameters
NAME         OB 2115 dry
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150716
Time               17.55
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  318
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ            1.3631488 sec
RG               203.57
DW               20.800 usec
DE                6.50 usec
TE                295.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1        45.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                 32768
SF          100.6127685 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

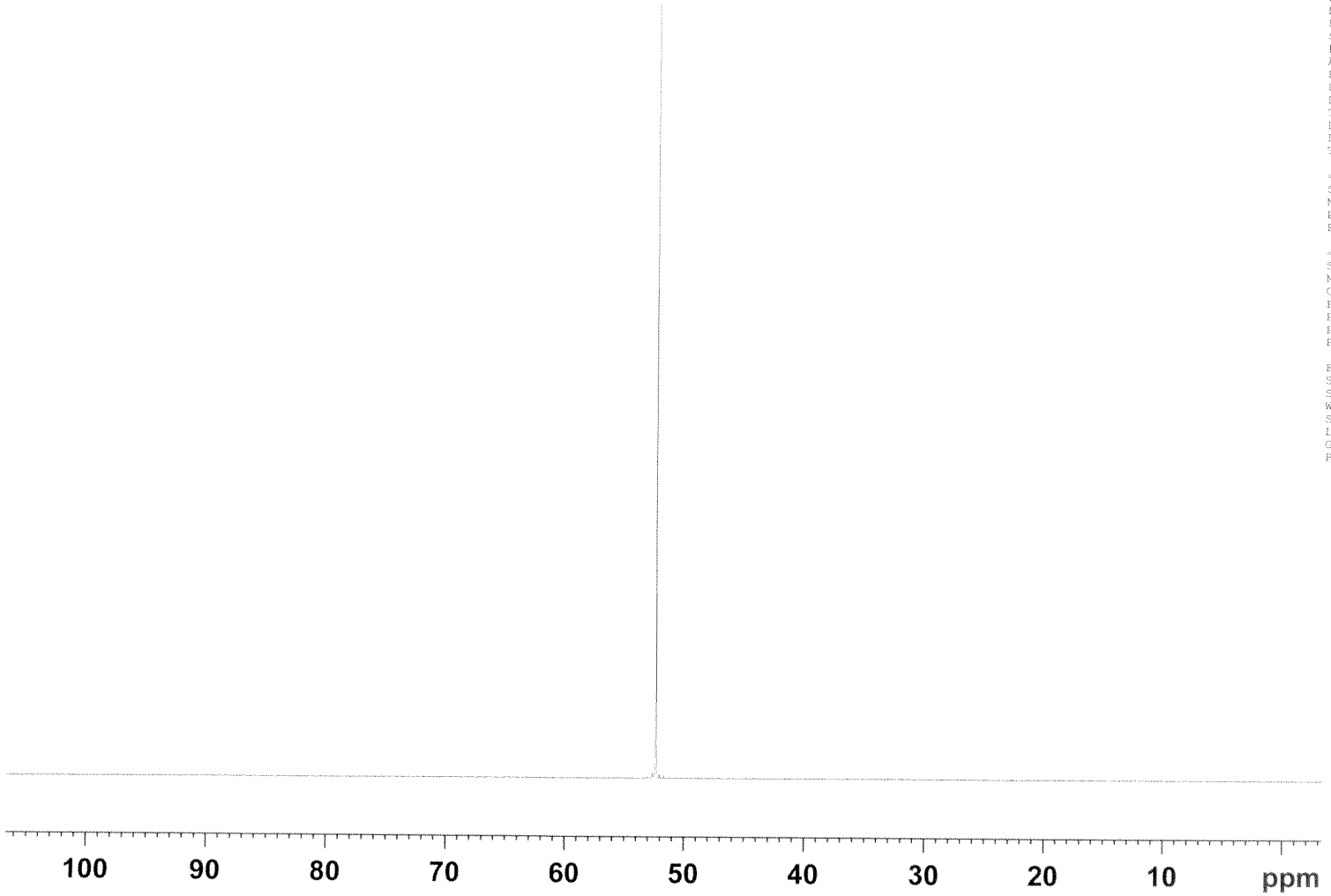


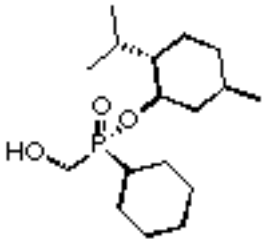
Compound (R_p)-39

$^{31}P\{^{1}H\}$ NMR decoupled

BRUKER

```
Current Data Parameters
NAME      OB 1930 2nd crystallization in CH3CN
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150210
Time              10.27
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE               296.4 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

========== CHANNEL f1 ==========
SFO1        161.9674942 MHz
NUC1               31P
P1                14.25 usec
PLW1        15.00000000 W

========== CHANNEL f2 ==========
SFO2        400.1316005 MHz
NUC2               1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

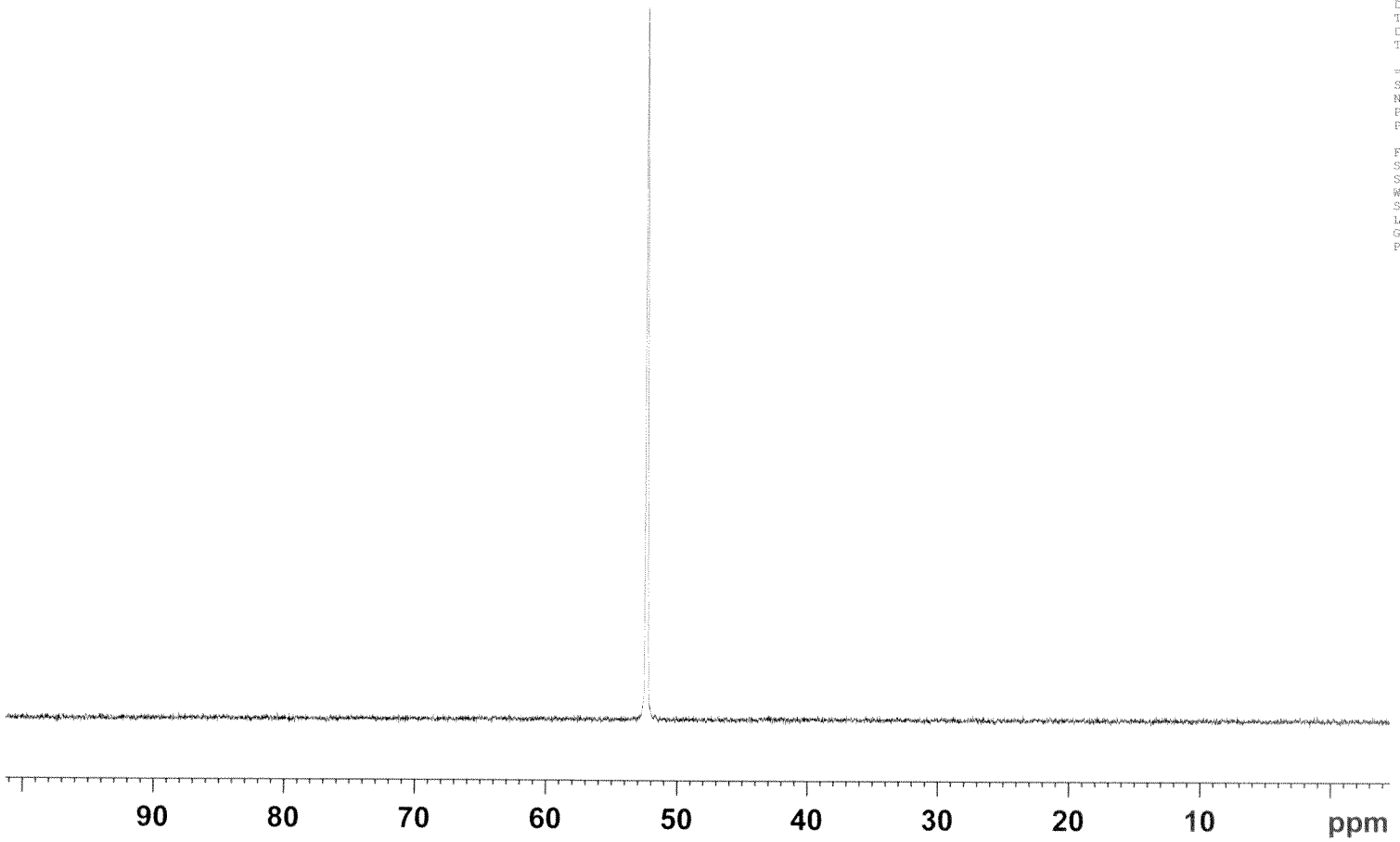


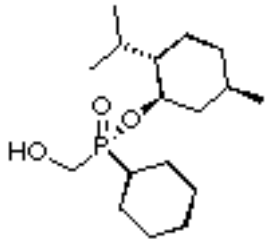


Compound (P_p)-39
$^{31}P\{^{1}H\}$ NMR coupled

BRUKER

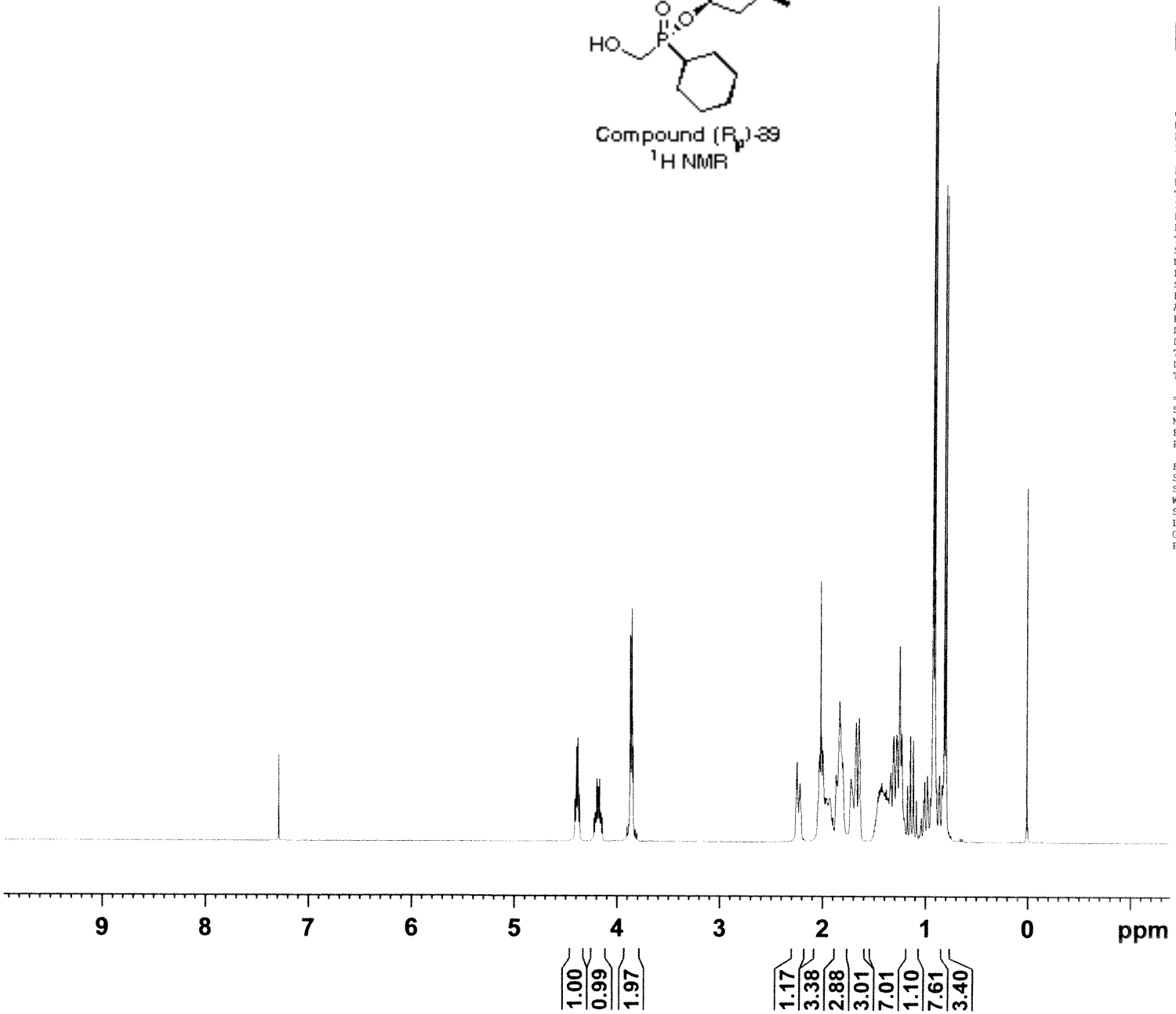
```
Current Data Parameters
NAME      OB 1930 2nd crystallization in CH3CN
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20150210
Time              10.29
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  32
DS                   4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                7.800 usec
DE                6.50 usec
TE                296.1 K
D1           2.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1               31P
P1               14.25 usec
PLW1        15.00000000 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



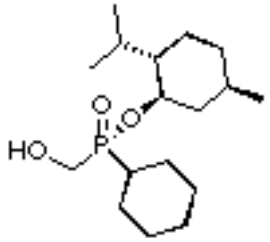


Compound (R_p)-39

^{1}H NMR

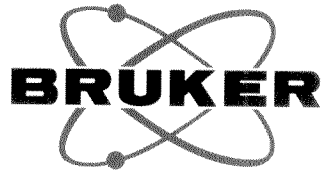



```
Current Data Parameters
NAME      OB 1930 2nd crystallization in CH3CN
EXPNO                 3
PROCNO                1

F2 - Acquisition Parameters
Date_          20150210
Time              10.32
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  10
DS                   2
SWH            8012.820 Hz
FIDRES            0.122266 Hz
AQ             4.0894465 sec
RG                32.38
DW                62.400 usec
DE                 6.50 usec
TE                296.1 K
D1             1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1               1H
P1                10.00 usec
PLW1         25.00300026 W

F2 - Processing parameters
SI                65536
SF           400.1300000 MHz
WDW                EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

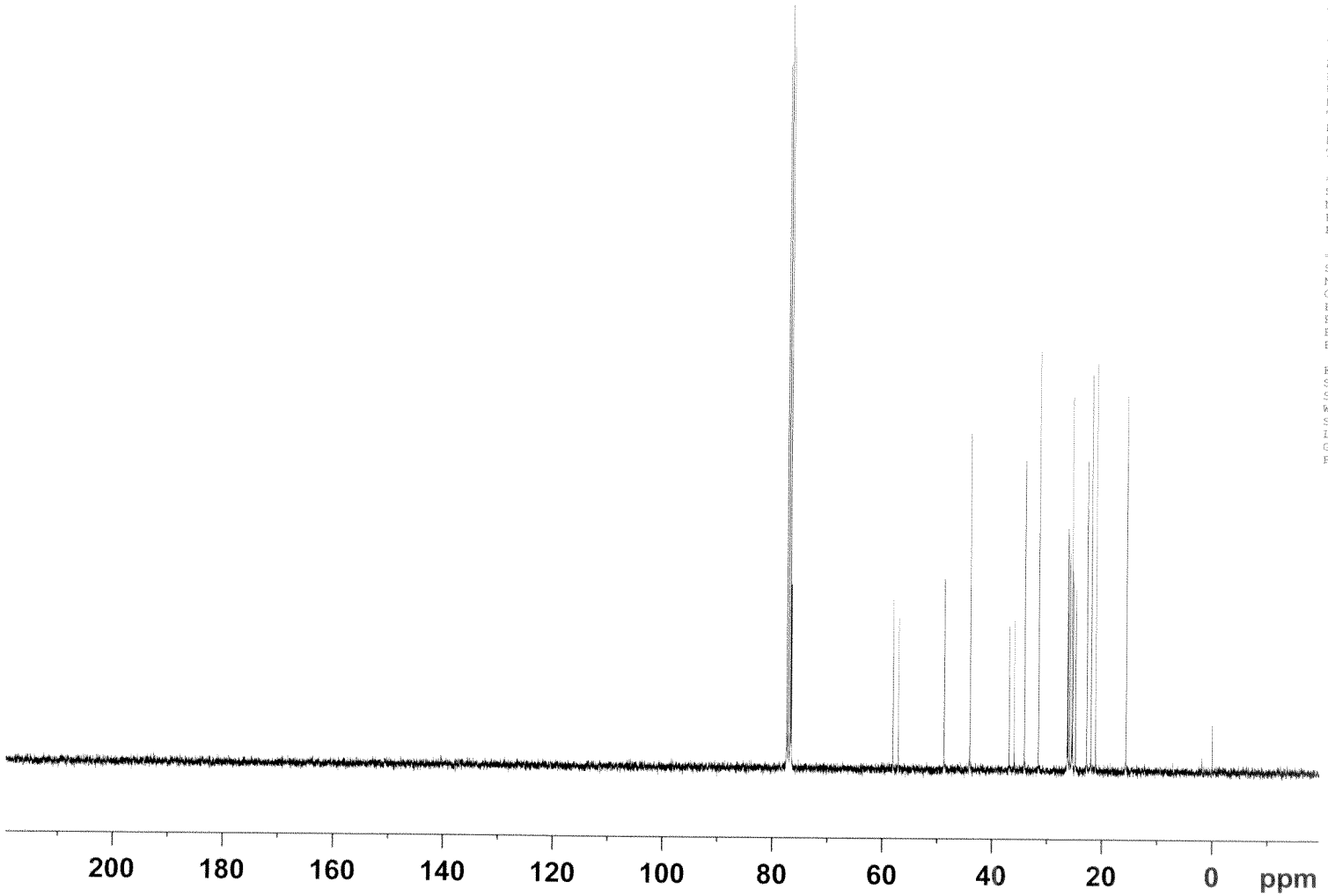


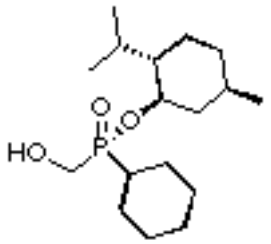
Compound (R_p)-39

^{13}C NMR




```
Current Data Parameters
NAME      OB 1930 2nd crystallization in CH3CN
EXPNO                 4
PROCNO                1

F2 - Acquisition Parameters
Date_          20150210
Time              10.56
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                 383
DS                   4
SWH          24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631488 sec
RG               203.57
DW               20.800 usec
DE                6.50 usec
TE               297.5 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1       100.6228293 MHz
NUC1              13C
P1              10.00 usec
PLW1        45.00000000 W

======== CHANNEL f2 ========
SFO2       400.1316005 MHz
NUC2              1H
CPDPRG[2        waltz16
PCPD2             90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                32768
SF          100.6127685 MHz
WDW                EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

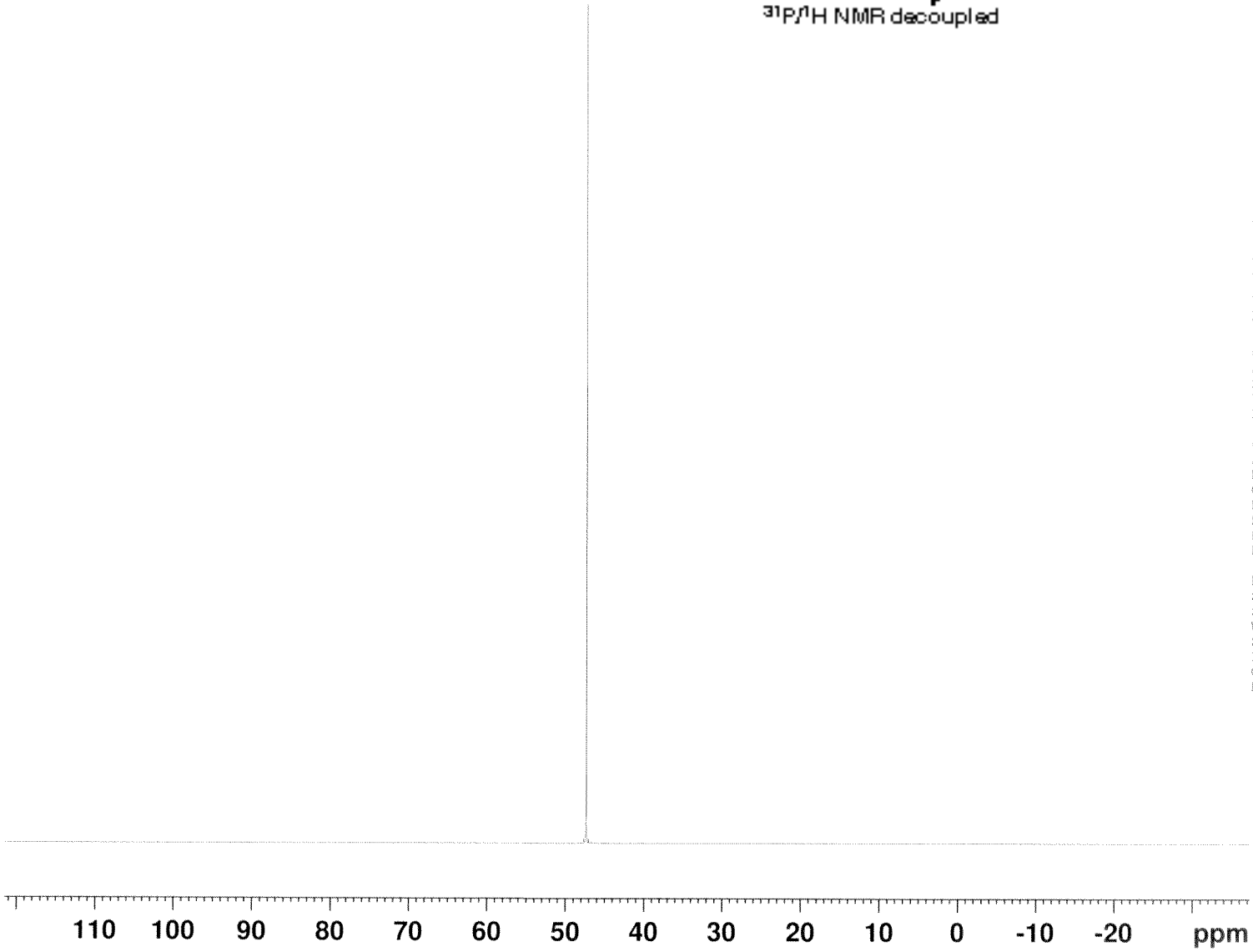


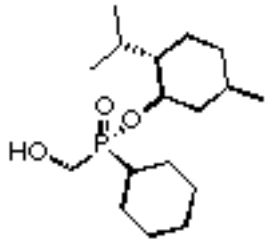


Compound (R_p)-39

$^{31}P\{^1H\}$ NMR decoupled

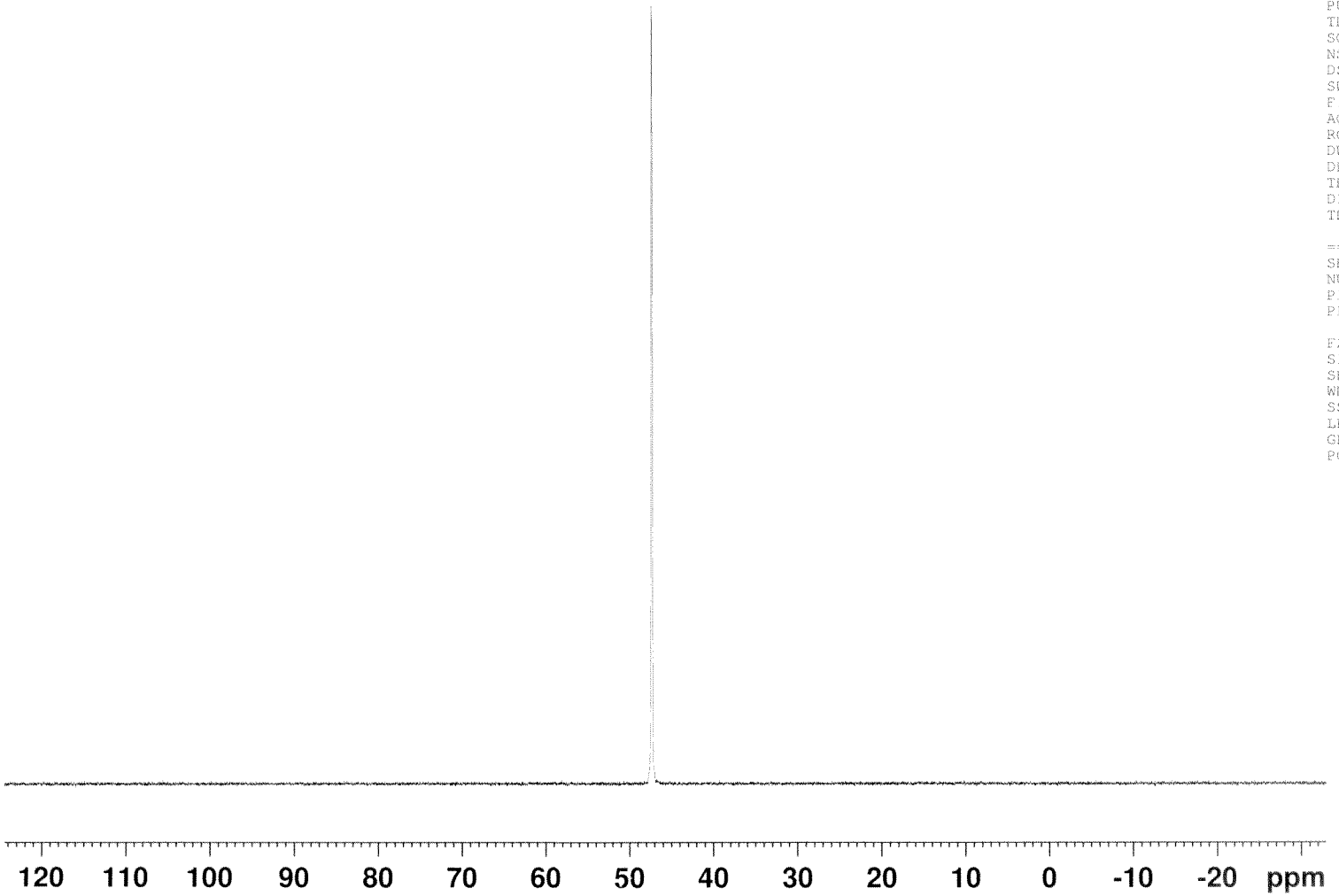
```
Current Data Parameters
NAME      OB 2148 4th crystallization
EXPNO                 1
PROCNO                1

F2 - Acquisition Parameters
Date_          20150902
Time              17.35
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT         Acetone
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE               295.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1        15.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2        10.00000000 W
PLW12        0.31604999 W
PLW13        0.25600001 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

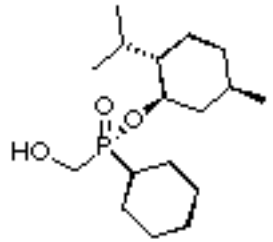




Compound (R_p)-39
$^{31}P\{^1H\}$ NMR coupled

```
Current Data Parameters
NAME      OB 2148 4th crystallization
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20150902
Time              17.37
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT         Acetone
NS                   32
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ             0.5111808 sec
RG                203.57
DW                7.800 usec
DE                6.50 usec
TE                294.7 K
D1            2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1                31P
P1                14.25 usec
PLW1        15.00000000 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```

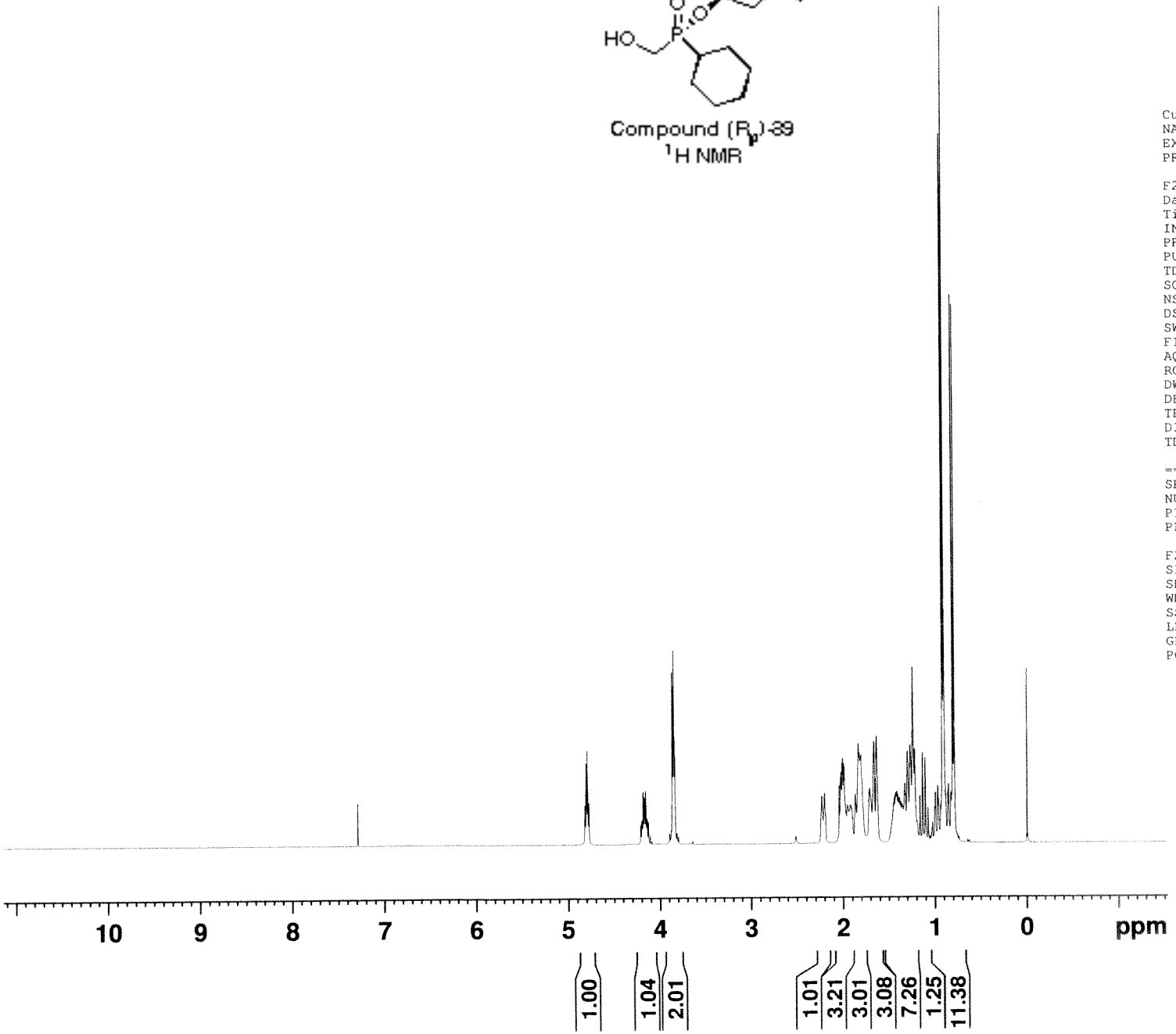


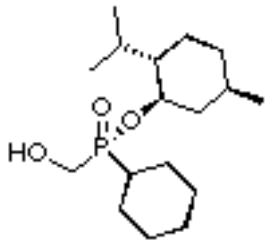


Compound (R_p)-89

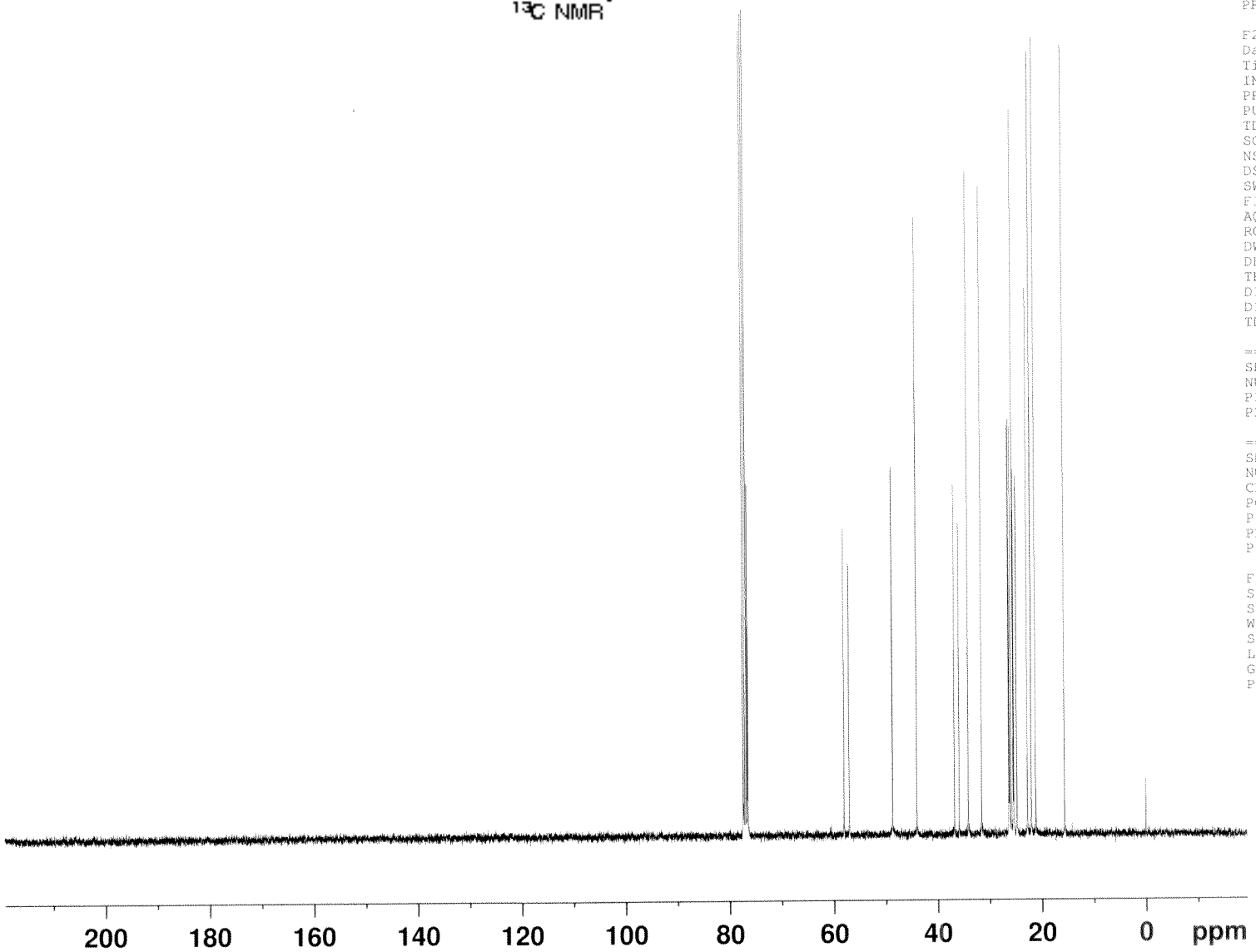
^{1}H NMR

```
Current Data Parameters
NAME      OB 2148 4th crystallization
EXPNO                3
PROCNO               1

F2 - Acquisition Parameters
Date_          20150902
Time              17.40
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                  14
DS                   2
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894465 sec
RG                32.38
DW               62.400 usec
DE                6.50 usec
TE               294.6 K
D1            1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1         400.1324710 MHz
NUC1               1H
P1               10.00 usec
PLW1         25.00300026 W

F2 - Processing parameters
SI                65536
SF           400.1300000 MHz
WDW                EM
SSB       0
LB               0.30 Hz
GB        0
PC               1.00
```





Compound (R_p)-39
^{13}C NMR



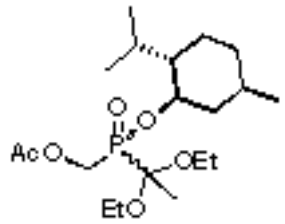

```
Current Data Parameters
NAME      OB 2148 4th crystallization
EXPNO                 4
PROCNO                1

F2 - Acquisition Parameters
Date_          20150903
Time              17.00
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD              65536
SOLVENT           CDCl3
NS                  442
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                295.6 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                13C
P1                10.00 usec
PLW1         45.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         10.00000000 W
PLW12         0.31604999 W
PLW13         0.25600001 W

F2 - Processing parameters
SI                32768
SF           100.6127685 MHz
WDW                 EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```

1056

OB 1056f1

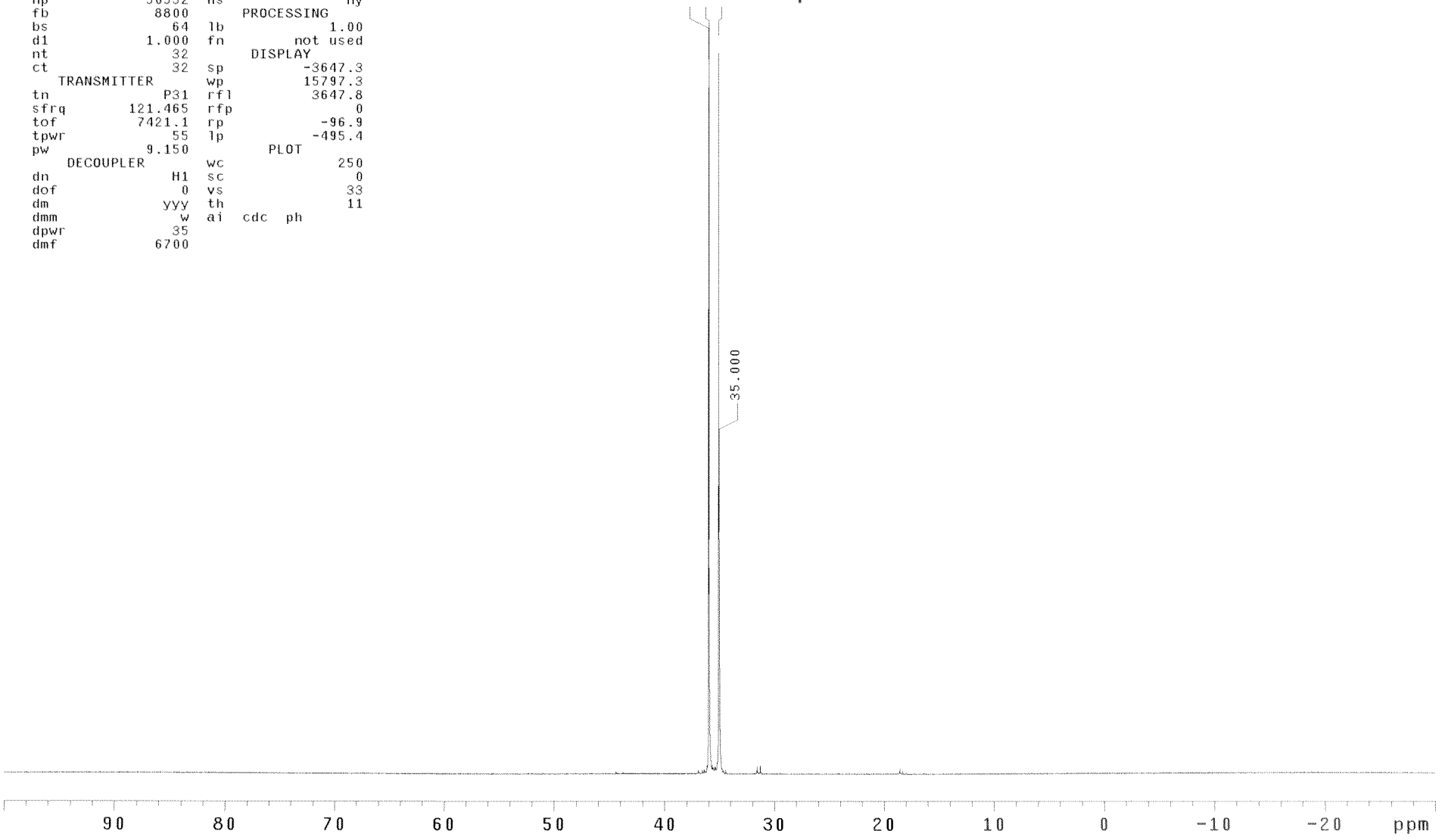
exp1 Phosphorus

SAMPLE		SPECIAL	
date	Apr 9 2013	temp	not used
solvent	cdcl3	gain	8
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2013.04.06/s_2013~		pw90	18.300
0409_41/data/cdcl3~		alfa	10.000
04.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	32	DISPLAY	
ct	32	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	-96.9
tpwr	55	lp	-495.4
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	33
dm	yyy	th	11
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



Compound 40

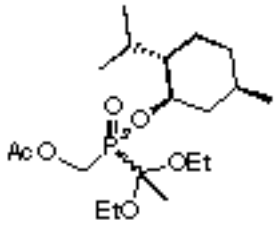
$^{31}P\{^{1}H\}$ NMR decoupled



OB 1055

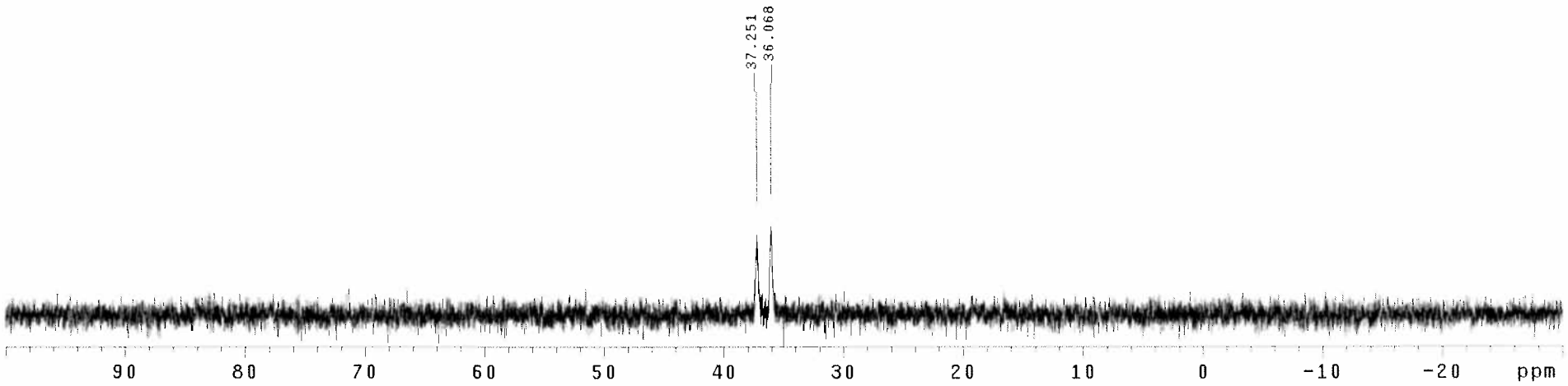
exp1 Phosphorus

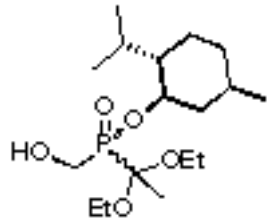
SAMPLE		SPECIAL	
date	Apr 9 2013	temp	not used
solvent	cdcl3	gain	25
file	/home/TCUuser~	spin	20
	/vnmrsys/data/auto~	hst	0.008
	_2013.04.06/s_2013~	pw90	18.300
	0409_22/data/cdcl3~	alfa	10.000
	_02.fid	FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	64	DISPLAY	
ct	64	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	27.0
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	344
dm	ynn	th	7
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



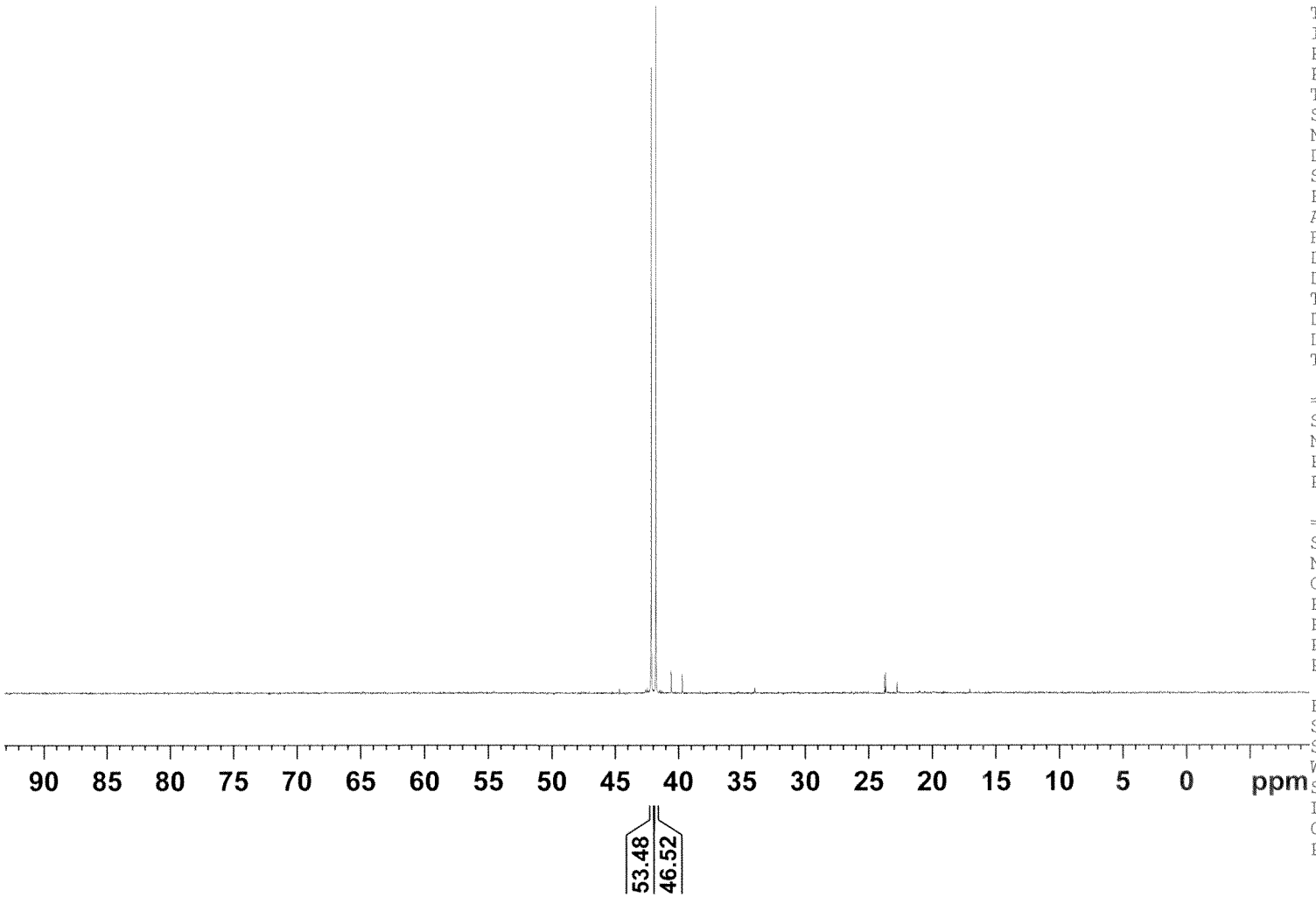
Compound 40

$^{31}P\{^{1}H\}$ NMR coupled

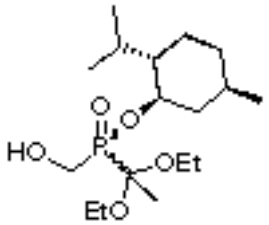




Compound 41
$^{31}P\{^{1}H\}$ NMR decoupled




```
Current Data Parameters
NAME                  OB 1592
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_                20140722
Time                     9.33
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT               Acetone
NS                         16
DS                          4
SWH                 64102.563 Hz
FIDRES               0.978127 Hz
AQ                   0.5111808 sec
RG                      203.57
DW                       7.800 usec
DE                       6.50 usec
TE                      295.5 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         1

========== CHANNEL f1 ==========
SFO1               161.9674942 MHz
NUC1                       31P
P1                       14.25 usec
PLW1               15.00000000 W

========== CHANNEL f2 ==========
SFO2               400.1316005 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2               10.00000000 W
PLW12               0.31604999 W
PLW13               0.25600001 W

F2 - Processing parameters
SI                         32768
SF                 161.9755930 MHz
WDW                           EM
SSB                0
LB                       1.00 Hz
GB                 0
PC                       1.40
```

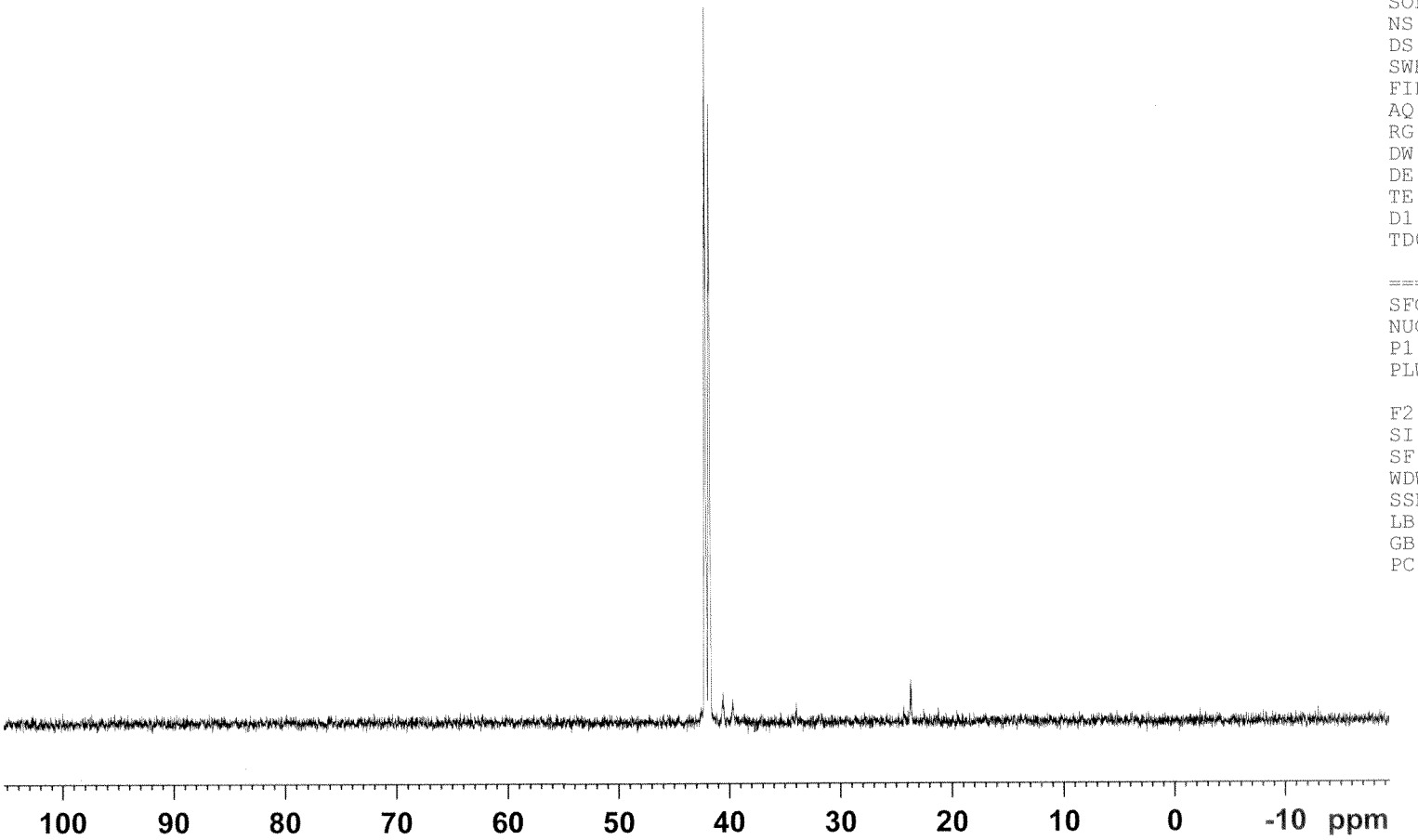


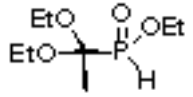
Compound 41

$^{31}P\{^{1}H\}$ NMR coupled

BRUKER

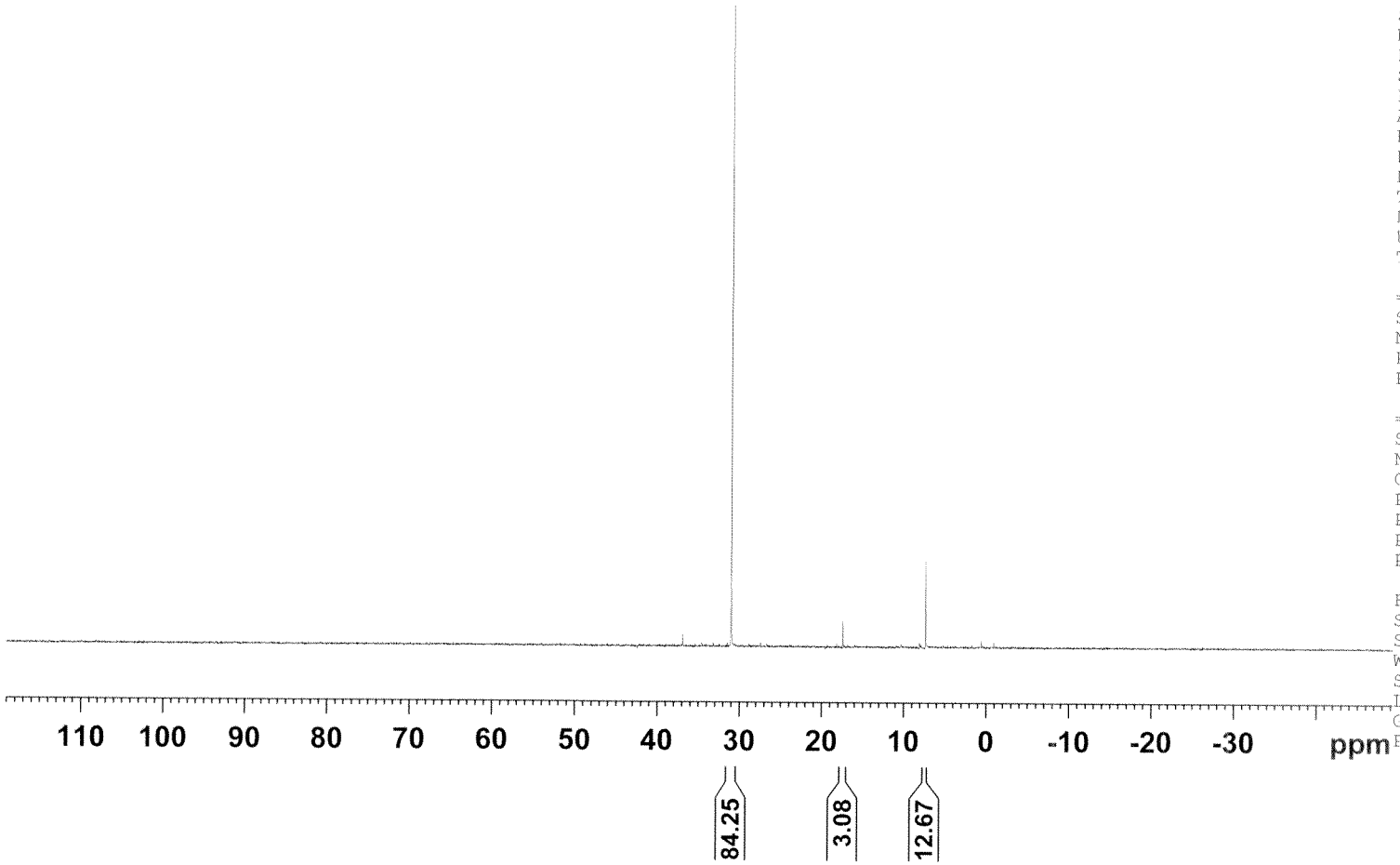
```
Current Data Parameters
NAME                  OB 1592
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20140722
Time                   9.35
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT             Acetone
NS                         32
DS                          4
SWH               64102.563 Hz
FIDRES             0.978127 Hz
AQ                 0.5111808 sec
RG                    203.57
DW                   7.800 usec
DE                   6.50 usec
TE                   295.2 K
D1               2.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1            161.9674942 MHz
NUC1                     31P
P1                   14.25 usec
PLW1            15.00000000 W

F2 - Processing parameters
SI                      32768
SF              161.9755930 MHz
WDW                        EM
SSB         0
LB                    1.00 Hz
GB          0
PC                    1.40
```



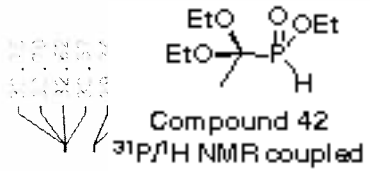


Compound 42

$^{31}P\{^{1}H\}$ NMR decoupled

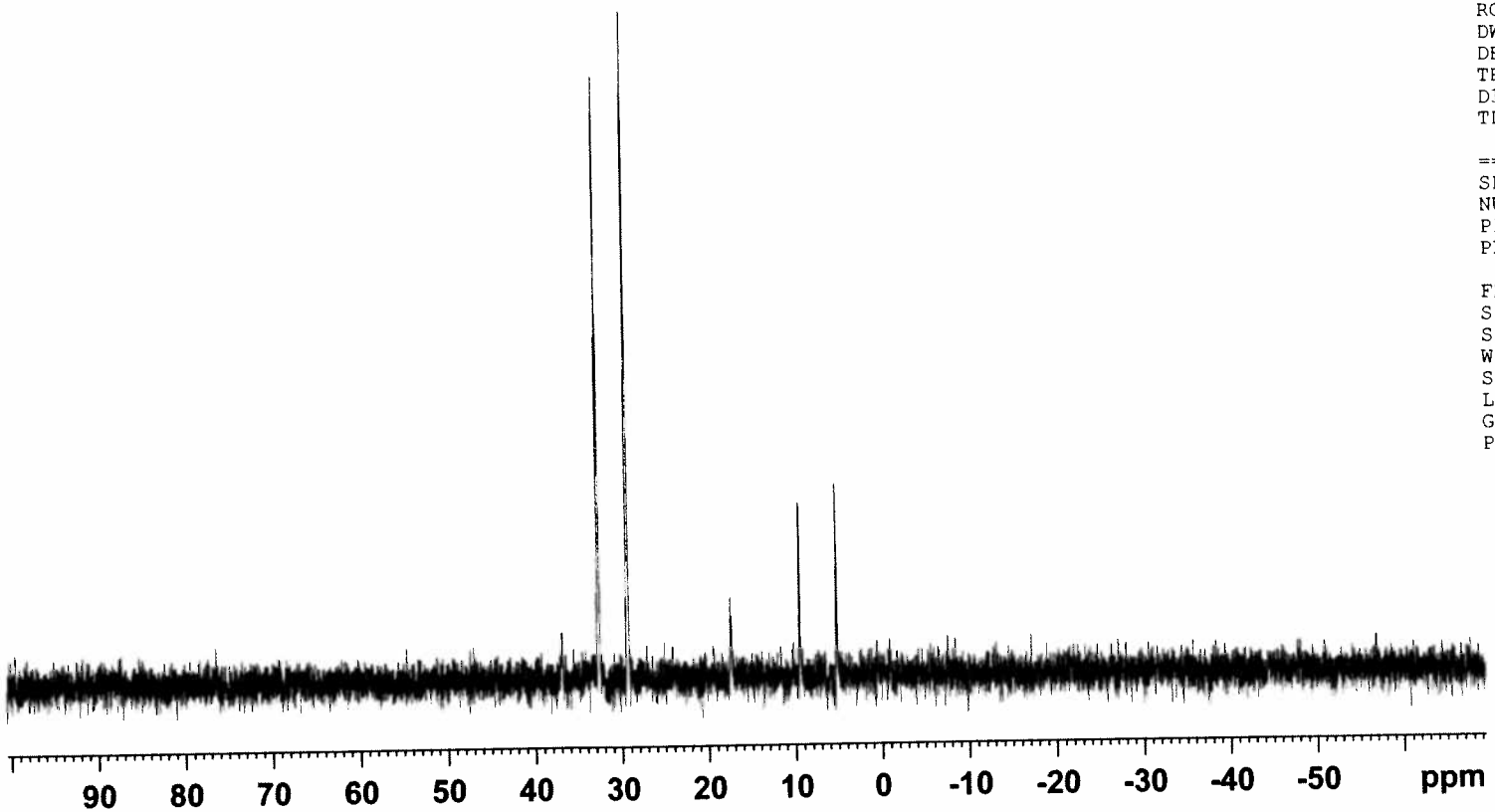



```
Current Data Parameters
NAME      OB 1725 after work-up
EXPNO                        5
PROCNO                       1

F2 - Acquisition Parameters
Date_              20140917
Time                  12.01
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        4
SWH               64102.563 Hz
FIDRES             0.978127 Hz
AQ                 0.5111808 sec
RG                    203.57
DW                   7.800 usec
DE                   6.50 usec
TE                   296.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                          1

========== CHANNEL f1 ==========
SFO1            161.9836917 MHz
NUC1                      31P
P1                    14.25 usec
PLW1           15.00000000 W

========== CHANNEL f2 ==========
SFO2            400.1316005 MHz
NUC2                      1H
CPDPRG[2             waltz16
PCPD2                 90.00 usec
PLW2           10.00000000 W
PLW12           0.31604999 W
PLW13           0.25600001 W

F2 - Processing parameters
SI                       32768
SF              161.9755930 MHz
WDW                         EM
SSB          0
LB                     1.00 Hz
GB           0
PC                     1.40
```



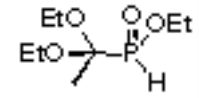
Compound 42

$^{31}P\{^{1}H\}$ NMR coupled

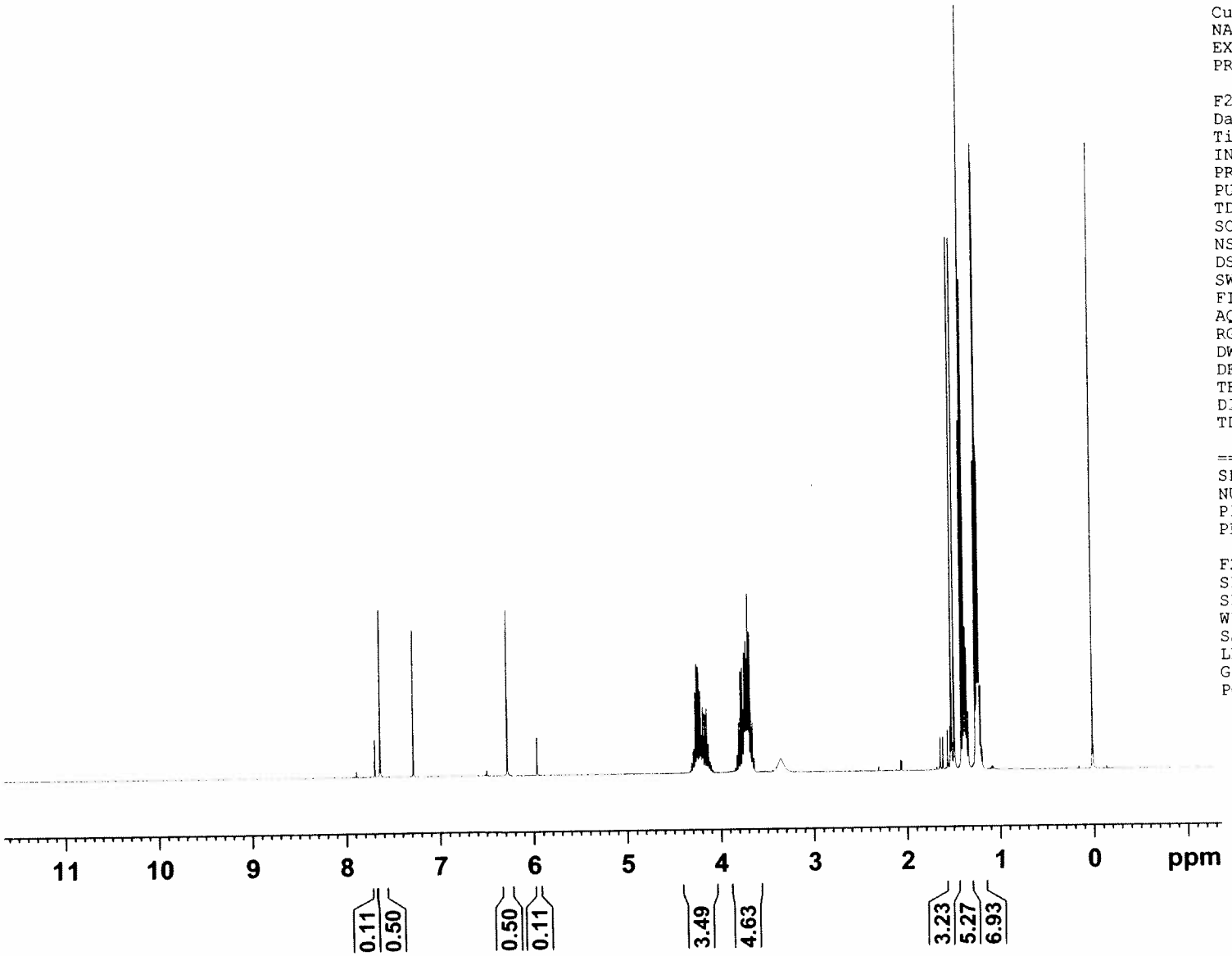


BRUKER

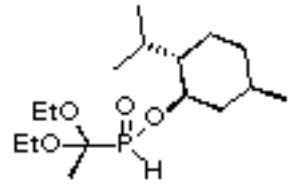
```
Current Data Parameters
NAME      OB 1725 after work-up
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_              20140917
Time_                 12.02
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      13
DS                       4
SWH               64102.563 Hz
FIDRES             0.978127 Hz
AQ                 0.5111808 sec
RG                    203.57
DW                   7.800 usec
DE                   6.50 usec
TE                   295.7 K
D1                2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           161.9674942 MHz
NUC1                   31P
P1                   14.25 usec
PLW1          15.00000000 W

F2 - Processing parameters
SI                   32768
SF             161.9755930 MHz
WDW                     EM
SSB       0
LB                   1.00 Hz
GB        0
PC                   1.40
```



Compound 42
^{1}H NMR

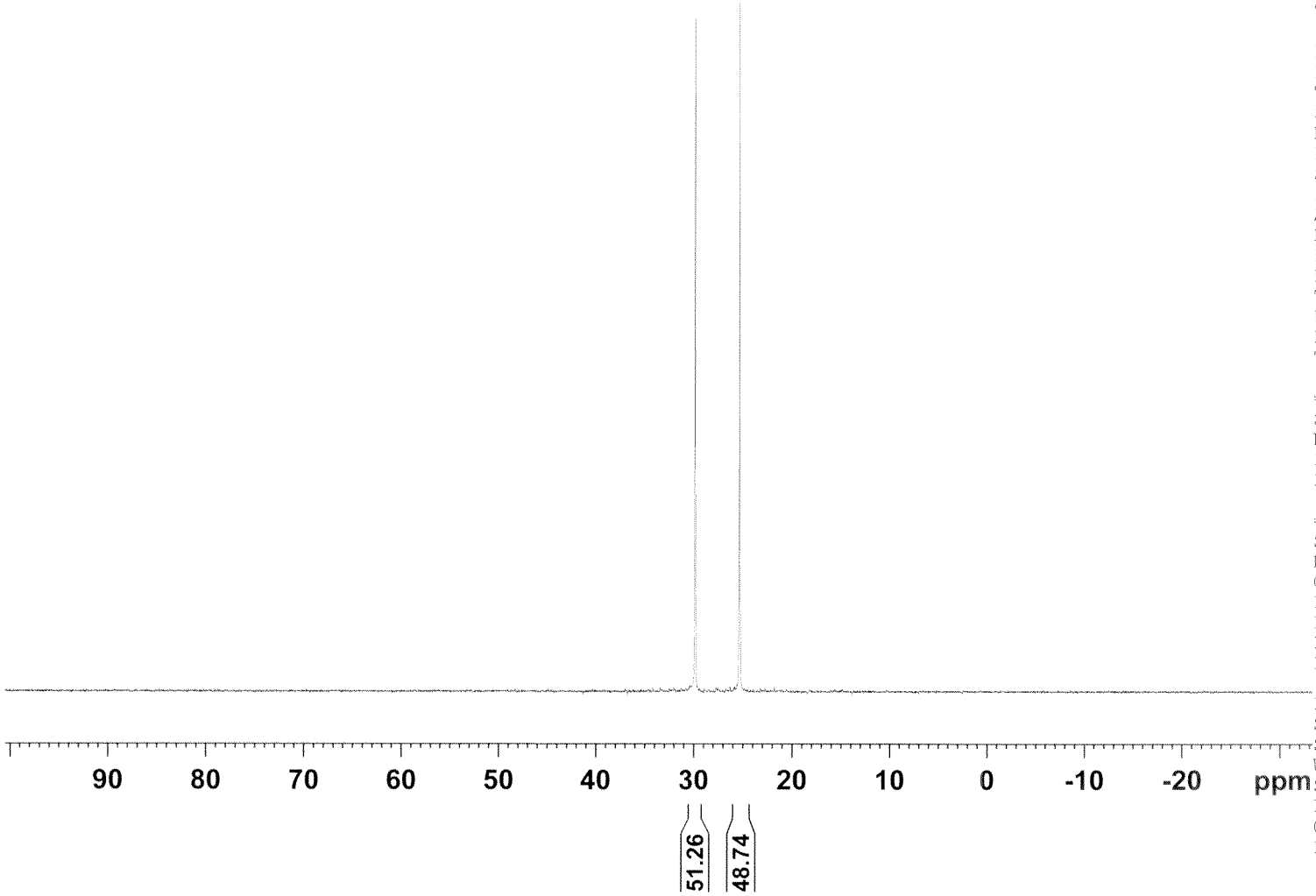



```
Current Data Parameters
NAME      OB 1725 after work-up
EXPNO                         3
PROCNO                        1

F2 - Acquisition Parameters
Date_              20140917
Time                  12.05
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       2
SWH               8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894465 sec
RG                  113.32
DW                  62.400 usec
DE                    6.50 usec
TE                   295.7 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          400.1324710 MHz
NUC1                    1H
P1                   10.00 usec
PLW1         25.00300026 W

F2 - Processing parameters
SI                   65536
SF            400.1300000 MHz
WDW                     EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```

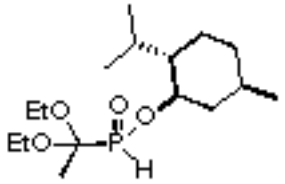


Compound 43

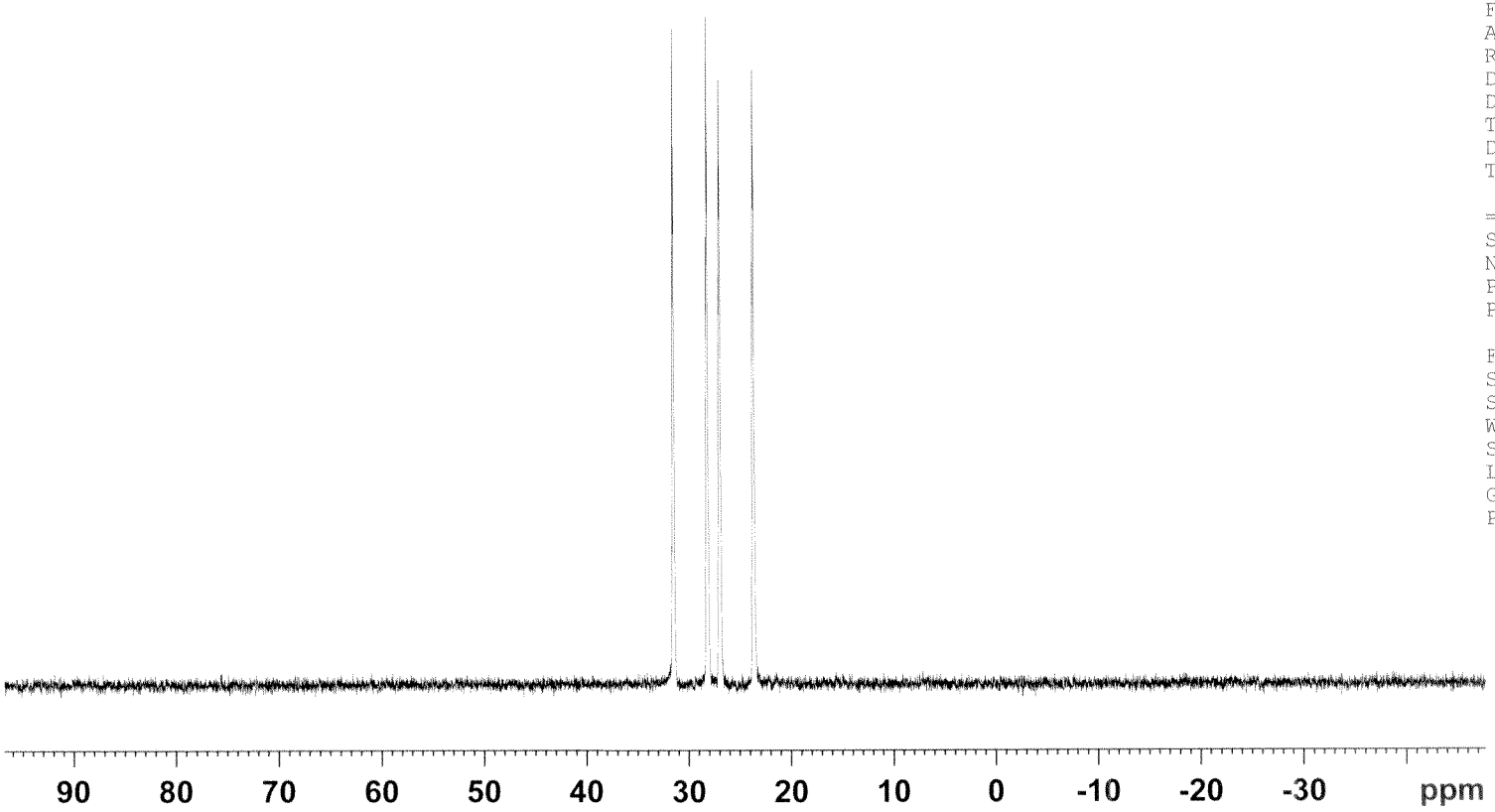
$^{31}P\{^{1}H\}$ NMR decoupled




```
Current Data Parameters
NAME                  OB 1701f2
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20140910
Time                       9.04
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                    DMSO
NS                           16
DS                            4
SWH                  64102.563 Hz
FIDRES                0.978127 Hz
AQ                    0.5111808 sec
RG                       203.57
DW                        7.800 usec
DE                        6.50 usec
TE                       295.6 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1               161.9674942 MHz
NUC1                        31P
P1                        14.25 usec
PLW1                15.00000000 W

======== CHANNEL f2 ========
SFO2               400.1316005 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                     90.00 usec
PLW2                10.00000000 W
PLW12                0.31604999 W
PLW13                0.25600001 W

F2 - Processing parameters
SI                        32768
SF                 161.9755930 MHz
WDW                          EM
SSB                 0
LB                        1.00 Hz
GB                  0
PC                        1.40
```



Compound 43
$^{31}P\{^{1}H\}$ NMR coupled

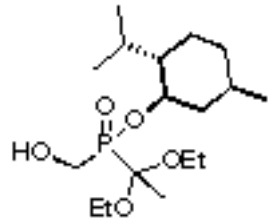



```
Current Data Parameters
NAME                  OB 1701f2
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20140910
Time                       9.06
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                    zg30
TD                        65536
SOLVENT                    DMSO
NS                           32
DS                            4
SWH                   64102.563 Hz
FIDRES                 0.978127 Hz
AQ                     0.5111808 sec
RG                        203.57
DW                         7.800 usec
DE                         6.50 usec
TE                        295.3 K
D1                    2.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                161.9674942 MHz
NUC1                        31P
P1                        14.25 usec
PLW1                15.00000000 W

F2 - Processing parameters
SI                        32768
SF                  161.9755930 MHz
WDW                          EM
SSB                0
LB                        1.00 Hz
GB                 0
PC                        1.40
```

OB 1037

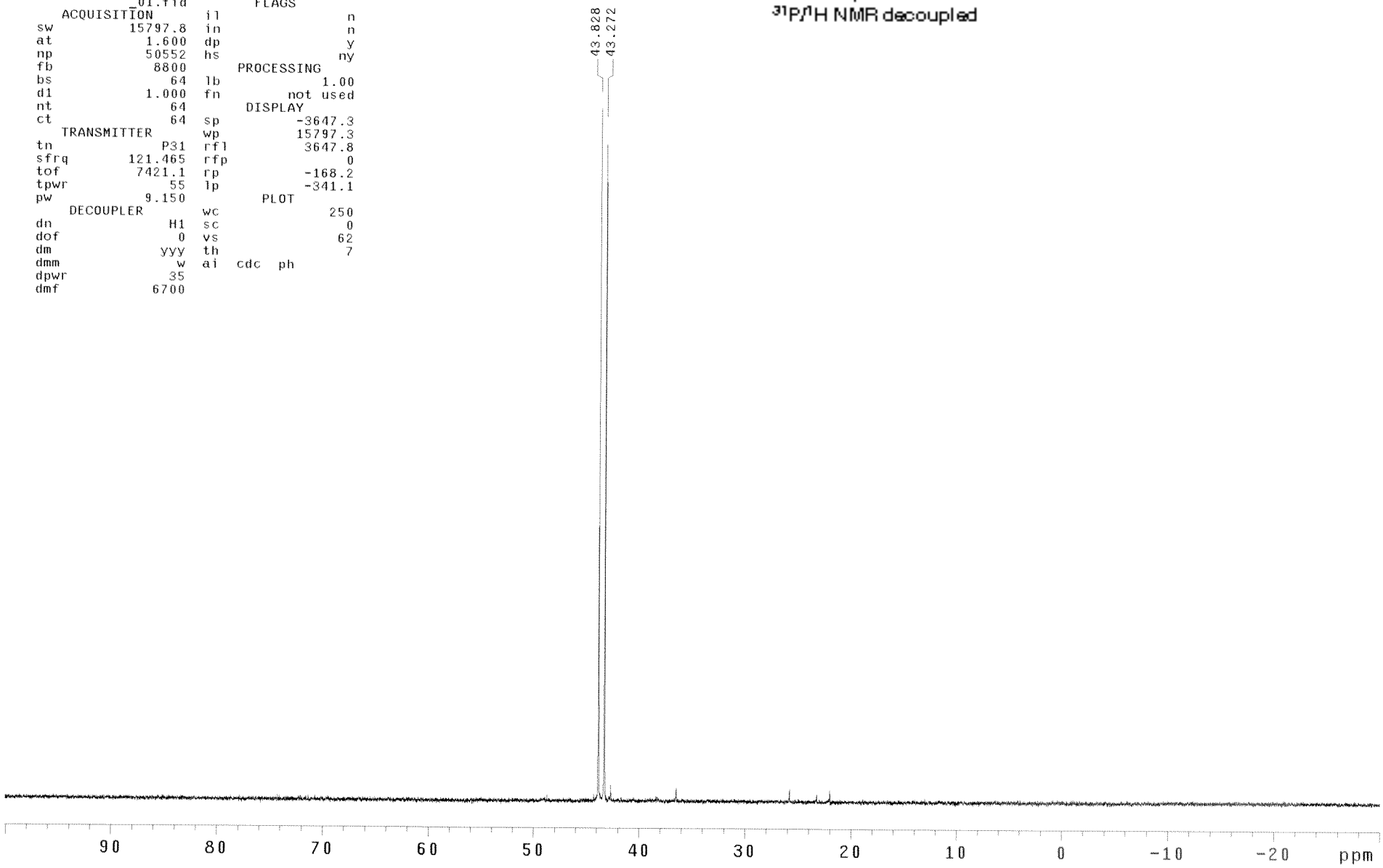
exp1 Phosphorus

SAMPLE		SPECIAL	
date	Mar 26 2013	temp	not used
solvent	cdcl3	gain	25
file	/home/TCUuser~	spin	20
	/vnmrsys/data/auto~	hst	0.008
	_2013.03.09/s_2013~	pw90	18.300
	0326_36/data/cdcl3~	alfa	10.000
	_01.fid	FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	64	DISPLAY	
ct	64	sp	-3647.3
TRANSMITTER		wp	15797.3
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	-168.2
tpwr	55	lp	-341.1
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	62
dm	yyy	th	7
dmm	w	ai	cdc ph
dpwr	35		
dmf	6700		



Compound 41

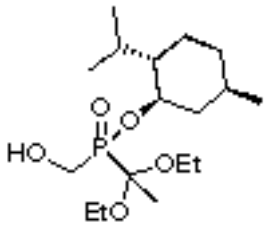
$^{31}P\{^{1}H\}$ NMR decoupled



OB 1037

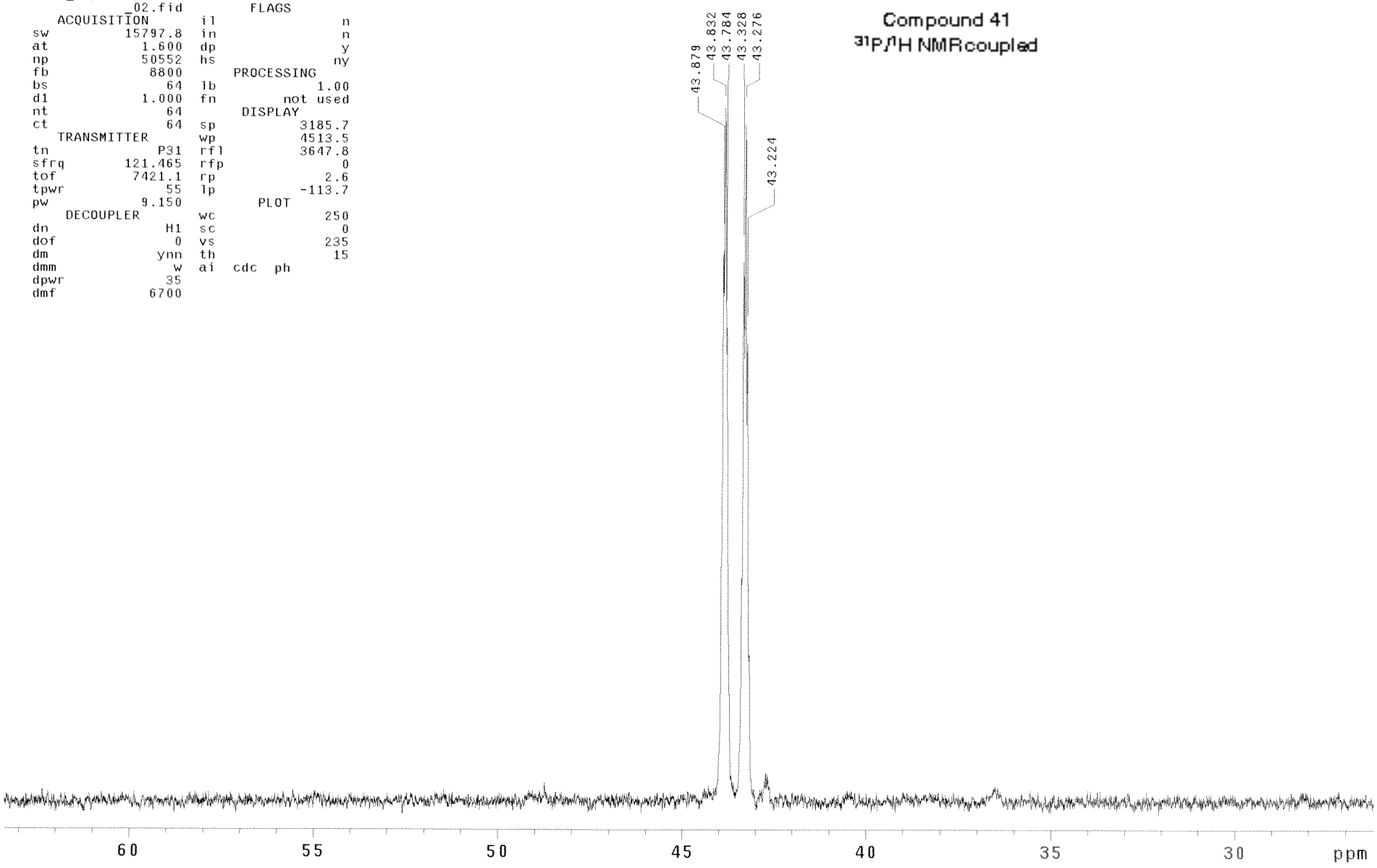
exp1 Phosphorus

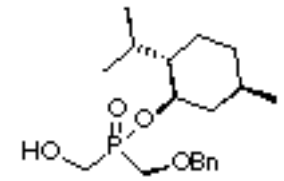
SAMPLE		SPECIAL	
date	Mar 26 2013	temp	not used
solvent	cdcl3	gain	25
file	/home/TCUuser~	spin	20
/vnmrsys/data/auto~		hst	0.008
_2013.03.09/s_2013~		pw90	18.300
0326_36/data/cdcl3~		alfa	10.000
02.fid		FLAGS	
ACQUISITION		il	n
sw	15797.8	in	n
at	1.600	dp	y
np	50552	hs	ny
fb	8800	PROCESSING	
bs	64	lb	1.00
d1	1.000	fn	not used
nt	64	DISPLAY	
ct	64	sp	3185.7
TRANSMITTER		wp	4513.5
tn	P31	rfl	3647.8
sfrq	121.465	rfp	0
tof	7421.1	rp	2.6
tpwr	55	lp	-113.7
pw	9.150	PLOT	
DECOUPLER		wc	250
dn	H1	sc	0
dof	0	vs	235
dm	ynn	th	15
dmm	w	ai cdc ph	
dpwr	35		
dmf	6700		



Compound 41

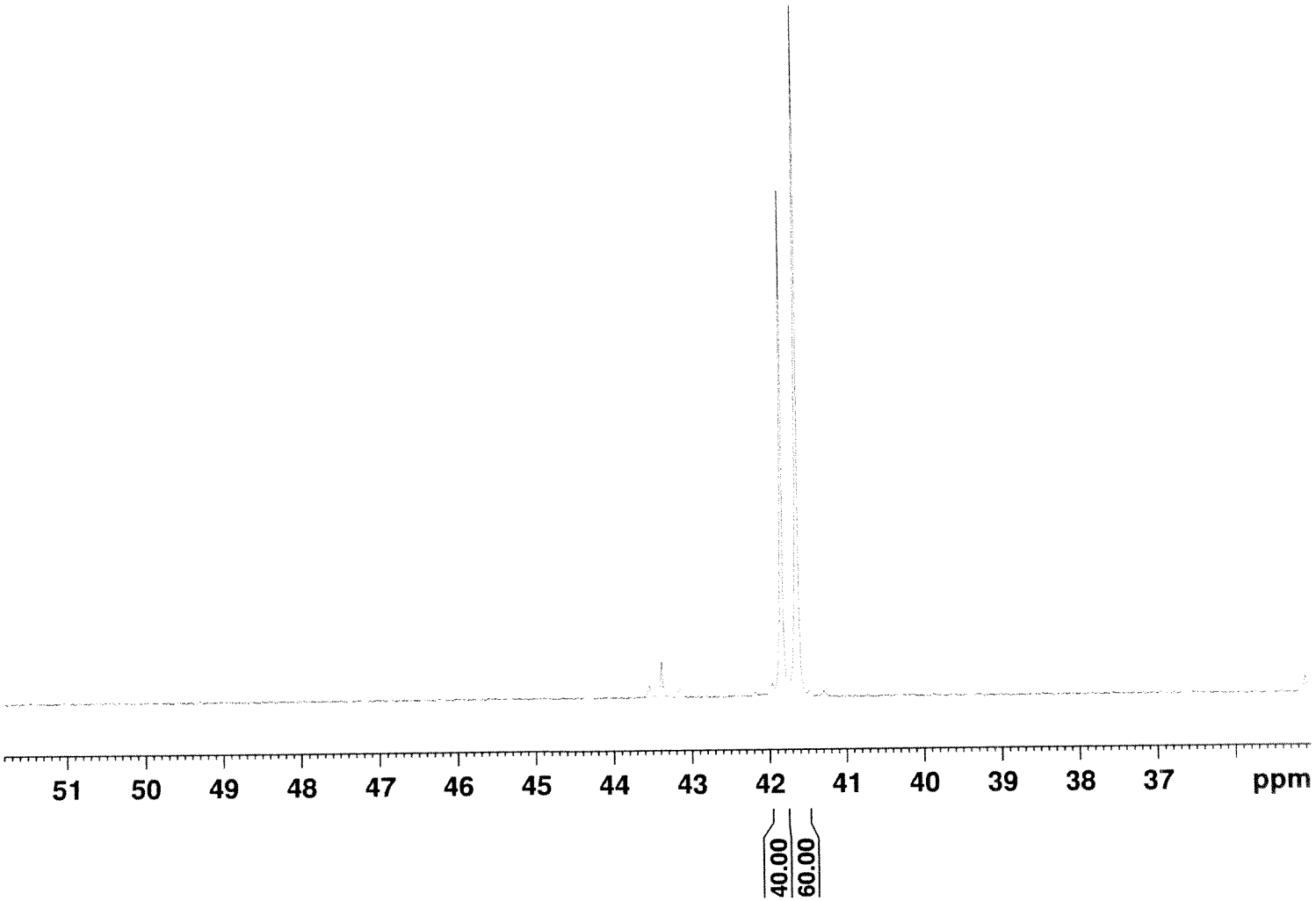
$^{31}P\{^{1}H\}$ NMR coupled



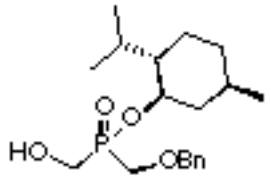


Compound 44

$^{31}P\{^{1}H\}$ NMR decoupled

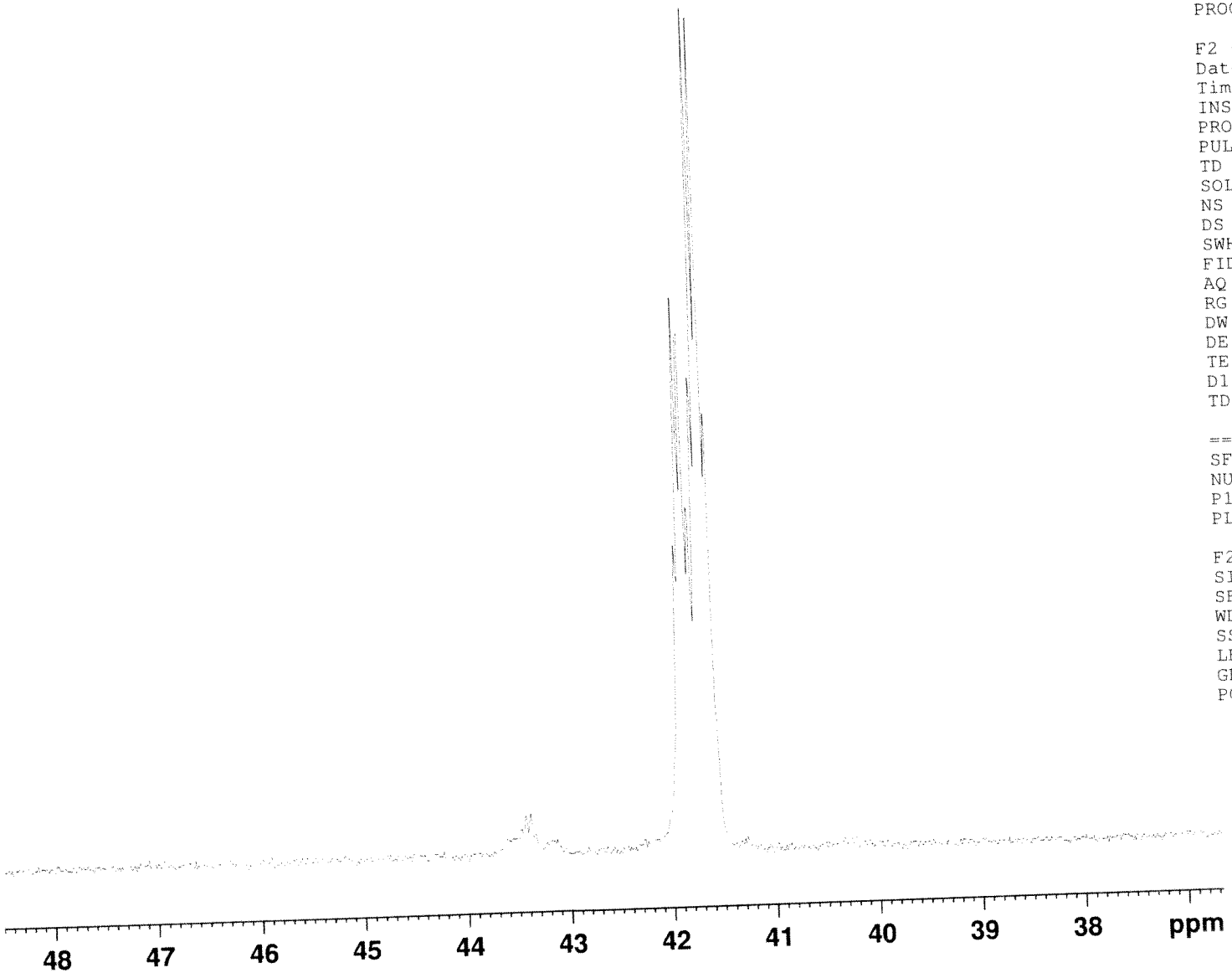



```
Current Data Parameters
NAME                  OB 2110
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_                20150709
Time                     9.23
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                Acetone
NS                          16
DS                           4
SWH                 64102.563 Hz
FIDRES               0.978127 Hz
AQ                   0.5111808 sec
RG                      203.57
DW                       7.800 usec
DE                       6.50 usec
TE                      294.3 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             161.9674942 MHz
NUC1                      31P
P1                      14.25 usec
PLW1             15.00000000 W

======== CHANNEL f2 ========
SFO2             400.1316005 MHz
NUC2                      1H
CPDPRG[2              waltz16
PCPD2                   90.00 usec
PLW2             10.00000000 W
PLW12             0.31604999 W
PLW13             0.25600001 W

F2 - Processing parameters
SI                       32768
SF               161.9755930 MHz
WDW                         EM
SSB          0
LB                       1.00 Hz
GB           0
PC                       1.40
```

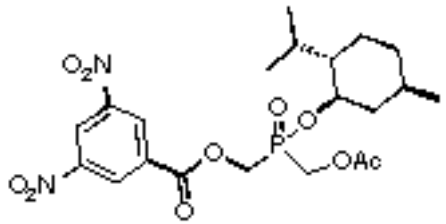


Compound 44

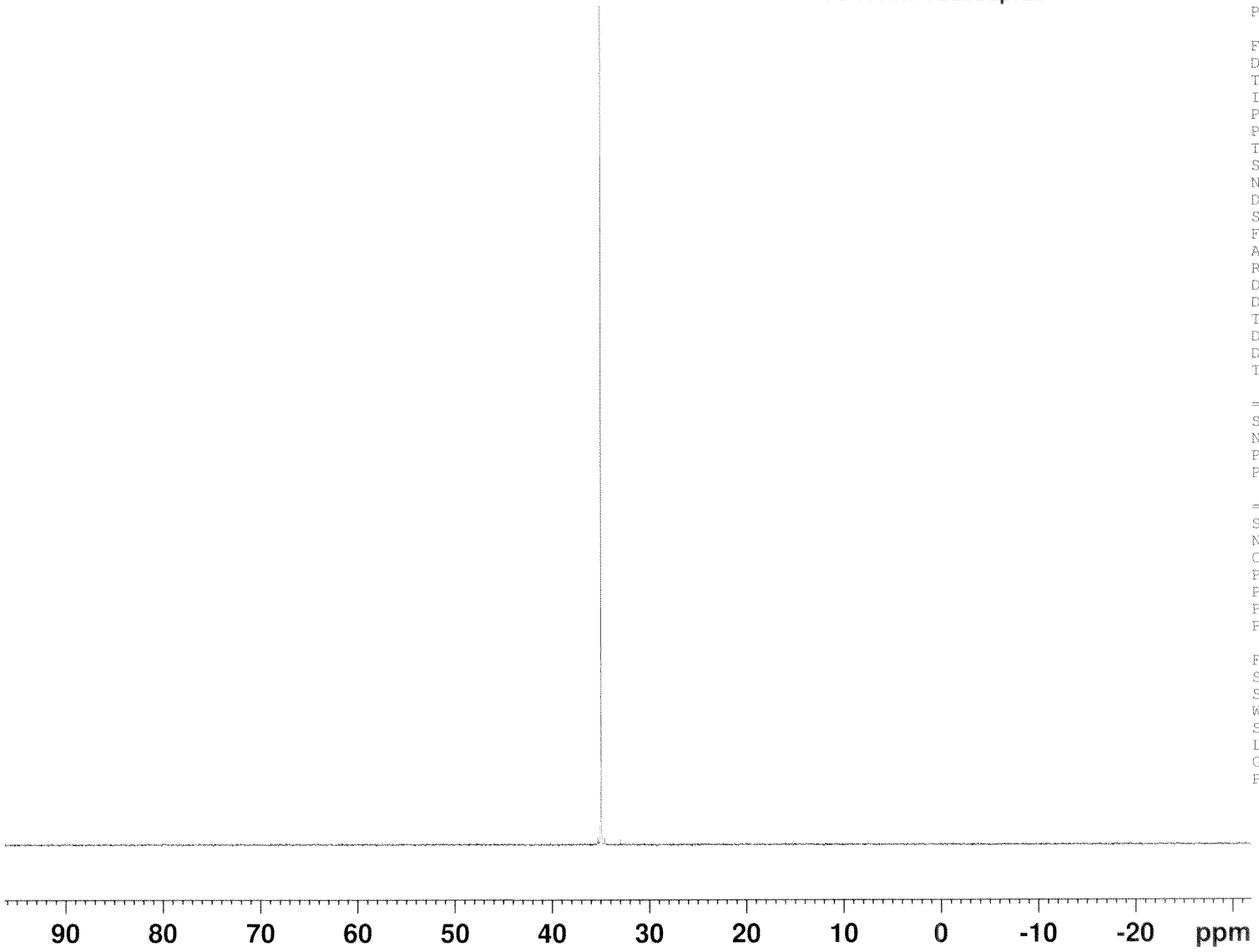
$^{31}P\{^{1}H\}$ NMR coupled



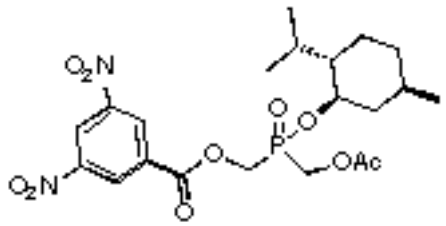

```
Current Data Parameters
NAME                  OB 2110
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_                20150709
Time                     9.25
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT              Acetone
NS                          32
DS                           4
SWH                64102.563 Hz
FIDRES             0.978127 Hz
AQ                 0.5111808 sec
RG                    203.57
DW                    7.800 usec
DE                    6.50 usec
TE                    293.9 K
D1                 2.00000000 sec
TD0                         1

======== CHANNEL f1 ========
SFO1             161.9674942 MHz
NUC1                      31P
P1                    14.25 usec
PLW1            15.00000000 W

F2 - Processing parameters
SI                      32768
SF               161.9755930 MHz
WDW                        EM
SSB                         0
LB                     1.00 Hz
GB                          0
PC                     1.40
```



Compound 46
$^{31}P\{^{1}H\}$ NMR decoupled

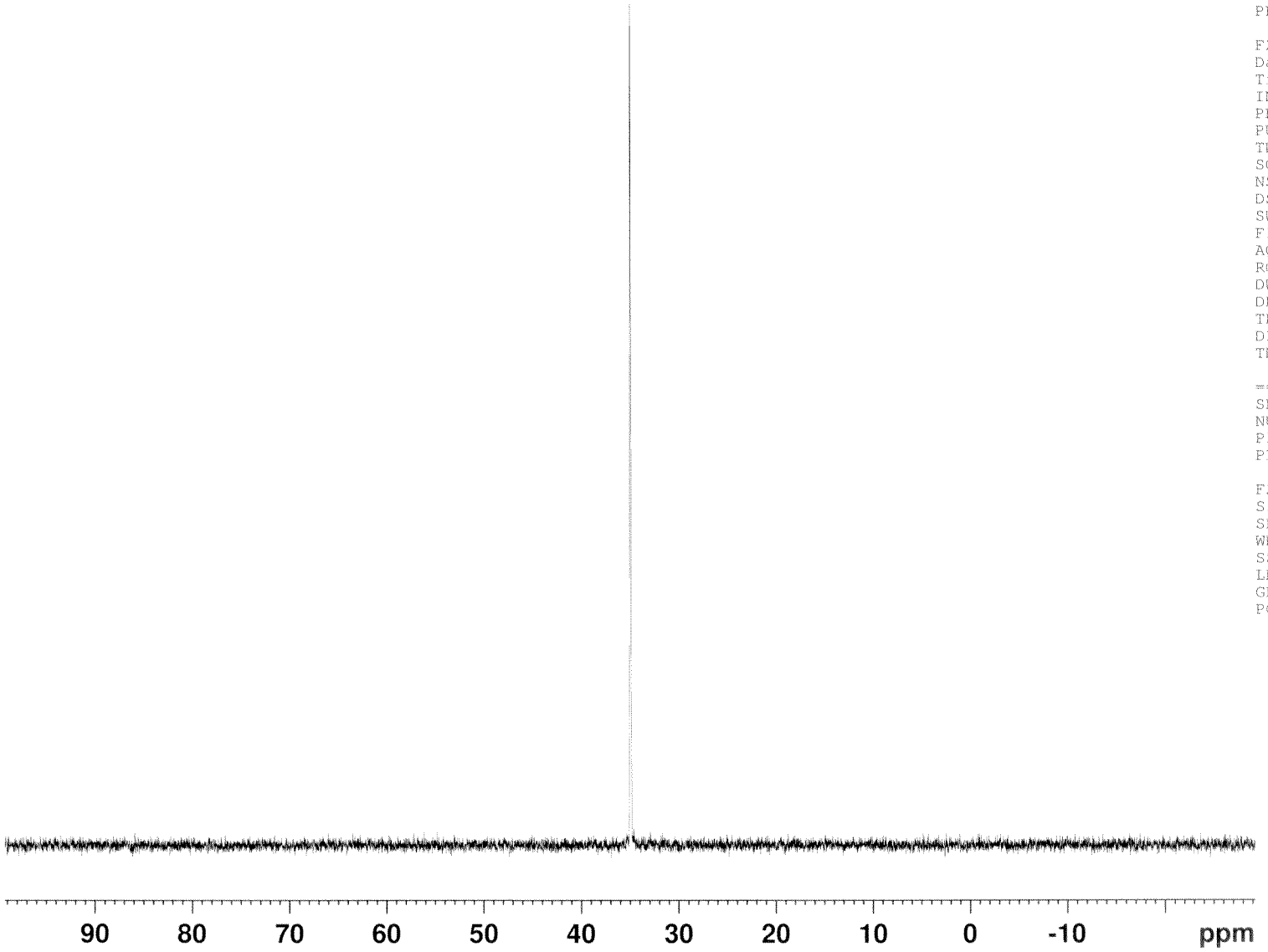


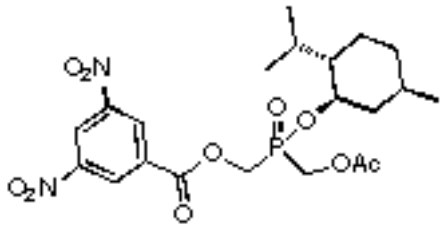

```
Current Data Parameters
NAME      OB 1474 recryst toluene
EXPNO                   1
PROCNO                  1

F2 - Acquisition Parameters
Date_           20140708
Time               18.51
INSTRUM            spect
PROBHD   5 mm BBI 1H/D-
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE                294.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1                31P
P1                14.44 usec
PLW1        50.00000000 W

======== CHANNEL f2 ========
SFO2        400.1316005 MHz
NUC2                1H
CPDPRG{2         waltz16
PCPD2             90.00 usec
PLW2        20.00000000 W
PLW12        0.07200000 W
PLW13        0.05832000 W

F2 - Processing parameters
SI                  32768
SF          161.9755930 MHz
WDW                    EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```



Compound 46
$^{31}P/^{1}H$ NMR coupled

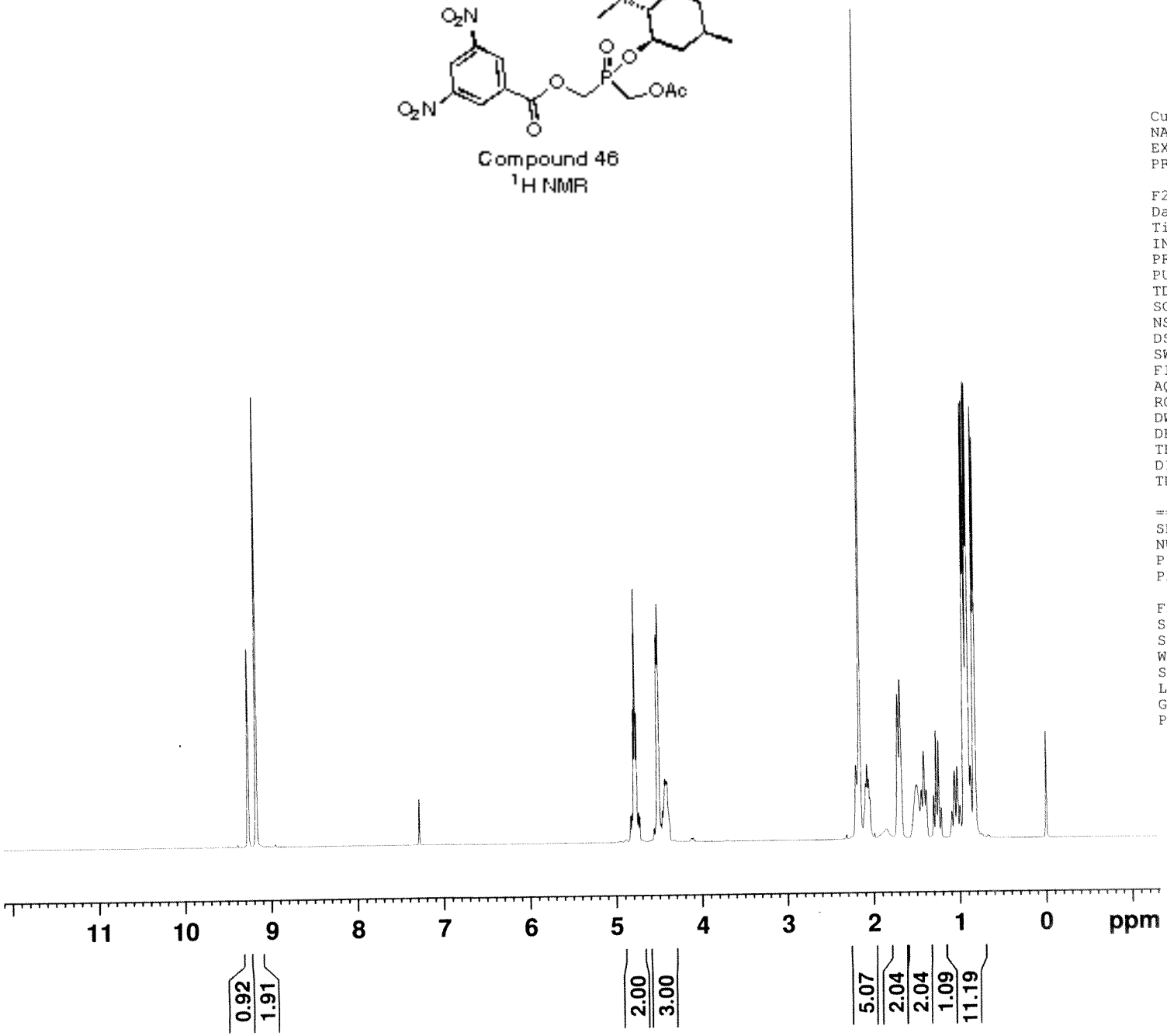
```
Current Data Parameters
NAME      OB 1474 recryst toluene
EXPNO                   2
PROCNO                  1

F2 - Acquisition Parameters
Date_           20140708
Time              18.52
INSTRUM           spect
PROBHD   5 mm BBI 1H/D-
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    4
SWH           64102.563 Hz
FIDRES         0.978127 Hz
AQ            0.5111808 sec
RG               203.57
DW                7.800 usec
DE                6.50 usec
TE                294.0 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        161.9674942 MHz
NUC1                31P
P1                14.44 usec
PLW1         50.00000000 W

F2 - Processing parameters
SI                32768
SF          161.9755930 MHz
WDW                 EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```



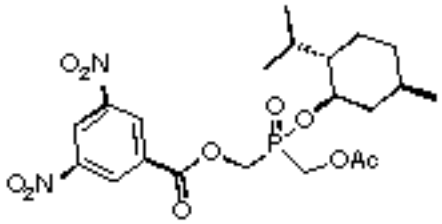


Compound 46

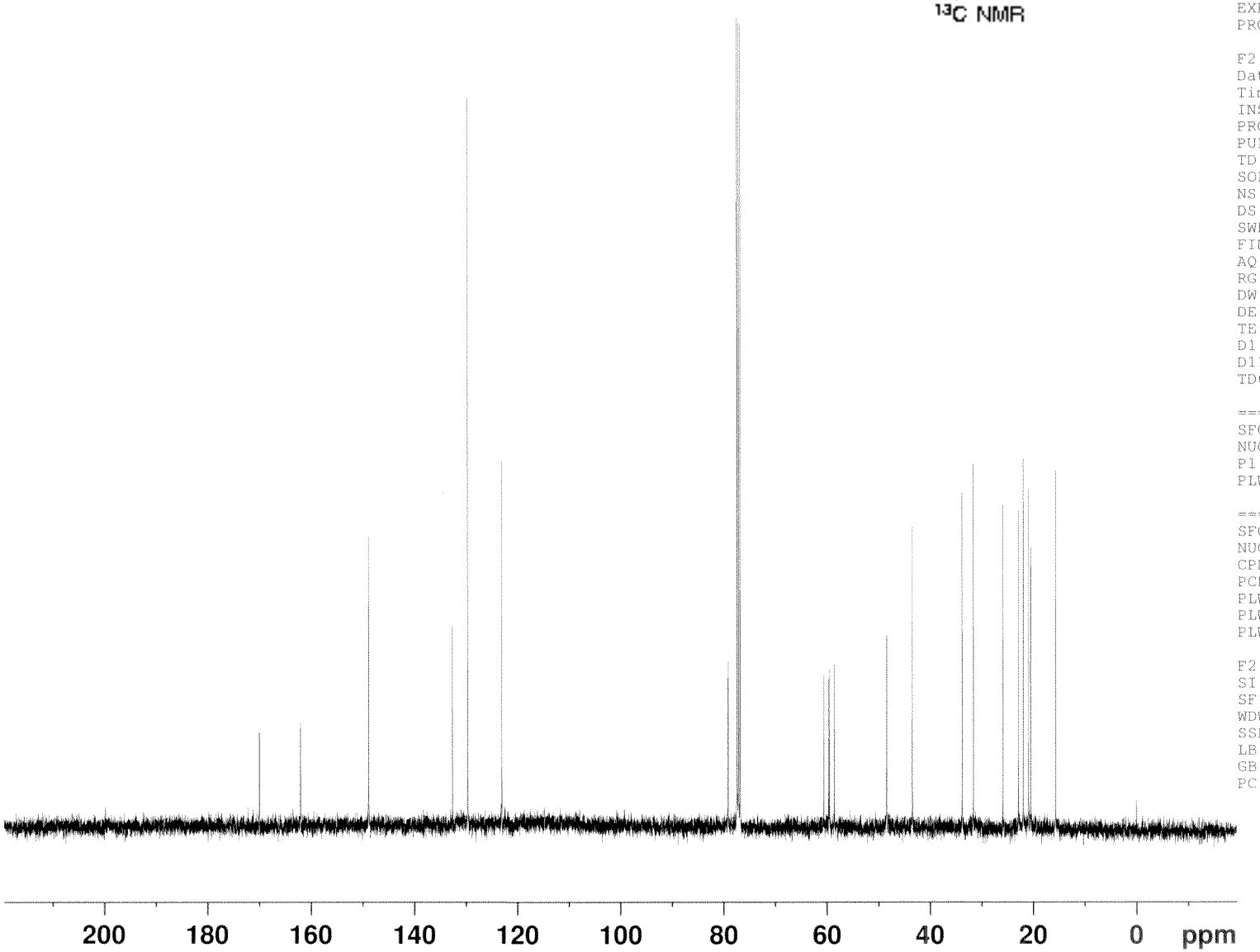
^{1}H NMR




```
Current Data Parameters
NAME      OB 1474 recryst toluene
EXPNO                           3
PROCNO                          1

F2 - Acquisition Parameters
Date_                    20140708
Time                        18.57
INSTRUM                     spect
PROBHD    5 mm BBI 1H/D-
PULPROG                      zg30
TD                          65536
SOLVENT                     CDCl3
NS                             16
DS                              2
SWH                      8012.820 Hz
FIDRES                   0.122266 Hz
AQ                       4.0894465 sec
RG                          32.38
DW                          62.400 usec
DE                           6.50 usec
TE                          294.0 K
D1                       1.00000000 sec
TD0                             1

======== CHANNEL f1 ========
SFO1                400.1324710 MHz
NUC1                        1H
P1                          5.75 usec
PLW1                20.00000000 W

F2 - Processing parameters
SI                          65536
SF                  400.1300000 MHz
WDW                            EM
SSB                 0
LB                          0.30 Hz
GB                  0
PC                          1.00
```



Compound 46
^{13}C NMR




```
Current Data Parameters
NAME      OB 1474 recryst toluene
EXPNO                   4
PROCNO                  1

F2 - Acquisition Parameters
Date_           20140708
Time              19.28
INSTRUM           spect
PROBHD   5 mm BBI 1H/D-
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  517
DS                    4
SWH           24038.461 Hz
FIDRES         0.366798 Hz
AQ             1.3631488 sec
RG                203.57
DW                20.800 usec
DE                 6.50 usec
TE                294.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         100.6228293 MHz
NUC1                13C
P1                12.95 usec
PLW1        100.00000000 W

======== CHANNEL f2 ========
SFO2         400.1316005 MHz
NUC2                1H
CPDPRG[2         waltz16
PCPD2             90.00 usec
PLW2         20.00000000 W
PLW12         0.07200000 W
PLW13         0.05832000 W

F2 - Processing parameters
SI                32768
SF           100.6127685 MHz
WDW                 EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```