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Supporting Information

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Synthesis and Enantioselectivity of P-Chiral Phosphine Ligands Possessing Alkynyl Groups

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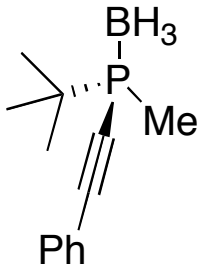
General.

All solvents used in reactions were dried and purified according to standard procedure. NMR spectra were measured with JEOL JMN-GXS-500 (500 MHz) or JEOR JMN-LA-400 (400 MHz) spectrometer in $CDCl_3$. Chemical shifts were reported in δ ppm. Optical rotations were measured with JASCO DIP-370 polarimeter. Enantiomeric excesses were determined by HPLC analysis using Chiralcel AD, OJ-H, OD-H, OB, IA columns and varying concentrations of 2-propanol/hexane as the mobile phase. X-ray crystal structure data were collected using a Bruker SMART APEX II diffractmeter with Mo-Kα radiation. Silica gel (Kanto Chemical, Silica Gel 60N for flash chromatography) was used for column chromatography.

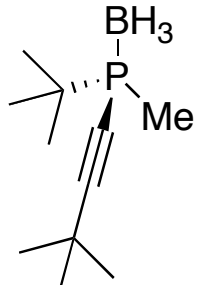
(*S*)-*tert*-Butylmethyl(phenylethynyl)phosphine–borane (3a)



To a solution of (*S*)-*tert*-butylmethylphosphine–borane (**1**) (>99% ee, 1.8 g, 15.4 mmol) in dry Et_2O (60 mL) was added *n*BuLi (11.6 mL of a 1.60 M solution in hexane, 18.5 mmol) at –78 °C under nitrogen, and the mixture was stirred for 15 min. 1,2-Dibromoethane (2.0 mL, 23.1 mmol) was added dropwise, and the reaction mixture was stirred at –78 °C. After 2 h, lithium phenylacetylide (30.8 mmol, 30 mL of Et_2O solution) was added to the reaction mixture, and the mixture was stirred at room temperature. After 1.5 h, the reaction was quenched with 1 M HCl. The mixture was extracted with EtOAc three times. The combined organic layers were washed with saturated $NaHCO_3$ and brine, and dried over Na_2SO_4. After filtration, the filtrate was concentrated under reduced pressure, and the residue was purified by chromatography on silica gel (hexane/EtOAc = 5/1) to give (*S*)-*tert*-butylmethyl(phenylethynyl)phosphine–borane (**3a**) (97% ee, 2.8 g, 12.8 mmol, 83%) as white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.42–0.98 (m, 4H), 1.29 (d, ^3J(H,P) = 15.6 Hz, 9H), 1.51 (d, ^2J(H,P) = 10.0 Hz, 3H), 7.33–7.37 (m, 2H), 7.41 (tt, ^3J(H,H) = 15.3, ^4J(H,H) = 1.7 Hz, 1H), 7.50–7.52 (m, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 8.37 (d, *J*(CP) = 40 Hz), 24.96 (d, *J*(CP) = 4 Hz), 29.03 (d, *J*(CP) = 37 Hz), 79.42 (d,

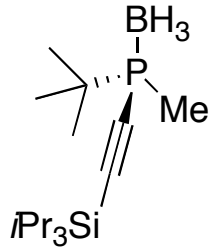
J(CP) = 90 Hz), 106.00 (d, *J*(CP) = 12 Hz), 120.77, 128.42, 130.01, 132.20. ^{31}P NMR (162 MHz, $CDCl_3$) δ 18.0. HRMS (FAB) calcd for $C_{13}H_{19}BP$ (M^+–H) 217.1320, found 217.1313. $[\alpha]^{22}_D$ = 0.97 cm^3 g^{-1} dm^{-1} (*c* = 0.0100 g cm^{-3} in $CHCl_3$), HPLC: Daicel Chiralcel OD-H, Hexane/*i*PrOH = 199/1, Flow rate = 0.5 mL/min, UV = 254 nm, t_R = 11.9 min (*R*), t_R = 13.1 min (*S*).

(*S*)-*tert*-Butyl(3,3-dimethyl-1-butynyl)methylphosphine–borane (3b)



In a similar manner (*S*)-*tert*-butyl(3,3-dimethyl-1-butynyl)methylphosphine–borane was prepared in 82% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.23–0.89 (m, 3H), 1.21 (d, ^3J(H,H) = 15.1 Hz, 9H), 1.27 (s, 9H), 1.38 (d, ^2J(H,H) = 10.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 8.54 (d, *J*(CP) = 40 Hz), 24.77 (d, *J*(CP) = 3 Hz), 28.62 (d, *J*(CP) = 35 Hz), 30.20 (d, *J*(CP) = 2 Hz), 68.70 (d, *J*(CP) = 95 Hz), 116.79 (d, *J*(CP) = 10 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 15.7. HRMS (FAB) calcd for $C_{11}H_{24}BP$ (M^++K) 237.1348, found 237.1351. $[\alpha]^{22}_D$ = 9.4 cm^3 g^{-1} dm^{-1} (*c* = 0.0013 g cm^{-3} in $CHCl_3$). Its enantiomeric excess was determined to be 98% ee by HPLC analysis. HPLC: Daicel Chiralpak IA, Hexane/*i*PrOH=1000/1, Flow rate = 0.5 mL/min, UV = 230 nm, t_R = 13.7 min (*R*), t_R = 15.3 min (*S*).

(*S*)-*tert*-Butylmethyl(triisopropylsilylethynyl)phosphine–borane (3c)



In a similar manner (*S*)-*tert*-butylmethyl(triisopropylsilylethynyl)phosphine–borane was prepared in 86% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.27–0.73 (m, 3H), 1.09–1.10 (m, 21H), 1.24 (d, ^3J(HP) = 15.4 Hz, 9H), 1.43 (d, ^2J(HP) = 10.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 8.45 (d, *J*(CP) = 40 Hz), 10.90, 18.43, 24.82 (d, *J*(CP) = 3 Hz), 28.62 (d, *J*(CP) = 38 Hz), 98.26 (d, *J*(CP) = 77 Hz), 113.26 (d, *J*(CP) = 3 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 17.5. HRMS (FAB) calcd for $C_{16}H_{35}BPSi$ (M^+–H) 297.2342, found 297.2332. $[\alpha]^{22}_D$ = –8.9 cm^3 g^{-1} dm^{-1} (*c* = 0.0101 g cm^{-3} in $CHCl_3$). Its enantiomeric excess was determined to be 98% ee by HPLC analysis. Daicel Chiralpak IA, Hexane/*i*PrOH = 1000/1, Flow rate = 0.5 mL/min, UV = 230 nm, t_R = 11.0 min (*R*), t_R = 13.6 min (*S*).

(*S*)-*tert*-Butylmethyl(trimethylsilylethynyl)phosphine–borane (3d)

In a similar manner (*S*)-*tert*-butylmethyl(trimethylsilylethynyl)phosphine–borane was prepared in 81% yield. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.22 (s, 9H), 1.23 (d, ^3J(HP) = 15.4 Hz, 9H), 1.42 (d, ^2J(HP) = 10.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 8.92 (d, *J*(CP) = 40 Hz), 25.45 (d, *J*(CP) = 4 Hz), 29.28 (d, *J*(CP) = 38 Hz), 96.79 (d, *J*(CP) = 77 Hz), 116.66 (d, *J*(CP)

= 3 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 17.1. $[\alpha]^{22}_D$ = –1.59 cm^3 g^{-1} dm^{-1} (c = 0.0100 g cm^{-3} in $CHCl_3$). Its enantiomeric excess was determined to be 99% ee by HPLC analysis. Daicel Chiralcel OJ-H, Hexane/*i*PrOH = 99/1, Flow rate = 0.5 mL/min, UV = 230 nm, t_R = 8.8 min (*R*), t_R = 9.7 min (*S*).

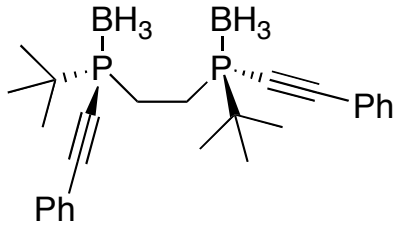
(*S*)-*tert*-Butylmethylphenylphosphine–borane (3e),[1–4] (S)-*tert*-Butyl(*o*-methoxyphenyl)-methylphosphine–borane (3f),[2,5] and (*S*)-Benzyl(*tert*-butyl)methylphosphine–borane (4b)[6,7]

The absolute configurations and enantiomeric excesses of the products (**3e**, **3f**, and **4b**) were determined by HPLC analysis in comparison with the reported data.[1–7] **3e**: Daicel Chiralcel OJ-H, Hexane/*i*PrOH = 9/1, Flow rate = 0.5 mL/min, UV = 230 nm, t_R = 25.5 min (*R*), t_R = 27.7 min (*S*); **3f**: Daicel Chiralcel OD-H, Hexane/*i*PrOH = 99/1, Flow rate = 0.5 mL/min, UV = 230 nm, t_R = 11.0 min (*R*), t_R = 11.6 min (*S*); **4b**: Daicel Chiralcel OD-H, Hexane/*i*PrOH = 9/1, Flow rate = 0.5 mL/min, UV = 230 nm, t_R = 16.9 min (*R*), t_R = 18.9 min (*S*).

(*S*)-*n*-Butyl(*tert*-butyl)methylphosphine–borane (4a)

The absolute configuration and the optical purity of compound **4a** ($[\alpha]_D$ = –4.1 cm^3 g^{-1} dm^{-1} (c = 0.010 g cm^{-1} in $CHCl_3$)) were determined by comparison of the sign of rotation and specific rotation of the authentic sample ($[\alpha]_D$ = –4.5 cm^3 g^{-1} dm^{-1} (c = 0.010 g cm^{-3} in $CHCl_3$)) prepared by the reaction of compound **1** with *n*BuLi and bromobutane.

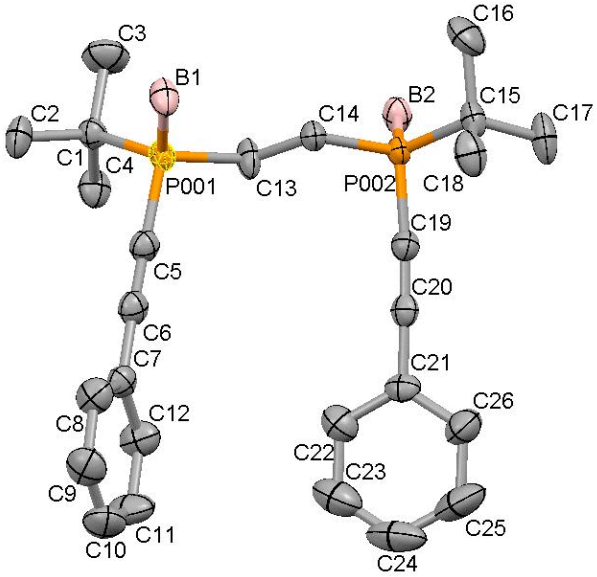
(*S*,*S*)-1,2-Bis(boranato(*tert*-butyl)(phenylethynyl)phosphino)ethane (6a)



To a solution of (*S*)-*tert*-butylmethyl(phenylethynyl)-phosphine–borane (**3a**) (97% ee, 0.95 g, 4.4 mmol) and TMEDA (0.8 mL, 5.2 mmol) in dry Et_2O (13 mL) was added *s*-BuLi (5.2 mL of a 1.0 M solution in hexane, 5.2 mmol) at –78 °C under nitrogen, and the mixture was stirred for 1 h and –50 °C for 10 min. Copper(II) chloride (1.5 g, 11 mmol) was added with vigorous stirring and the resulting mixture was gradually warmed to room temperature. After 2 h, the reaction was quenched with saturated NH_4Cl. The mixture was extracted with EtOAc three times. The combined organic layers were washed with brine, and dried over Na_2SO_4. After filtration, the filtrate was concentrated under reduced pressure, and the residue was purified by chromatography on silica gel (hexane/EtOAc = 4/1) to give (*S*,*S*)-1,2-bis(boranato(*tert*-butyl)(phenylethynyl)phosphino)ethane (**6a**) (0.69 g, 1.6 mmol, 73%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.57–0.80 (m, 6H), 1.32–1.35 (m, 18H), 2.13–2.28 (m, 4H), 7.29–7.32 (m, 4H), 7.41 (tt, ^3J(H,H) = 15.0 Hz, ^4J(H,H) = 2.5 Hz, 2H), 7.46–7.47 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 17.01 (d, J(CP) = 34 Hz), 25.30 (t,

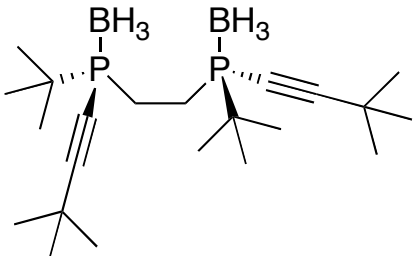
J(CP) = 3 Hz), 30.11 (d, *J*(CP) = 36 Hz), 77.88 (d, *J*(CP) = 88 Hz), 107.38, 120.39, 128.48, 130.22, 132.24. ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.6. $[\alpha]^{22}_D$ = 111 cm^3 g^{-1} dm^{-1} (*c* =0.0100 g cm^{-3} in $CHCl_3$).

Recrystallization of compound **6a** from a mixed solvent of EtOAc and *n*-hexane gave analysis. Empirical prisms. Absolute configuration of this compound was determined by



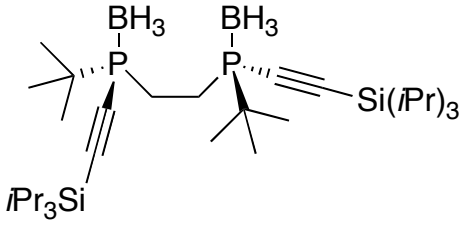
single crystal X-ray Formula $C_{26}H_{48}B_2P_2$; Formula weight 444.20; Temperature 173 K; Wavelength 0.71073 Å; Crystal system Orthorhombic; Space group P2(1)2(1)2; Unit cell dimensions *a* =22.202(5) Å, α = 90°, *b* = 10.932(3) Å, β = 90°, *c* = 11.379(3) Å, γ = 90°; Volume 2761.8(11) $Å^3$; *Z* = 5; Density (calculated) 1.335 Mg/m^3; Crystal size 0.45 x 0.35 x 0.15 mm^3; GOF = 1.034; Final *R* indice [$I>2\sigma(I)$] *R*1 = 0.0446, *wR*2 = 0.1045; *R* indices (all data) *R*1 = 0.0524, *wR*2 = 0.1095; Absolute structure parameter 0.06(10). CCDC-641148.

(*S*,*S*)-1,2-Bis(boranato(*tert*-butyl)(3,3-dimethyl-1-butynyl)phosphino)ethane (6b)



This compound was prepared in 73% yield by the oxidative coupling of **4b**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.22–0.88 (m, 6H), 1.22–1.26 (d, ^3J(H,P) = 15.1 Hz, 18H), 1.28 (s, 18H), 1.94–2.02 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 17.05 (d, *J*(CP) = 34 Hz), 25.12, 28.58, 29.58 (d, *J*(CP) = 37 Hz), 30.26, 67.35 (d, *J*(CP) = 93 Hz), 118.42 (d, *J*(CP) = 10 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.8. HRMS (FAB) calcd for $C_{22}H_{46}B_2P_2$ (M^++K) 433.2906, found 433.2881. $[\alpha]^{22}_D$ = 19.4 cm^3 g^{-1} dm^{-1} (*c* = 0.0096 g cm^{-3} in $CHCl_3$).

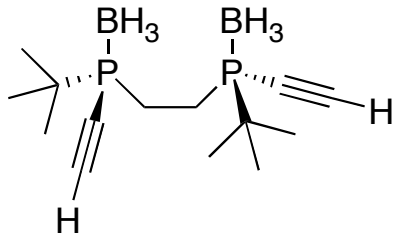
(*S*,*S*)-1,2-Bis(boranato(*tert*-butyl)triisopropylsilylethynylphosphino)ethane (5c)



This compound was prepared in 78% yield by the oxidative coupling of **4c**. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.27–0.73 (m, 6H), 1.06–1.14 (m, 42H), 1.25–1.29 (m, 18H), 1.98–2.13 (m, 4H). ^{13}C NMR

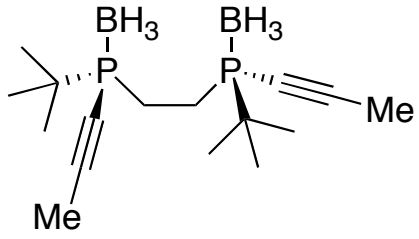
(100 MHz, $CDCl_3$) δ 10.92, 16.73 (d, *J*(CP) = 32 Hz), 18.50 (d, *J*(CP) = 2 Hz), 25.19, 29.63 (d, *J* = 36 Hz), 96.47 (d, *J* = 74 Hz), 115.49. ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.3. HRMS (FAB) calcd for $C_{32}H_{70}B_2P_2Si_2$ (M^++K) 633.4327, found 633.4327. $[\alpha]^{22}_D$ = 25.1 $cm^3\ g^{-1}\ dm^{-1}$ (c = 0.0100 g cm^{-3} in $CHCl_3$).

(*S*,*S*)-1,2-Bis(boranato(*tert*-butyl)ethynylphosphino)ethane (6d)



Tetrabutylammonium fluoride (0.4 mL of a 1.0 M solution in THF, 0.4 mmol) was added to (*S*,*S*)-1,2-bis(boranato(*tert*-butyl)(triisopropylsilylethynyl)phosphino)ethane (**6c**) (60 mg, 0.1 mmol) under nitrogen, and the mixture was stirred at room temperature. After 6 h, the reaction was quenched by the addition of water. The mixture was extracted with EtOAc three times. The combined organic layers were washed with brine, and dried over Na_2SO_4. After filtration, the filtrate was concentrated under reduced pressure, and the residue was purified by chromatography on silica gel (hexane/EtOAc = 3/1) to give (*S*,*S*)-1,2-bis(boranato(*tert*-butyl)ethynylphosphino)ethane (**6d**) (15 mg, 0.052 mmol, 52%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.24–0.92 (m, 6H), 1.27–1.31 (m, 18H), 2.01–2.15 (m, 4H), 3.08–3.10 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 16.31 (d, *J*(CP) = 33 Hz), 25.08, 29.66 (d, *J*(CP) = 35 Hz), 74.06 (d, *J*(CP) = 81 Hz), 96.28. ^{31}P NMR (162 MHz, $CDCl_3$) δ 30.9. HRMS (FAB) calcd for $C_{14}H_{30}B_2P_2Si_2$ (M^++K) 321.1651, found 321.1662. $[\alpha]^{22}_D$ = –13.9 $cm^3\ g^{-1}\ dm^{-1}$ (c = 0.0024 g cm^{-3} in $CHCl_3$).

(*S*,*S*)-1,2-Bis(boranato(*tert*-butyl)(1-propynyl)phosphino)ethane (6e)



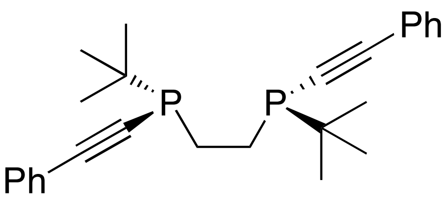
To a solution of (*S*,*S*)-1,2-bis(boranato(*tert*-butyl)ethynylphosphino)ethane (**6d**) 31 mg, 0.11 mmol) in THF (0.55 mL) was added *s*BuLi (0.28 mL of a 1.0 M solution in hexane, 0.28 mmol) at –78 °C under nitrogen, and the mixture was stirred for 1 h. Iodomethane (34 μL, 0.55 mmol) was added to the solution with stirring and the mixture was warmed to room temperature during 2 h. The reaction was quenched with saturated NH_4Cl. The mixture was extracted with EtOAc three times. The combined organic layers were washed with brine, and dried over Na_2SO_4. After filtration, the filtrate was concentrated under reduced pressure, and the residue was purified by chromatography on silica gel (hexane/EtOAc = 4/1) to give (*S*,*S*)-1,2-bis(boranato(*tert*-butyl)(1-propynyl)phosphino)ethane (**6e**) (30 mg, 88%) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 0.24–0.86 (m, 6H), 1.23–1.27 (m, 18H), 2.00–2.06 (m, 10H). ^{13}C NMR (100 MHz,

$CDCl_3$) δ 5.17, 16.86 (d, *J*(CP) = 35 Hz), 25.22, 29.70 (d, *J*(CP) = 37 Hz), 68.20 (d, *J*(CP) = 95 Hz), 106.53. ^{31}P NMR (162 MHz, $CDCl_3$) δ 28.1. HRMS (FAB) calcd for $C_{16}H_{34}B_2P_2$ (M^++K) 349.1965, found 349.1953. $[\alpha]^{22}_{D}$ = 21.6 cm^3 g^{-1} dm^{-1} (*c* = 0.00050 g cm^{-1} in $CHCl_3$).

Preparation of (*S,S*)-1,2-Bis(*tert*-butyl)(1-alkynyl)phosphine)ethanes (AlkynylP*). A typical Procedure

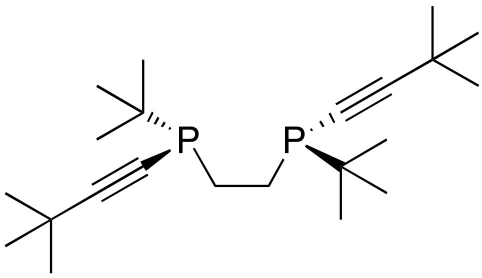
Compound **6a** (36 mg, 0.084 mmol) and 1,4-diazabicyclo[2.2.2]octane (56 mg, 0.51 mmol) were dissolved in degassed THF (0.5 mL) under nitrogen. The solution was stirred under nitrogen at 60 °C for 1 h and the reaction mixture concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel using degassed diethyl ether as the eluent under nitrogen to give diphosphine **5a** (32 mg, 94%). In a similar manner, ligands **5b**, **5c**, **5d**, and **5e** were prepared in 94–98% yield.

(*S*,*S*)-1,2-Bis(*tert*-butyl(phenylethynyl)phosphino)ethane (5a)



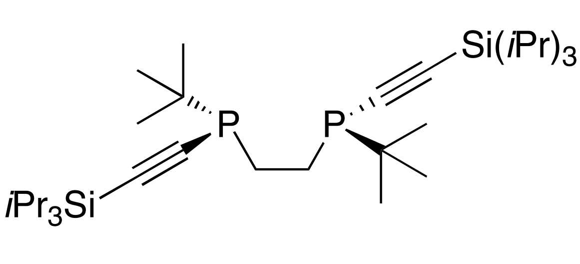
^{1}H NMR (400 MHz, $CDCl_3$) δ 1.19–1.26 (m, 18H), 1.67–1.80 (m, 2H), 2.09–2.19 (m, 2H), 7.23–7.31 (m, 3H), 7.40–7.43 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.27, 27.55, 29.99, 87.96, 105.38, 123.25, 128.19, 128.25, 131.58. ^{31}P NMR (202 MHz, $CDCl_3$) δ –16.43.

(*S*,*S*)-1,2-Bis(*tert*-butyl(3,3-dimethyl-1-butynyl)phosphino)ethane (5b)



^{1}H NMR (400 MHz, $CDCl_3$) δ 1.11–1.19 (m, 18H), 1.25 (s, 18H), 1.48–1.58 (m, 2H), 1.82–1.90 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.24, 27.28, 28.56, 29.35, 30.99, 75.61, 115.28. ^{31}P NMR (202 MHz, $CDCl_3$) δ –15.48.

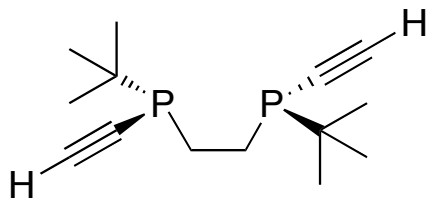
(*S*,*S*)-1,2-Bis(*tert*-butyl(triisopropylsilylethynyl)phosphino)ethane (5c)



^{1}H NMR (400 MHz, $CDCl_3$) δ 1.01–1.07 (m, 42H), 1.13–1.18 (m, 18H), 1.53–1.1.64 (m, 2H), 1.96–2.05 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ □□□□□□□□□□□□□□□□□□□□□□□□ □□□□□□□□□□□□□□□□□□□□□□□□□

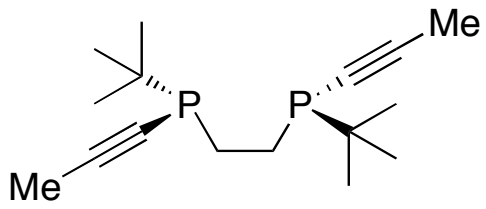
□□□□□ ^{31}P NMR (202 MHz, $CDCl_3$) δ –13.09.

(*S*,*S*)-1,2-Bis(*t*-butyl(ethynyl)phosphino)ethane (5d)



^{1}H NMR (400 MHz, $CDCl_3$) δ 1.1–1.18 (m, 18H), 1.54–1.65 (m, 2H), 1.96–2.03 (m, 2H), 2.86 (s, CCH). (500 MHz, $CDCl_3$/CD_3OD) δ 1.14–1.18 (m, 18H), 1.55–1.65 (m, 2H), 1.96–2.03 (m, 2H) (The signal of CCH was not observed.). ^{13}C NMR (100 MHz, $CDCl_3$) δ 19.76, 27.31, 29.40, 83.30–83.53 (m), 93.52. ^{31}P NMR (202 MHz, $CDCl_3$) δ –14.25.

(*S*,*S*)-1,2-Bis(*tert*-butyl(1-propynyl)phosphino)ethane (5e)



^{1}H NMR (400 MHz, $CDCl_3$) δ 1.10–1.15 (m, 18H), 1.49–1.60 (m, 2H), 1.84–1.94 (m, 2H), 1.98 (s, 6H). (Diethyl ether was strongly bound to this ligand and could not be removed in vacuo). ^{13}C NMR (125 MHz, $CDCl_3$) δ 5.14, 20.36 27.39, 29.39 (d, *J*(CP) = 3 Hz), 76.61, 102.52. ^{31}P NMR (202 MHz, $CDCl_3$) δ –13.00.

General Procedure for Rhodium-Catalyzed Asymmetric Hydrogenation of Methyl (*Z*)-α-Acetamidocinnamate

A 50 mL-hydrogenation tube was charged with methyl (*Z*)-α-acetamidocinnamate (0.5 mmol). The tube was connected to the hydrogen tank via stainless steel tubing. The vessel was evacuated and filled with 1 atm of hydrogen gas (Nippon Sanso, 99.9999%). A solution of $[Rh(nbd)_2]BF_4$ (1.9 mg, 5.0 μmol) and (*S*,*S*)-1,2-bis(1-alkynyl(*tert*-butyl)phosphino)ethane (AlkynylP*) (5.5 μmol) in degassed MeOH (1 mL) was added via a syringe to the tube, and the hydrogen pressure was increased to 1 atm. After 3 h, the reaction mixture was evaporated and the residue was purified by flash chromatography on silica gel using EtOAc as an eluent. The product was characterized by specral data and analyzed by HPLC using a chiral column. The absolute configuration and enantiomeric excess of the product were determined by comparison of the retention times with reported values.[8] Conditions for the HPLC analysis of *N*-acetylphenylalanine methyl ester: Chiralcel OJ, hexane/2-propanol = 9:1, 0.5 mL/min, wavelength = 254 nm, retention times: 22.3 min (*R*), 31.7 min (*S*).

General Procedure for Rhodium-Catalyzed Asymmetric 1,4-Addition of Organoboronic Acids to Enones

A solution of $[Rh(nbd)_2]BF_4$ (1.9 mg, 10 μmol) and ligand (*S*,*S*)-1,2-bis(1-alkynyl(*tert*-butyl)phosphino)ethane (AlkynylP*) (11 μmol) in dioxane (1 mL) was stirred at 40 °C for 15 min under nitrogen. To the reaction mixture was added KOH (0.1 mL, 1.5M, 0.15 mmol) in water and the solution was stirred for 15 min. Arylboronic acid (1.0 mmol) and α,β-unsaturated carbonyl compound (0.50 mmol) was added to the solution. After stirring at 40 °C for 2 h, the reaction mixture was quenched with saturated $NaHCO_3$ and extracted with ether five times. The combined organic layers were dried over sodium sulfate and concentrated under reduced pressure. The residue was purified by preparative TLC (silica gel, hexane/ EtOAc = 3/1). The products were characterized by their spectral data and analyzed by HPLC using chiral columns. The absolute configurations and ee values of the products were determined by comparison of the retention times with reported values.[9]

Conditions for the determination of the ee's by HPLC analysis. 3-Phenylcyclohexanone: Chiralcel OD-H, hexane:2-propanol = 98:2, 0.5 mL/min, wavelength 254 nm, retention times: 26.4 min (*S*), 28.2 min (*R*). 3-(4-Trifluoromethylphenyl)cyclohexanone: Chiralcel OJ, hexane:2-propanol = 98:2, 1.0 mL/min, wavelength 254 nm, retention times: 18.1 min (*R*), 20.5 min (*S*). 3-(4-Methoxyphenyl)cyclohexanone: Chiralcel OJ, hexane:2-propanol = 98:2, 1.0 mL/min, wavelength 254 nm, retention times: 34.6 min (*R*), 42.9 min (*S*). 3-Phenylcyclopentanone: Chiralcel OB, hexane:2-propanol = 98:2, 1.0 mL/min, wavelength 254 nm, retention times: 16.5 min (*S*), 22.4 min (*R*). 5-Methyl-4-phenylhexan-2-one: Chiralcel OJ, hexane:2-propanol = 98:2, 0.5 mL/min, wavelength 254 nm, retention times: 16.3 min (*R*), 20.0 min (*S*).

In a similar manner, 2-cyclohexenone (0.50 mmol) was reacted with phenylboronic acid in the presence of $[Rh(nbd)_2]BF_4$/*t*Bu-BisP* (2 mol%) at 40 °C. The reaction was sluggish and after 2 h the reaction mixture was worked up to give 3-phenylcyclohexanone in 37% yield. The ee of this product was determined to be 20% by HPLC analysis.

General Procedure for the Alkylative Ring-Opening of Oxabenzonorbornadiene Derivatives

A solution of $PdCl_2(cod)$ (1.1 mg, 8.0 μmol) and (*S*,*S*)-1,2-bis(1-alkynyl(*tert*-butyl)phosphino)ethane (AlkynylP*) (8.8 μmol) in CH_2Cl_2 (1 mL) was stirred at 40 °C under nitrogen for 15 min. To the solution was added a solution of oxabenzonorbornadiene (58 mg, 0.4 mmol) in CH_2Cl_2 (3 mL), followed by dimethylzinc (0.6 mL of 1.0 M hexane solution). The resulting solution was stirred at room temperature until completion of the reaction. The reaction was quenched by the addition of a few drops of water and the mixture was passed through a short plug of Celite and then concentrated. The residue was purified by preparative TLC (hexane/EtOAc).

The products were characterized by their spectral data and analyzed by HPLC using chiral columns. The absolute configurations and ee values of the products were determined by comparison of the retention times with reported values.[10]

Conditions for the determination of the ee's by HPLC analysis. 2-Methyl-1,2-dihydronaphthalen-1-ol: Chiralcel OD-H, hexane:2-propanol = 199:1, 1 mL/min, wavelength 254 nm, retention times: 28.5 min (1*R*,2*R*), 30.6 min (1*S*,2*S*); 2-Ethyl-1,2-dihydronaphthalen-1-ol: Chiralcel OD-H, hexane:2-propanol = 199:1, 1 mL/min, wavelength 254 nm, retention times: 26.0 min (1*R*,2*R*), 29.6 min (1*S*,2*S*). 2-Methyl-1,2-dihydro-5,8-bis(methoxymethoxy)-naphthalen-1-ol: Chiralcel OD-H, hexane:2-propanol = 9:1, 1.0 mL/min, wavelength 254 nm, retention times: 9.0 min (1*R*,2*R*), 11.1 min (1*S*,2*S*). 2-Ethyl-1,2-dihydro-5,8-bis(methoxymethoxy)-naphthalen-1-ol: Chiralcel OD-H, hexane:2-propanol = 9:1, 0.5 mL/min, wavelength 254 nm, retention times: 15.5 min (1*R*,2*R*), 17.0 min (1*S*,2*S*).

In a similar manner, oxabenzonorbornadiene (0.4 mmol) was reacted with diethylzinc in the presence of $PdCl_2$(cod)/*t*Bu-BisP* (2 mol%) at room temperature for 6 h to give (1*S*,2*S*)-2-ethyl-1,2-dihydronaphthalen-1-ol in 93% yield. The ee of this product was determined to be 94% by HPLC analysis.

References

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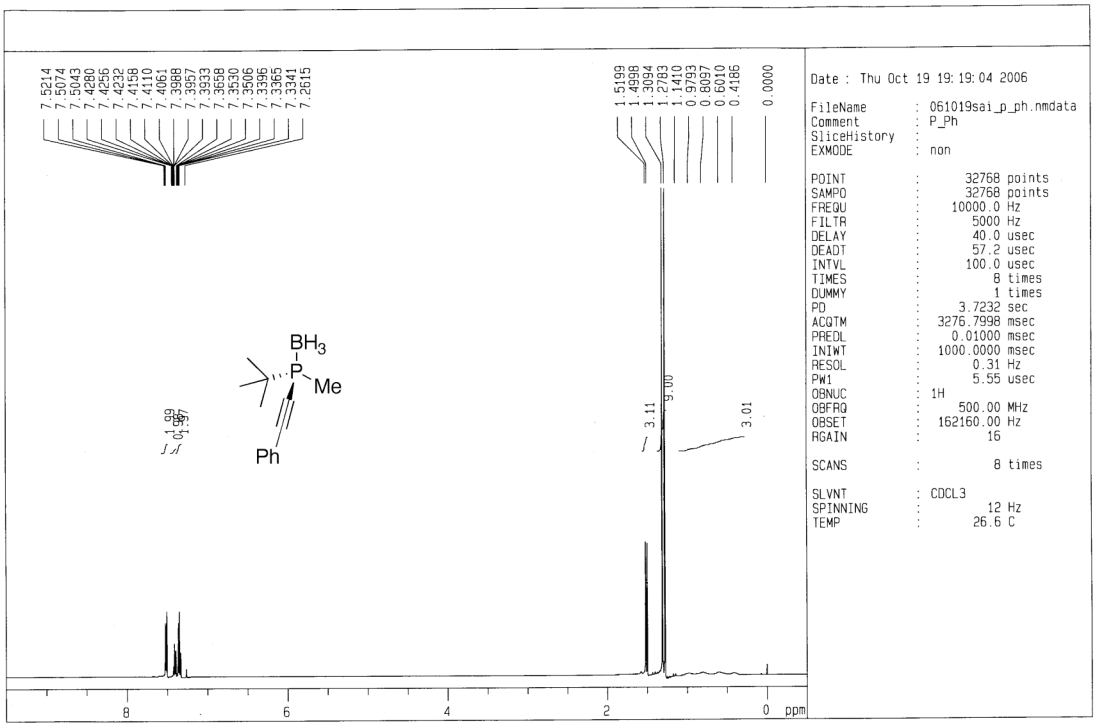
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7.3933
7.3658
7.3530
7.3506
7.3396
7.3365
7.3341
7.2615
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1.3094
1.2783
1.1410
0.9793
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Ph
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9.00
3.01
8
6
4
2
0 ppm
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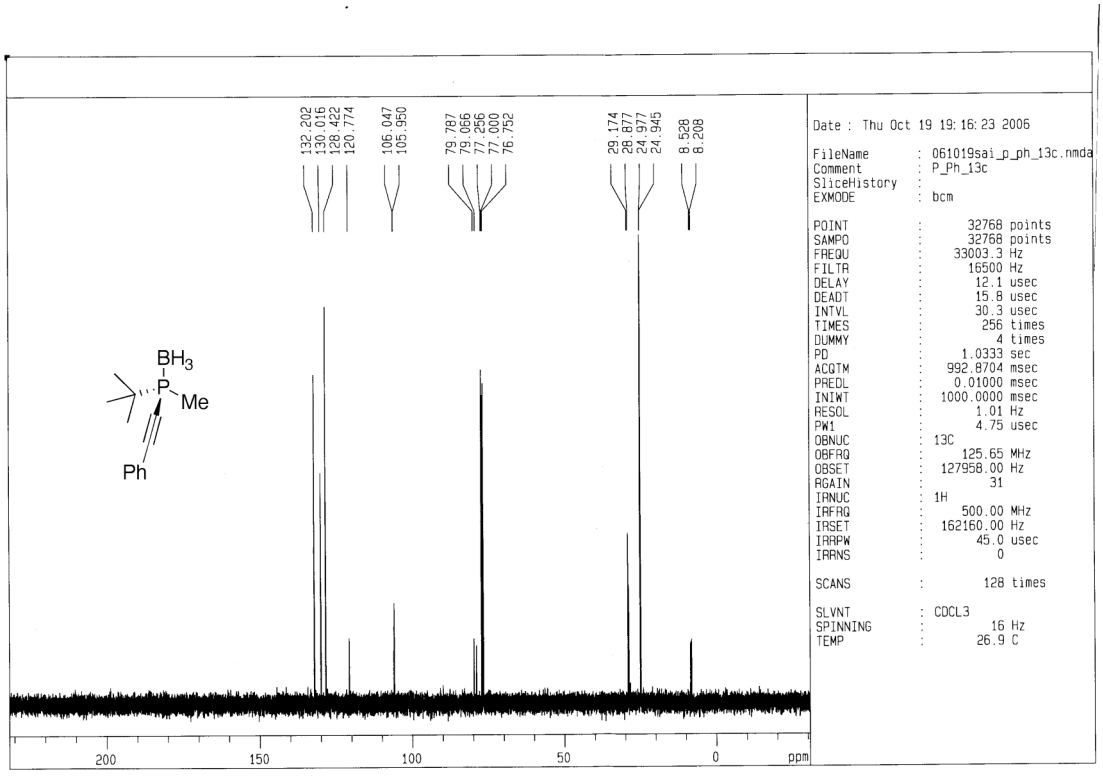
TEMP : 26.6 C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Ph
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100
50
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OBFRQ : 125.65 MHz
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IRNUC : 1H
IRFRQ : 500.00 MHz
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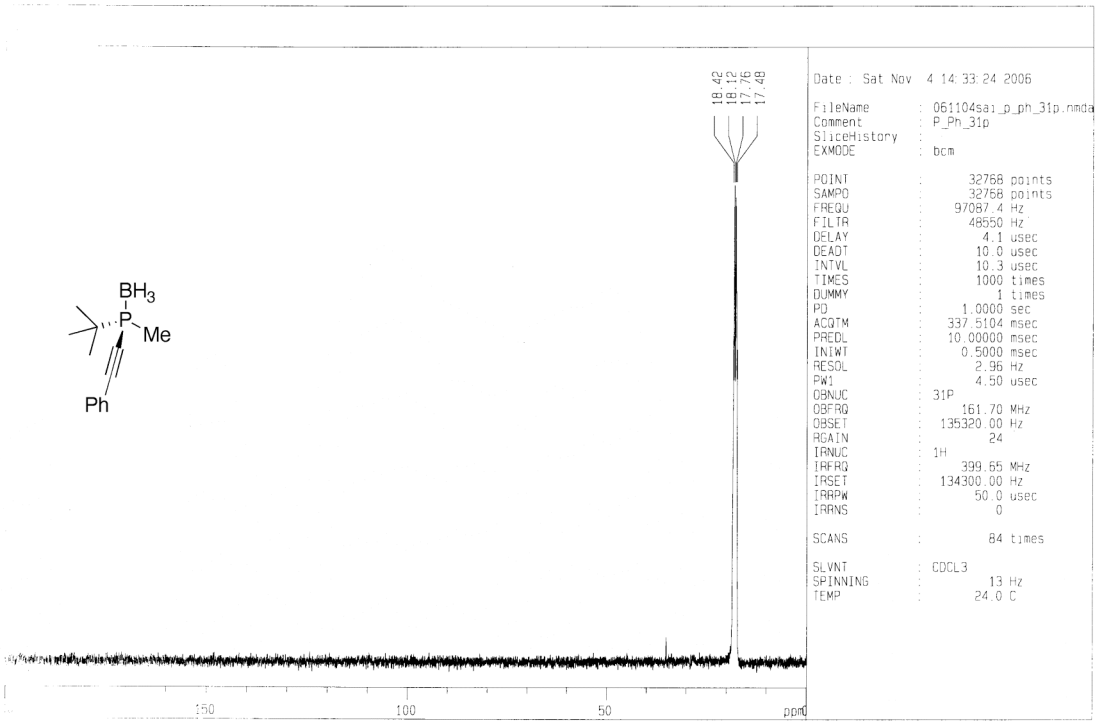
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ppm
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INTVL : 10.3 usec
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IRFRQ : 399.65 MHz
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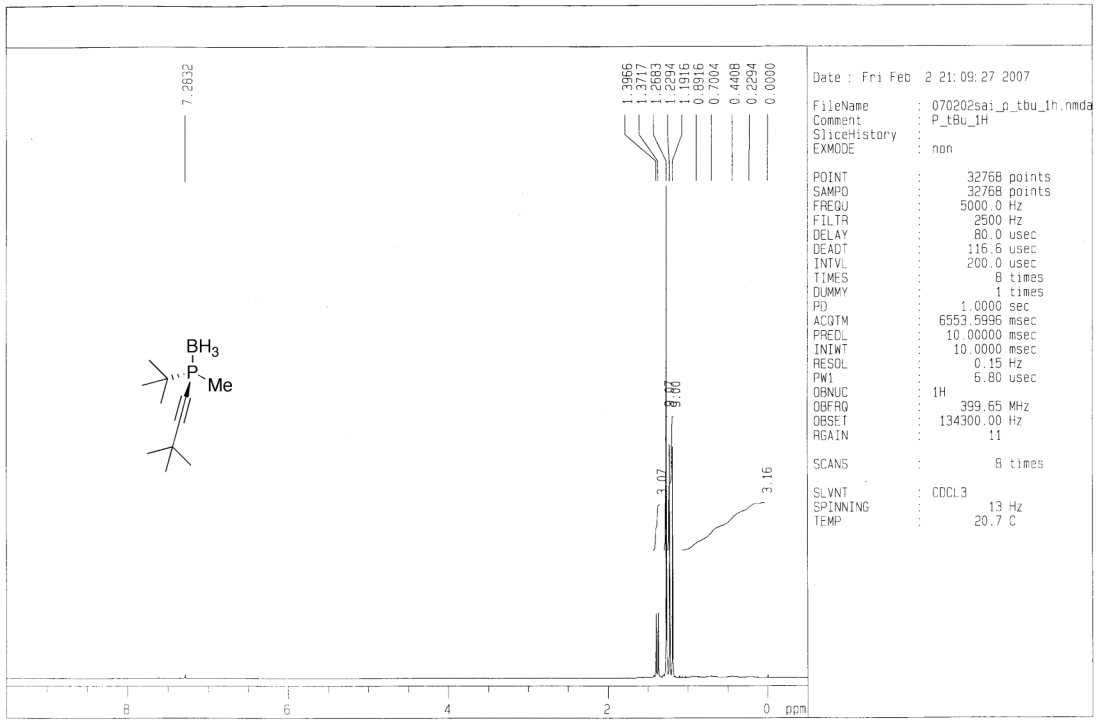
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6
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2
0
ppm
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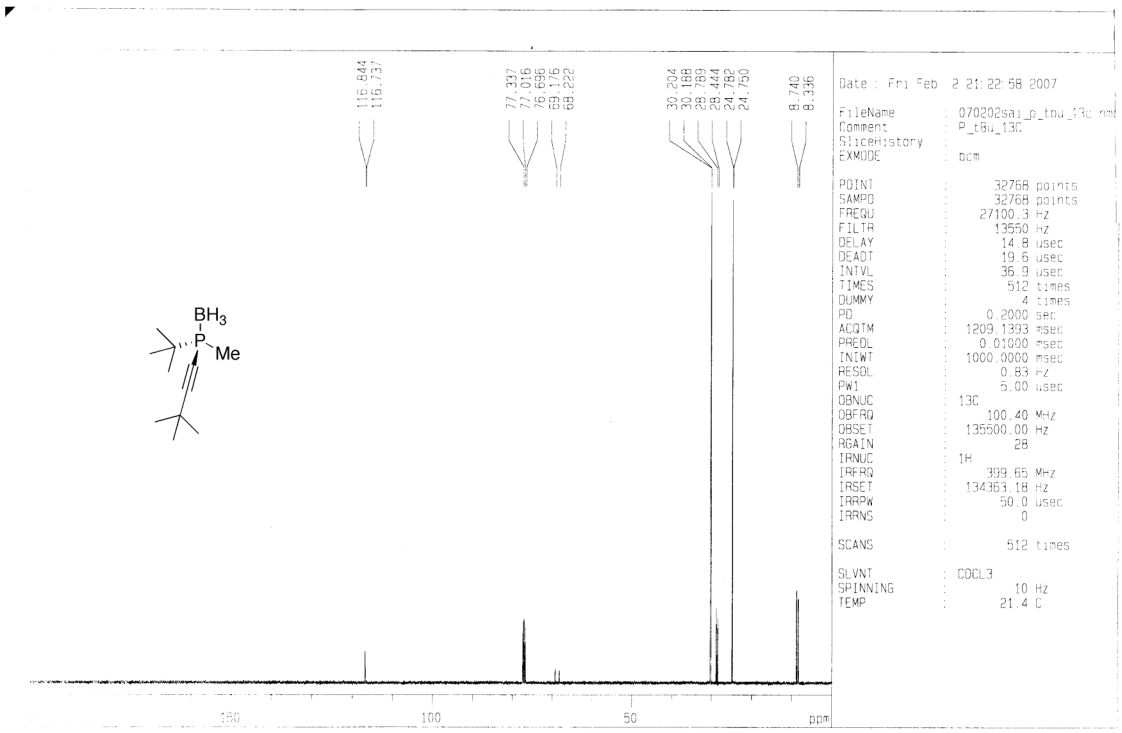
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ppm
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IRFRQ : 399.65 MHz
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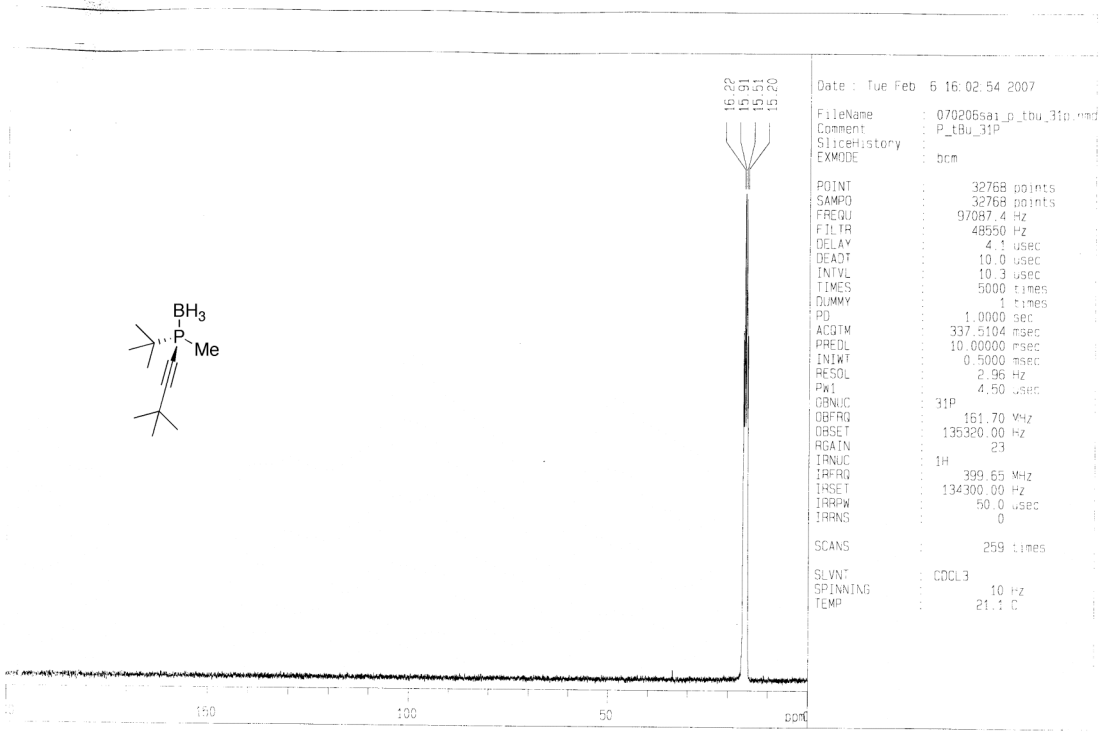
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INTVL : 10.3 usec
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OBFRQ : 161.70 MHz
OBSET : 135320.00 Hz
RGAIN : 23
IRNUC : 1H
IRFRQ : 399.65 MHz
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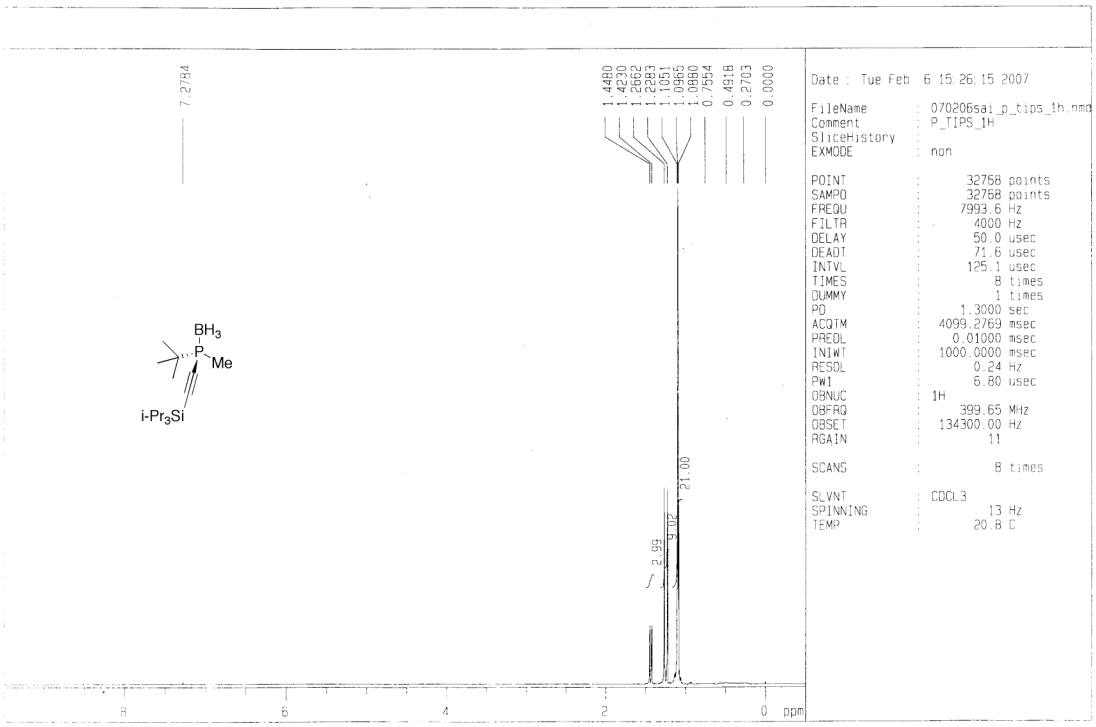
TEMP : 21.1 C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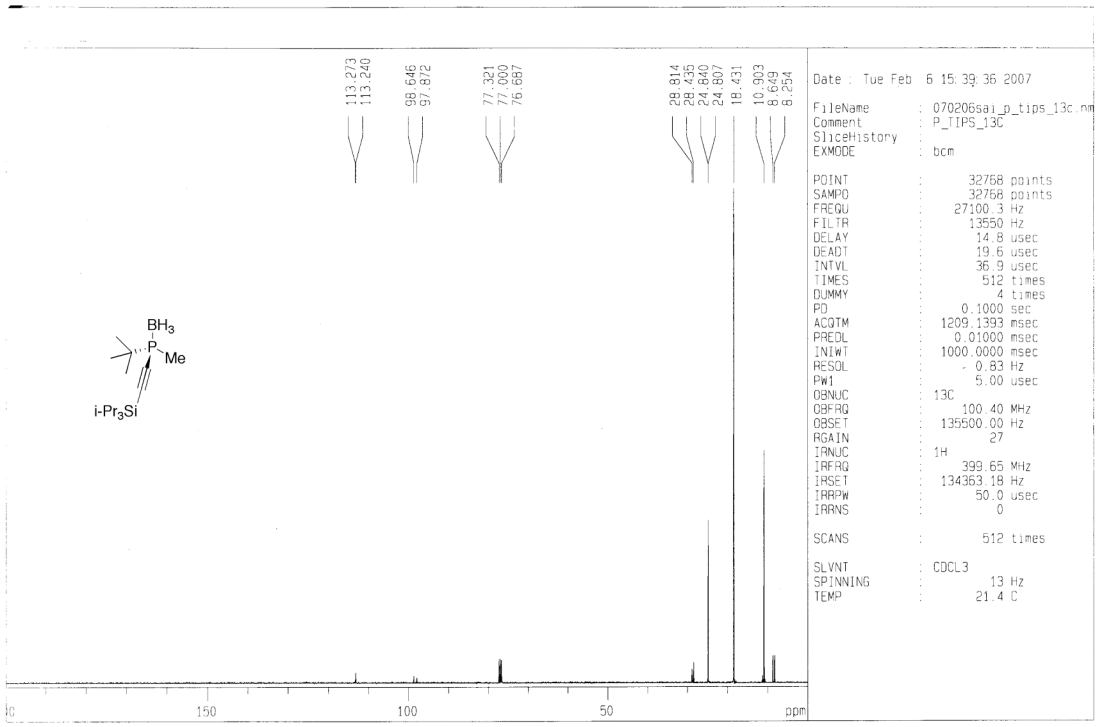
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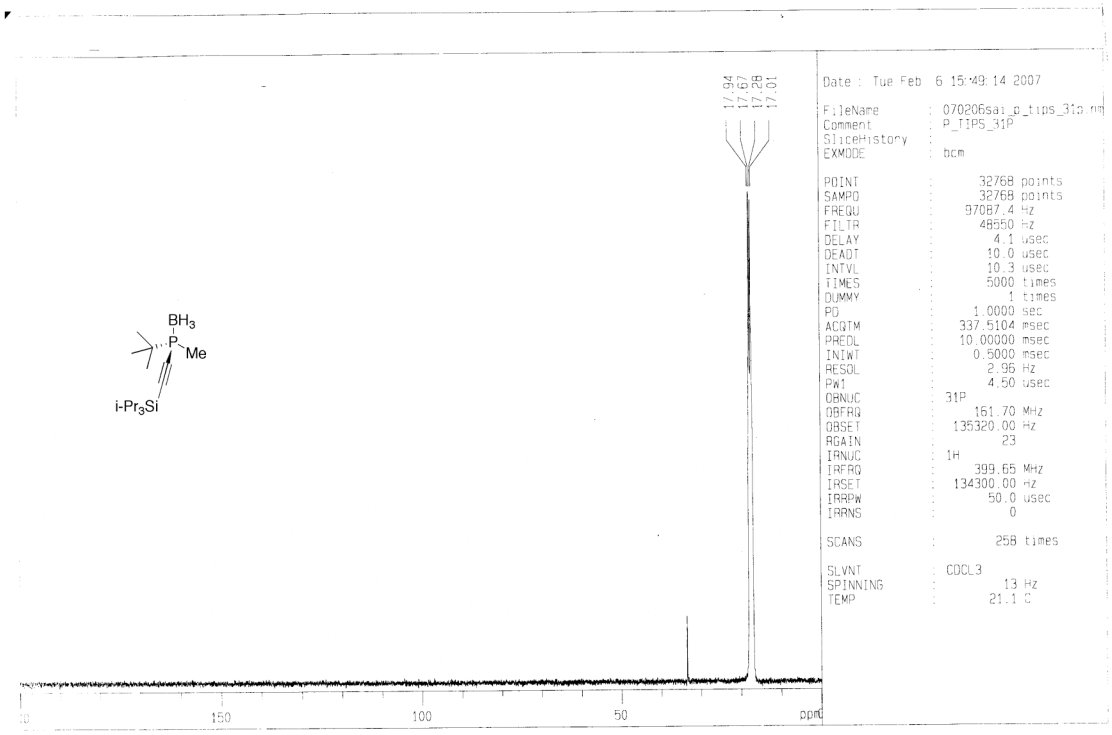
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FILTR : 48550 Hz
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DEADT : 10.0 usec
INTVL : 10.3 usec
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PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 2.96 Hz
PW1 : 4.50 usec
OBNUC : 31P
OBFRQ : 161.70 MHz
OBSET : 135320.00 Hz
RGAIN : 23
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134300.00 Hz
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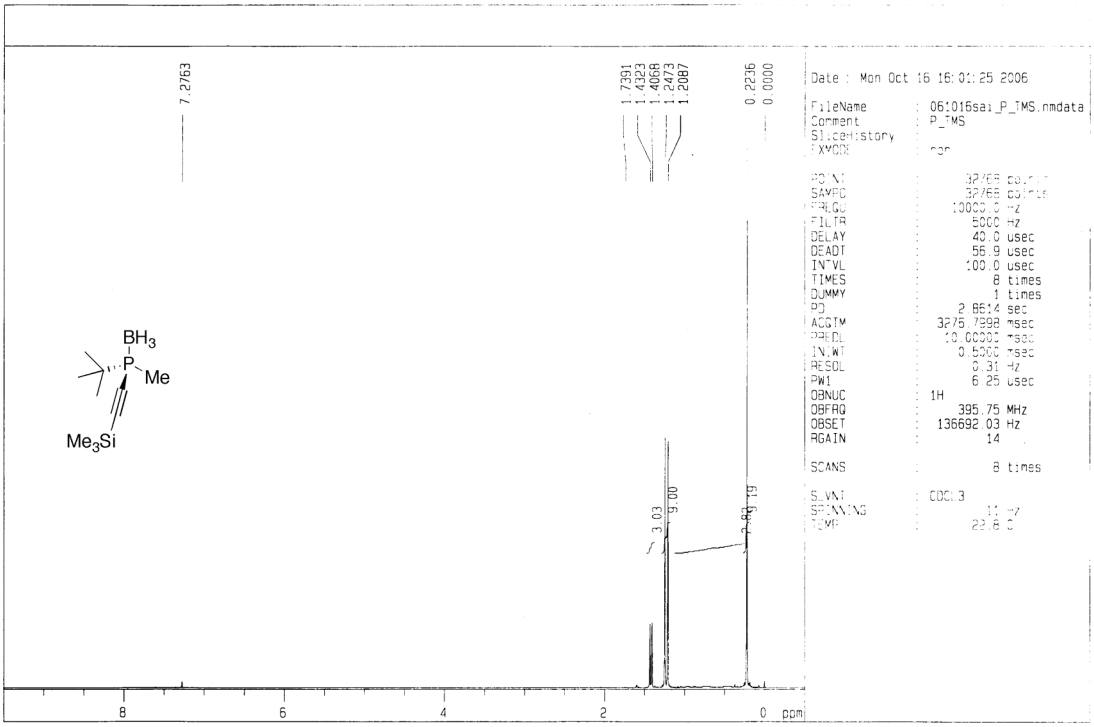
SPINNING : 13 Hz
TEMP : 21.1 C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DUMMY : 1 times
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PREDL : 10.00000 msec
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RESOL : 0.31 Hz
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OBNUC : 1H
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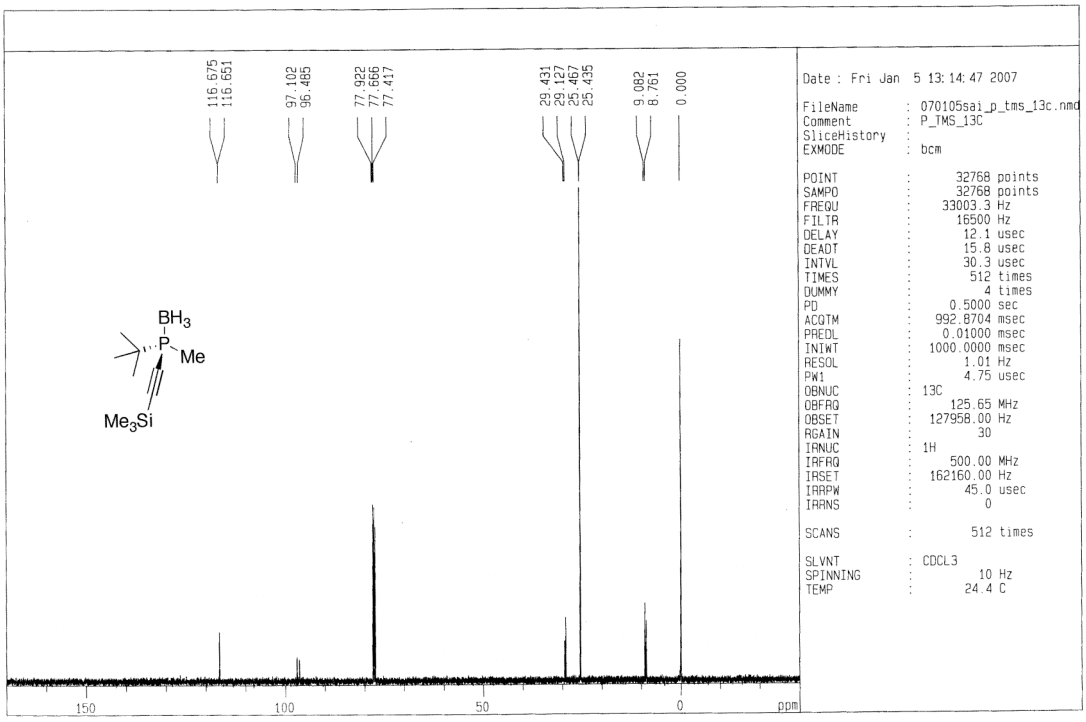
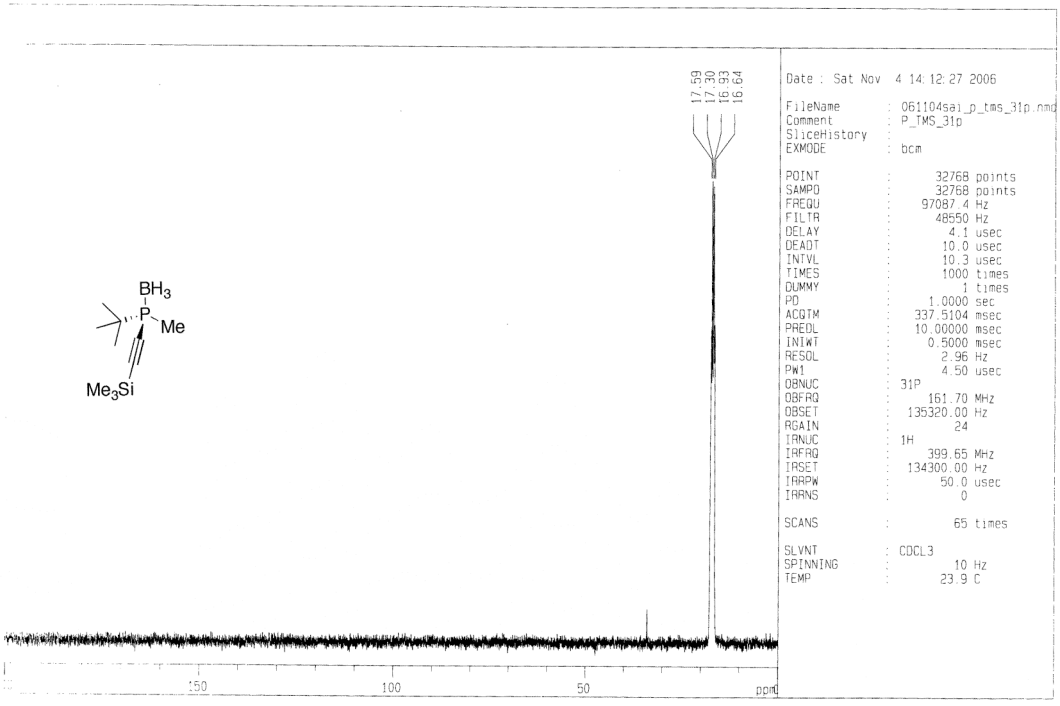
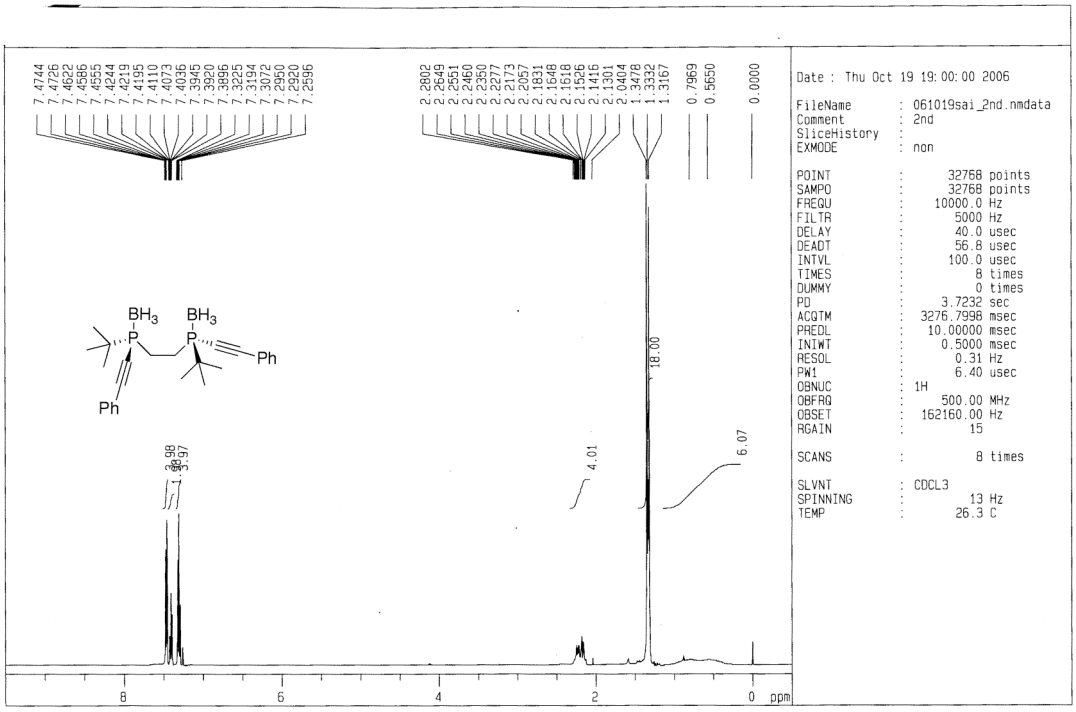
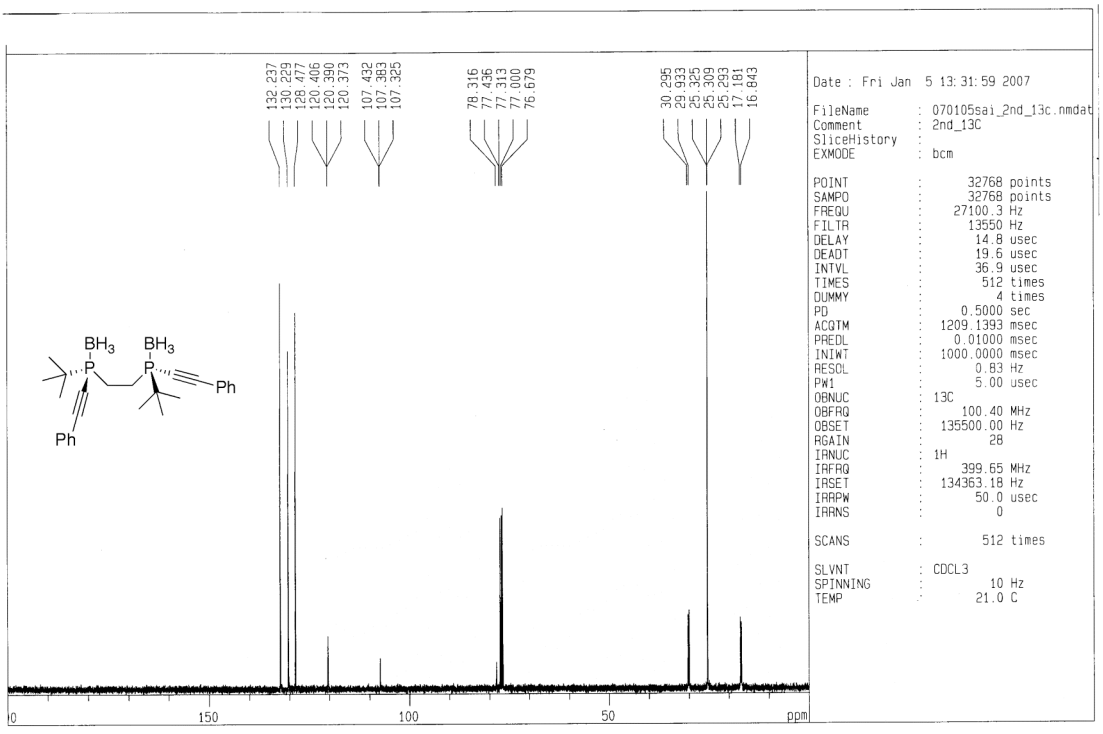
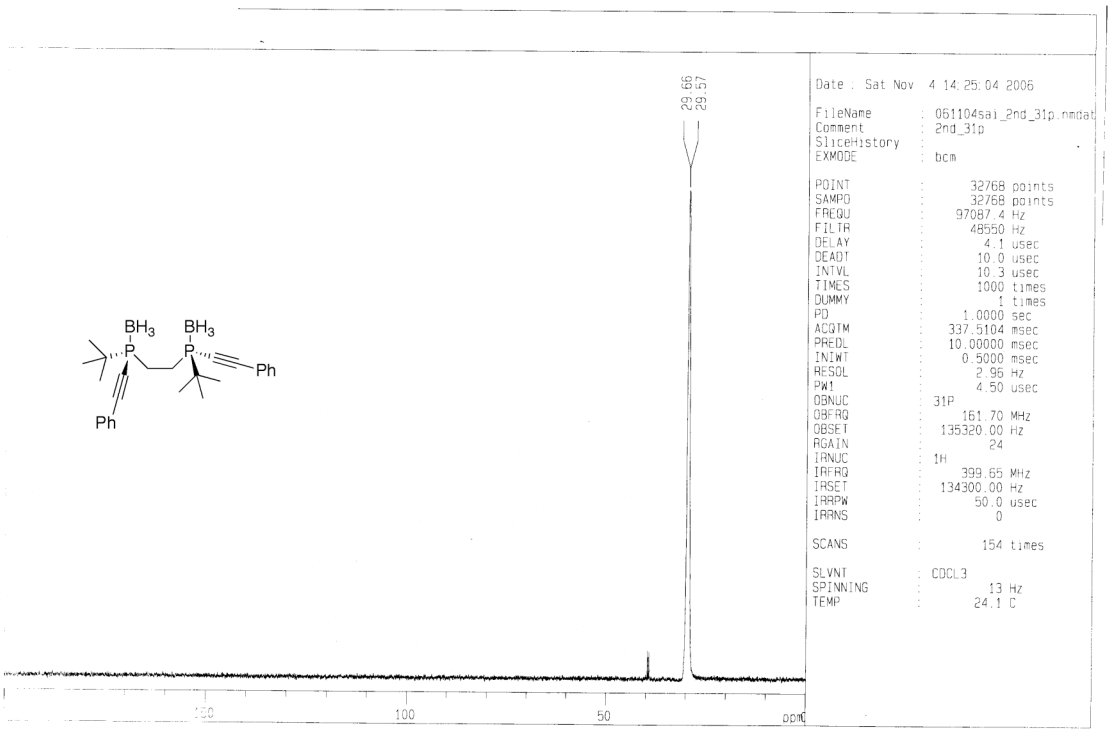
TEMP : 23.8 C

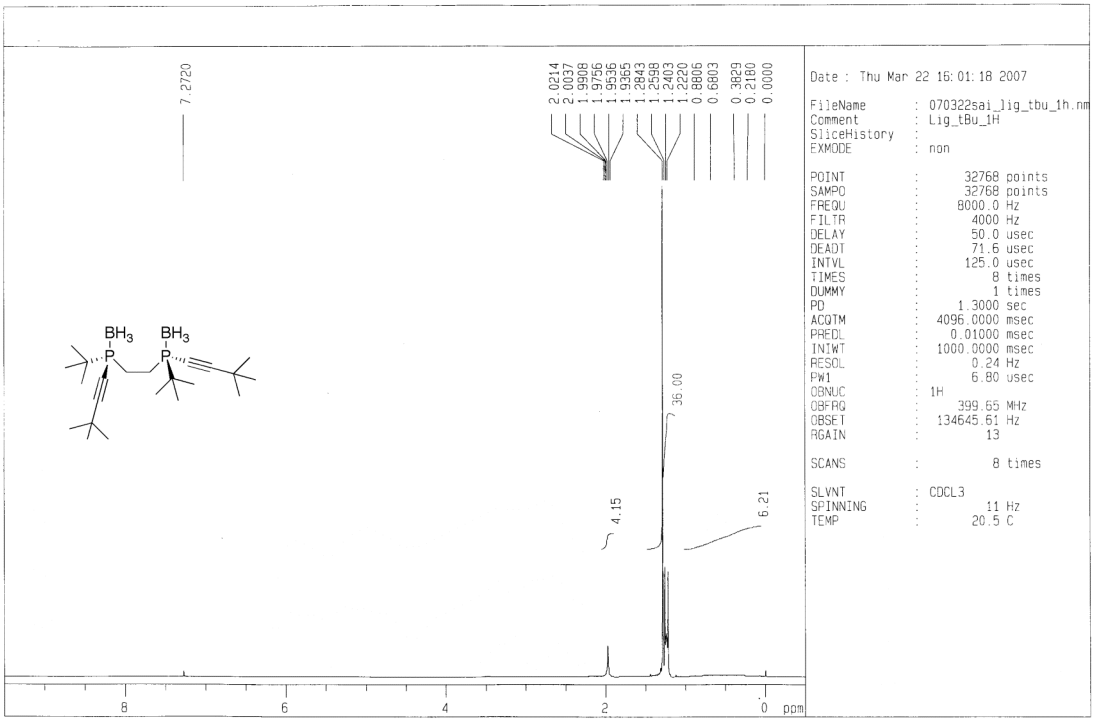

116.675
116.651
97.102
96.485
77.922
77.666
77.417
29.431
29.127
25.467
25.435
9.082
8.761
0.000
BH3
P
Me
Me3Si
150
100
50
0
ppm
Date : Fri Jan 5 13:14:47 2007
FileName : 070105sai_p_tms_13c.nmd
Comment : P_TMS_13C
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 33003.3 Hz
FILTR : 16500 Hz
DELAY : 12.1 usec
DEADT : 15.8 usec
INTVL : 30.3 usec
TIMES : 512 times
DUMMY : 4 times
PD : 0.5000 sec
ACQTM : 992.8704 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 1.01 Hz
PW1 : 4.75 usec
OBNUC : 13C
OBFRQ : 125.65 MHz
OBSET : 127958.00 Hz
RGAIN : 30
IRNUC : 1H
IRFRQ : 500.00 MHz
IRSET : 162160.00 Hz
IRPW : 45.0 usec
IRNS : 0
SCANS : 512 times
SLVNT : CDCL3
SPINNING : 10 Hz
TEMP : 24.4 C

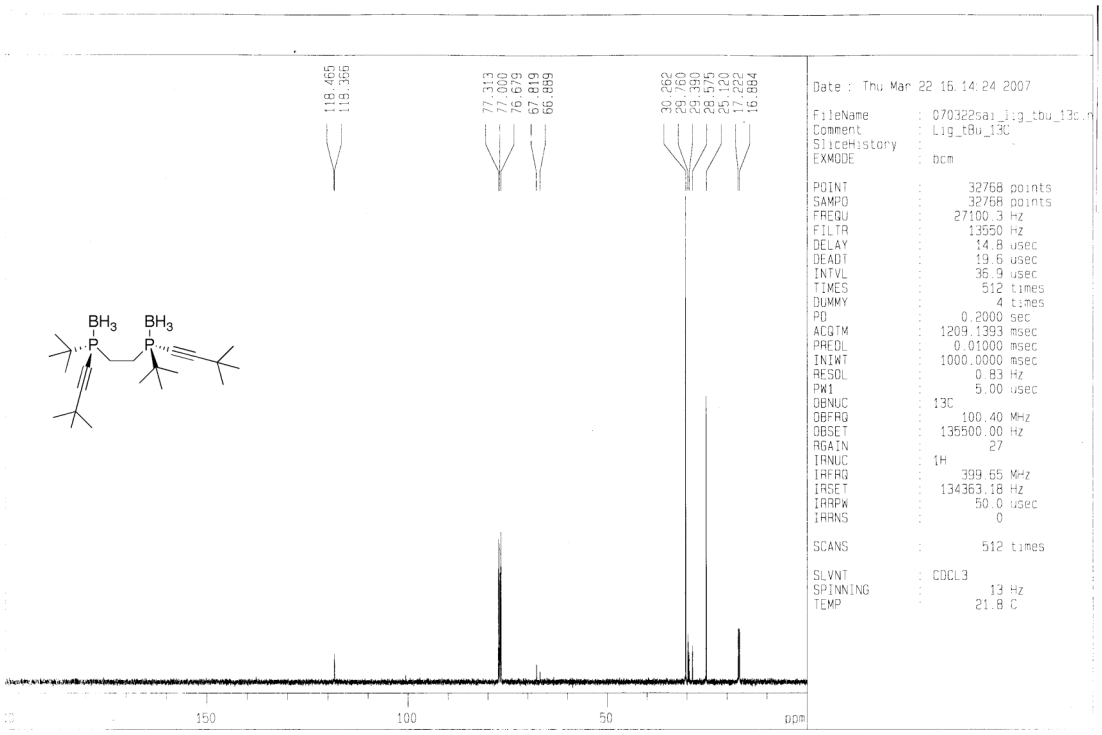

17.59
17.30
16.93
16.64
BH3
P
Me
Me3Si
150
100
50
ppm
Date : Sat Nov 4 14:12:27 2006
FileName : 061104sai_p_tms_31p.nmd
Comment : P_TMS_31p
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 97087.4 Hz
FILTR : 48550 Hz
DELAY : 4.1 usec
DEADT : 10.0 usec
INTVL : 10.3 usec
TIMES : 1000 times
DUMMY : 1 times
PD : 1.0000 sec
ACQTM : 337.5104 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 2.96 Hz
PW1 : 4.50 usec
OBNUC : 31P
OBFRQ : 161.70 MHz
OBSET : 135320.00 Hz
RGAIN : 24
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134300.00 Hz
IRPW : 50.0 usec
IRNS : 0
SCANS : 65 times
SLVNT : CDCL3
SPINNING : 10 Hz
TEMP : 23.9 C

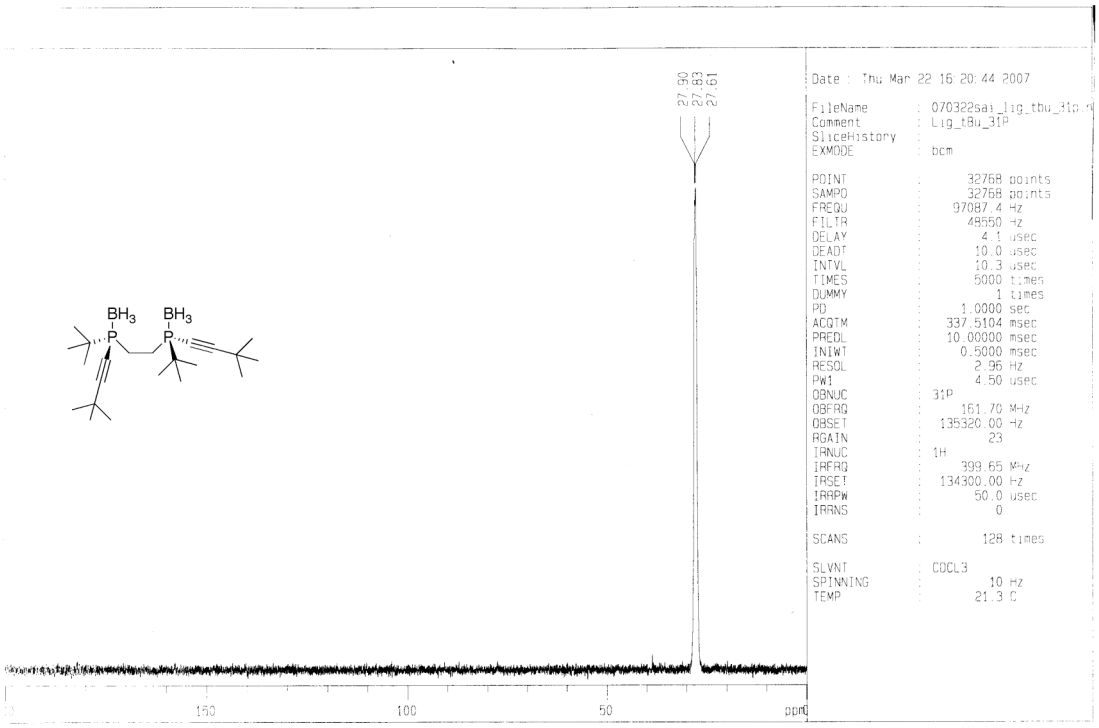

7.4744
7.4726
7.4622
7.4586
7.4555
7.4244
7.4219
7.4195
7.4110
7.4073
7.4036
7.3945
7.3920
7.3896
7.3225
7.3194
7.3072
7.2950
7.2920
7.2596
2.2802
2.2649
2.2561
2.2460
2.2350
2.2277
2.2173
2.2057
2.1831
2.1648
2.1618
2.1526
2.1416
2.1301
2.0404
1.3478
1.3332
1.3167
0.7969
0.5650
0.0000
BH3
BH3
P
P
Ph
Ph
2.98
3.00
3.97
4.01
18.00
6.07
8
6
4
2
0 ppm
Date : Thu Oct 19 19:00:00 2006
FileName : 061019sai_2nd.nmdata
Comment : 2nd
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 56.8 usec
INTVL : 100.0 usec
TIMES : 8 times
DUMMY : 0 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 0.31 Hz
PW1 : 6.40 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
RGAIN : 15
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 26.3 C

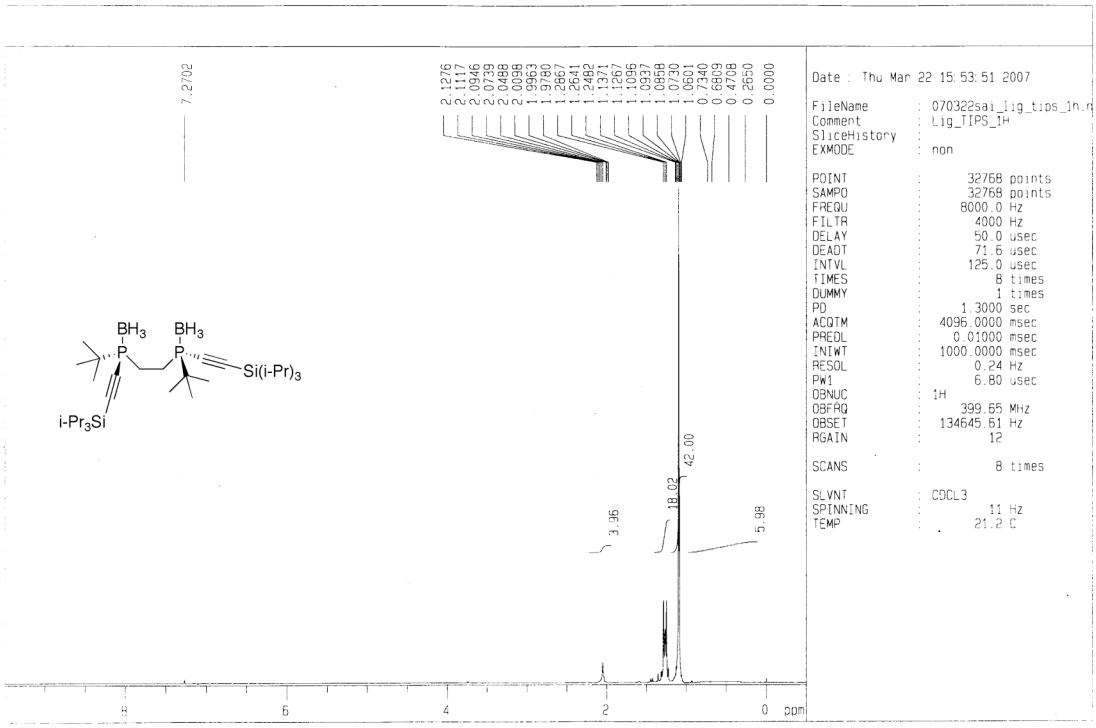

132.237
130.229
128.477
128.406
128.390
128.373
107.432
107.383
107.325
78.316
77.436
77.313
77.000
76.679
30.295
29.933
25.325
25.309
25.293
17.181
16.843
BH3
BH3
P
P
Ph
Ph
150
100
50
ppm
Date : Fri Jan 5 13:31:59 2007
FileName : 070105sai_2nd_13c.nmdat
Comment : 2nd_13C
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 27100.3 Hz
FILTR : 13550 Hz
DELAY : 14.8 usec
DEADT : 19.6 usec
INTVL : 36.9 usec
TIMES : 512 times
DUMMY : 4 times
PD : 0.5000 sec
ACQTM : 1209.1393 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.83 Hz
PW1 : 5.00 usec
OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
RGAIN : 28
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134363.18 Hz
IRRPW : 50.0 usec
IRRNS : 0
SCANS : 512 times
SLVNT : CDCL3
SPINNING : 10 Hz
TEMP : 21.0 C

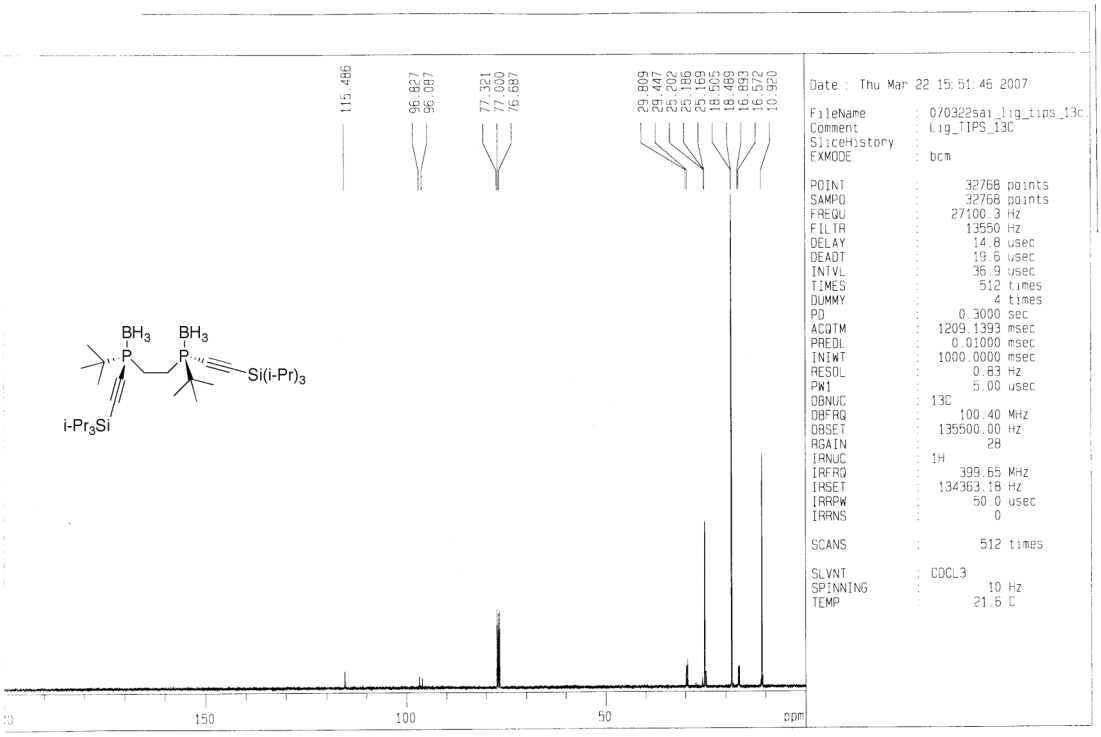

29.66
29.57
BH3
BH3
P
P
Ph
Ph
100
50
ppm
Date : Sat Nov 4 14:25:04 2006
FileName : 061104sai_2nd_31p.nmdat
Comment : 2nd_31p
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 97087.4 Hz
FILTR : 48550 Hz
DELAY : 4.1 usec
DEADT : 10.0 usec
INTVL : 10.3 usec
TIMES : 1000 times
DUMMY : 1 times
PD : 1.0000 sec
ACQTM : 337.5104 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 2.96 Hz
PW1 : 4.50 usec
OBNUC : 31P
OBFRQ : 161.70 MHz
OBSET : 135320.00 Hz
RGAIN : 24
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134300.00 Hz
IRPW : 50.0 usec
IRRNS : 0
SCANS : 154 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 24.1 C

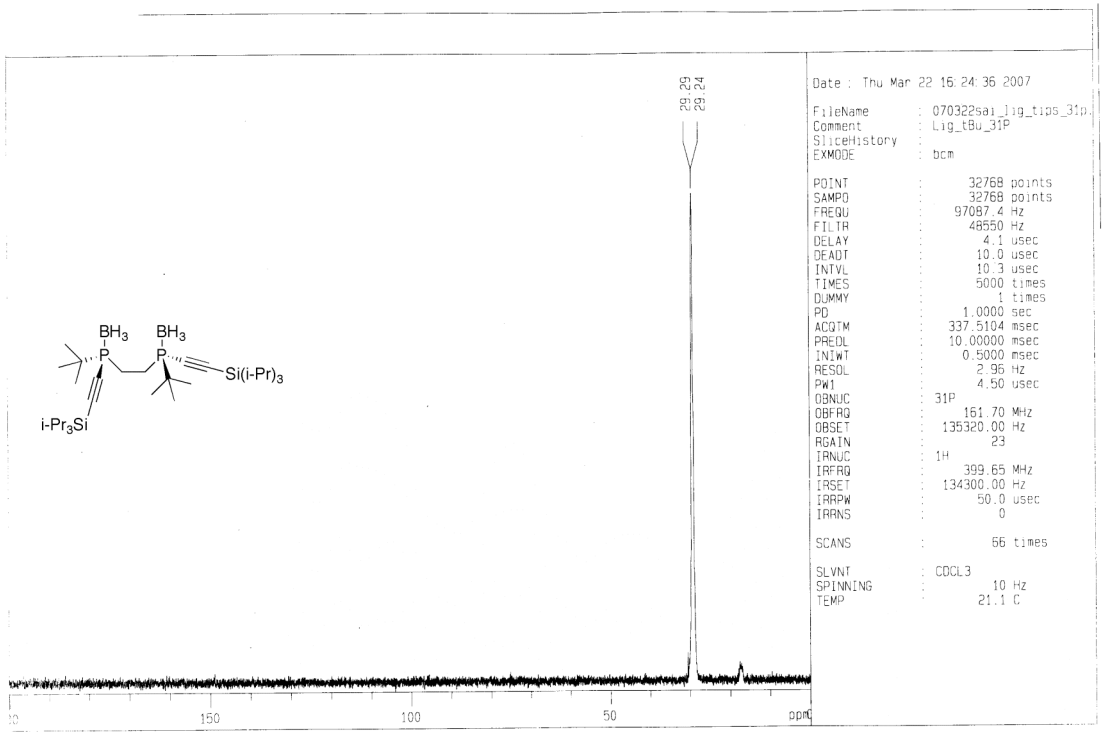

7.2720
2.0214
2.0037
1.9908
1.9756
1.9536
1.9365
1.2843
1.2598
1.2403
1.2220
0.8806
0.6803
0.3829
0.2180
0.0000
36.00
4.15
6.21
BH3
BH3
P
P
8
6
4
2
0
ppm
Date : Thu Mar 22 16:01:18 2007
FileName : 070322sai_lig_tbu_1h.nm
Comment : Lig_tBu_1H
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 8000.0 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 71.6 usec
INTVL : 125.0 usec
TIMES : 8 times
DUMMY : 1 times
PD : 1.3000 sec
ACQTM : 4096.0000 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 6.80 usec
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134645.61 Hz
RGAIN : 13
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 11 Hz
TEMP : 20.5 C


118.465
118.356
77.313
77.000
76.679
67.819
66.889
30.262
29.760
29.490
28.575
25.120
17.222
16.884
BH3
BH3
P
P
150
100
50
ppm
Date : Thu Mar 22 16:14:24 2007
FileName : 070322sai_lig_tbu_13c.n
Comment : Lig_tBu_13C
SliceHistory :
EXMODE : bcm
PDINT : 32768 points
SAMPO : 32768 points
FREQU : 27100.3 Hz
FILTR : 13550 Hz
DELAY : 14.8 usec
DEADT : 19.6 usec
INTVL : 36.9 usec
TIMES : 512 times
DUMMY : 4 times
PD : 0.2000 sec
ACQTM : 1209.1393 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.83 Hz
PW1 : 5.00 usec
OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
RGAIN : 27
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134363.18 Hz
IRRPW : 50.0 usec
IRRNS : 0
SCANS : 512 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 21.8 C


27.90
27.83
27.61
BH3
BH3
P
P
150
100
50
ppm
Date : Thu Mar 22 16:20:44 2007
FileName : 070322sai_lig_tbu_31p.n
Comment : Lig_tBu_31P
SliceHistory :
EXMODE : bcm
PDINT : 32768 points
SAMPO : 32768 points
FREQU : 97087.4 Hz
FILTR : 48550 Hz
DELAY : 4.1 usec
DEADT : 10.0 usec
INTVL : 10.3 usec
TIMES : 5000 times
DUMMY : 1 times
PD : 1.0000 sec
ACQTM : 337.5104 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 2.96 Hz
PW1 : 4.50 usec
OBNUC : 31P
OBFRQ : 161.70 MHz
OBSET : 135320.00 Hz
RGAIN : 23
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134300.00 Hz
IRRPW : 50.0 usec
IRRNS : 0
SCANS : 128 times
SLVNT : CDCL3
SPINNING : 10 Hz
TEMP : 21.3 C

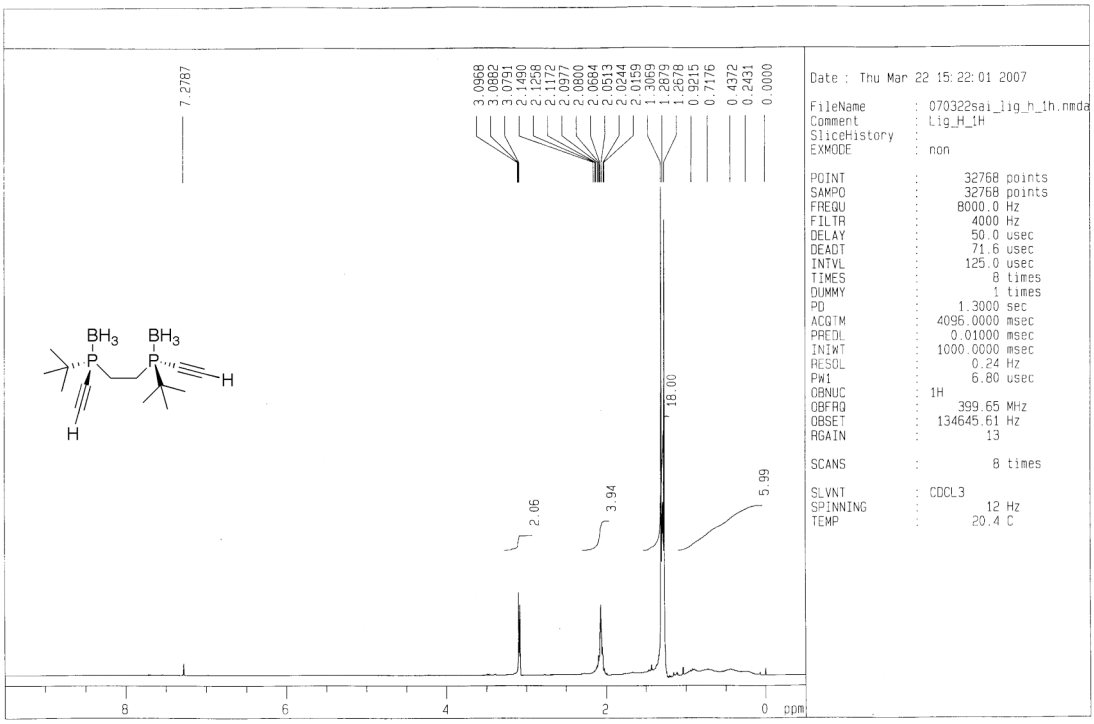

7.2702
2.1276
2.1117
2.0946
2.0739
2.0488
2.0096
1.9963
1.9780
1.2867
1.2641
1.2482
1.1371
1.1267
1.1096
1.0937
1.0858
1.0730
1.0601
0.7340
0.6809
0.4708
0.2650
0.0000
3.96
18.02
42.00
5.98
BH3
BH3
P
P
Si(i-Pr)3
i-Pr3Si
8
6
4
2
0 ppm
Date : Thu Mar 22 15:53:51 2007
FileName : 070322sai_lig_tips_1h.n
Comment : Lig_TIPS_1H
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 8000.0 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 71.6 usec
INTVL : 125.0 usec
TIMES : 8 times
DUMMY : 1 times
PD : 1.3000 sec
ACQTM : 4096.0000 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 6.80 usec
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134645.51 Hz
RGAIN : 12
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 11 Hz
TEMP : 21.2 C


115.486
96.827
96.087
77.321
77.000
76.687
29.809
29.447
29.202
25.186
25.169
18.565
18.489
16.693
16.572
10.920
BH3
BH3
P
P
Si(i-Pr)3
i-Pr3Si
150
100
50
ppm
Date : Thu Mar 22 15:51:46 2007
FileName : 070322sai_lig_tips_13c
Comment : Lig_TIPS_13C
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 27100.3 Hz
FILTR : 13550 Hz
DELAY : 14.8 usec
DEADT : 19.6 usec
INTVL : 36.9 usec
TIMES : 512 times
DUMMY : 4 times
PD : 0.3000 sec
ACQTM : 1209.1393 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.83 Hz
PW1 : 5.00 usec
OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
RGAIN : 28
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134363.18 Hz
IRRPW : 50.0 usec
IRRNS : 0
SCANS : 512 times
SLVNT : CDCL3
SPINNING : 10 Hz
TEMP : 21.6 C



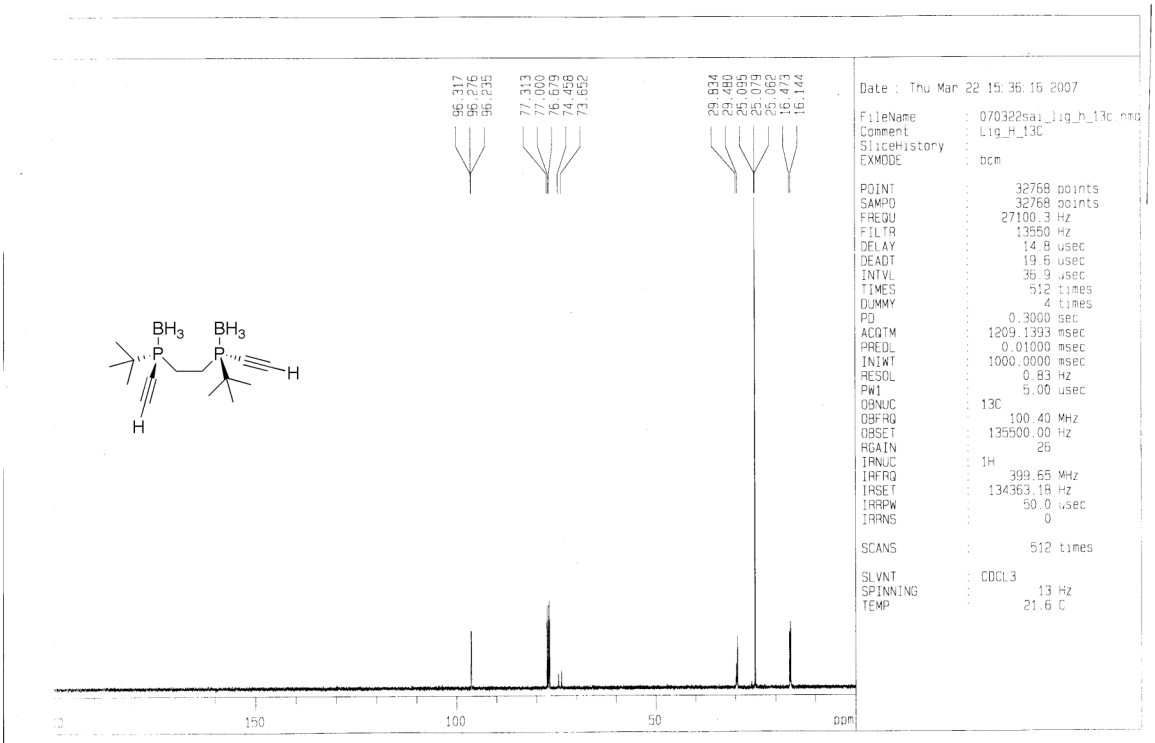
Date : Thu Mar 22 15:24:36 2007

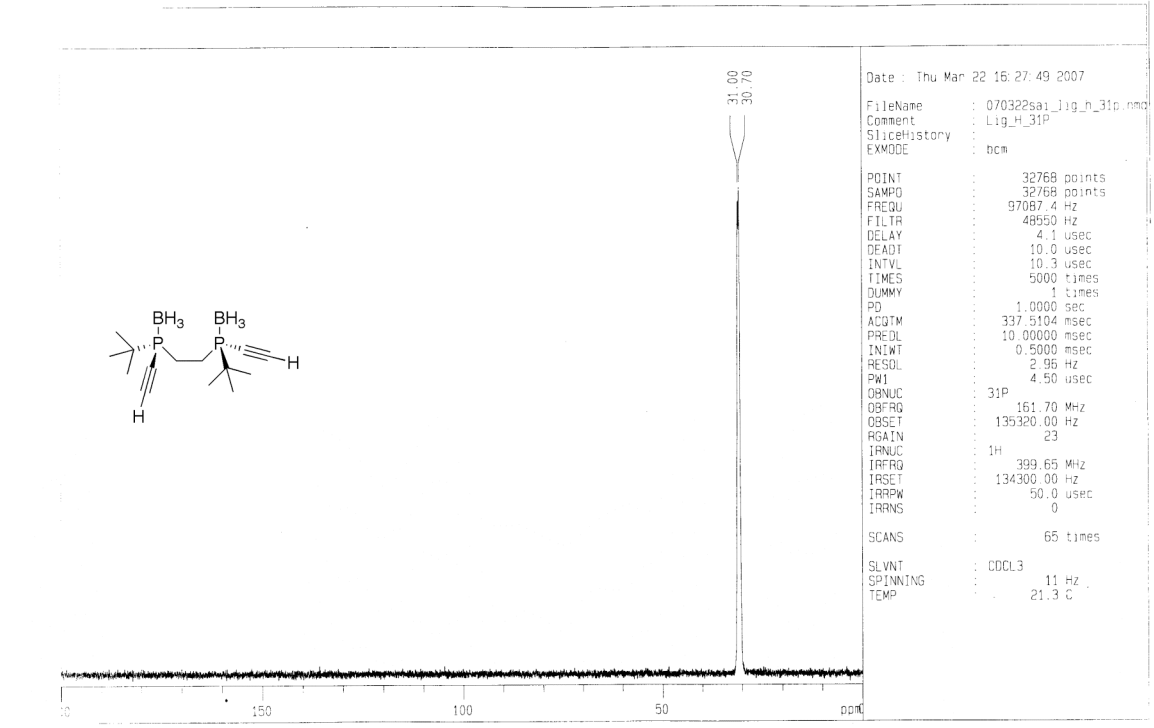
FileName	:	070322sai_lig_tips_31p
Comment	:	Lig_tBu_31P
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	97087.4 Hz
FILTR	:	48550 Hz
DELAY	:	4.1 usec
DEADT	:	10.0 usec
INTVL	:	10.3 usec
TIMES	:	5000 times
DUMMY	:	1 times
PD	:	1.0000 sec
ACQTM	:	337.5104 msec
PREDL	:	10.00000 msec
INIWT	:	0.5000 msec
RESOL	:	2.96 Hz
PW1	:	4.50 usec
OBNUC	:	31P
OBFRQ	:	161.70 MHz
OBSET	:	135320.00 Hz
RGAIN	:	23
IRNUC	:	1H
IRFRQ	:	399.65 MHz
IRSET	:	134300.00 Hz
IRPW	:	50.0 usec
IRNS	:	0
SCANS	:	66 times
SLVNT	:	CDCL3
SPINNING	:	10 Hz
TEMP	:	21.1 C

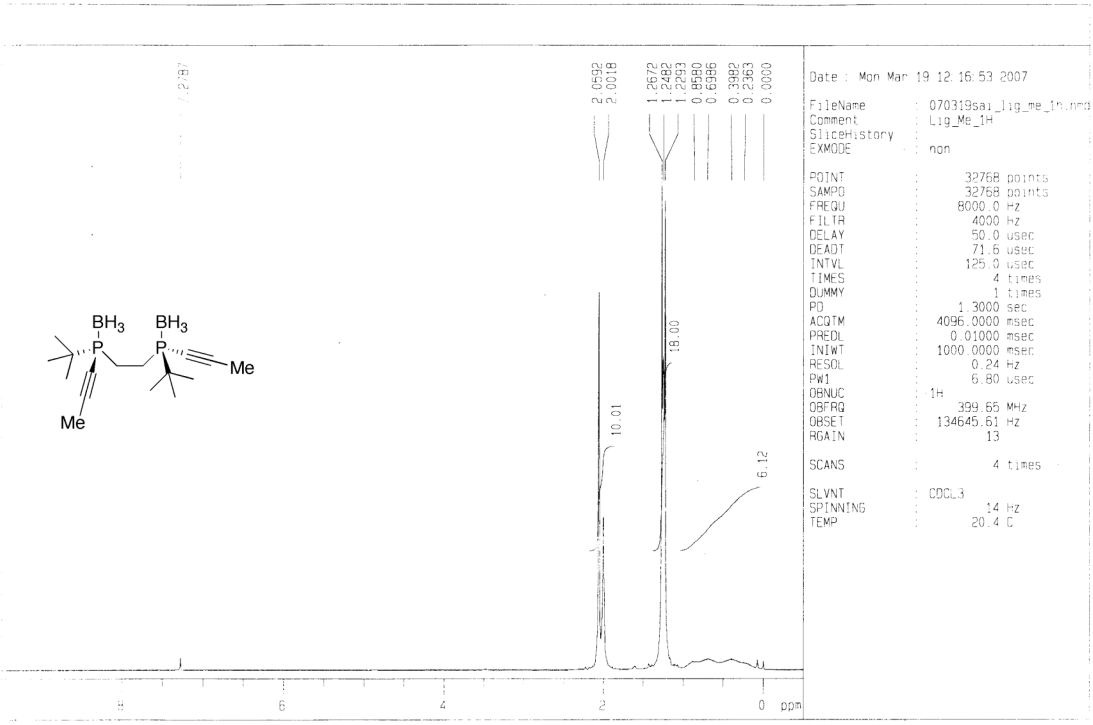


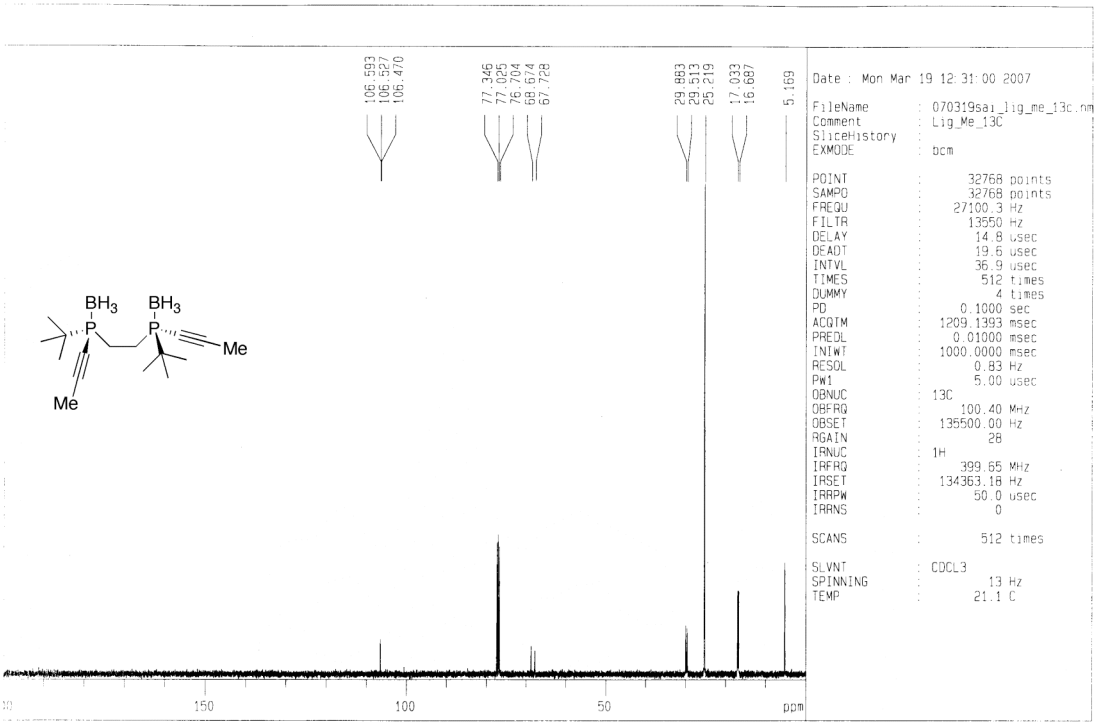
Date : Thu Mar 22 15:22:01 2007

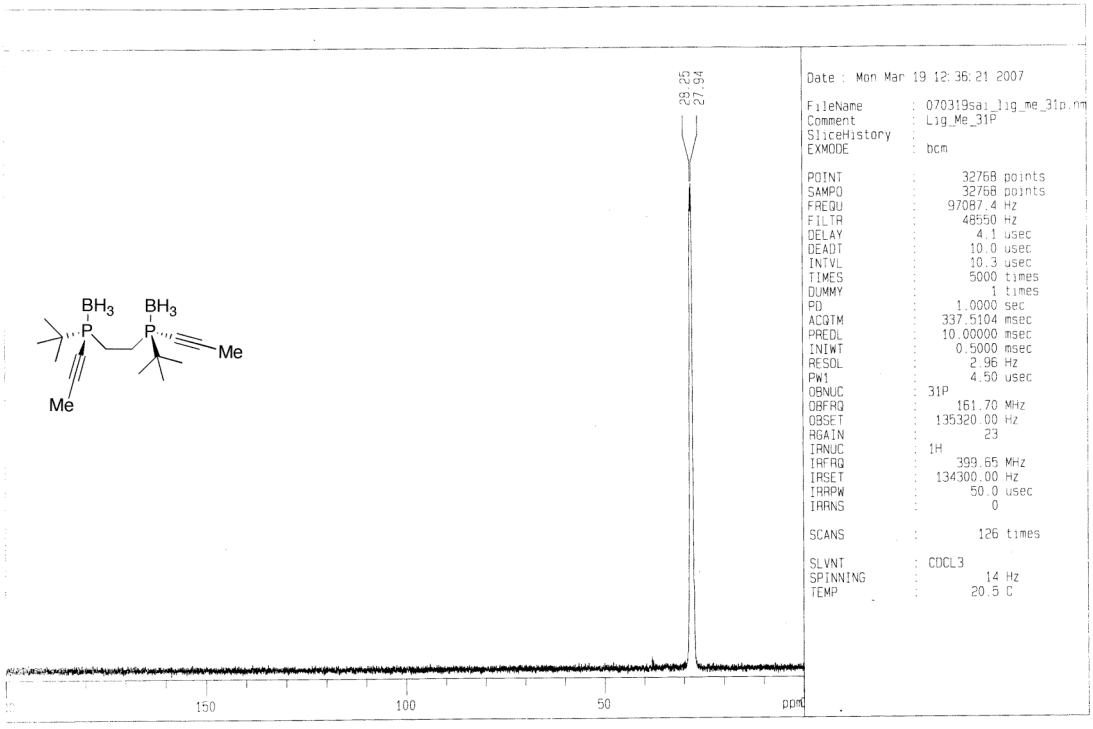
FileName	:	070322sai_lig_h_1h.nmda
Comment	:	Lig_H_1H
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	8000.0 Hz
FILTR	:	4000 Hz
DELAY	:	50.0 usec
DEADT	:	71.6 usec
INTVL	:	125.0 usec
TIMES	:	8 times
DUMMY	:	1 times
PD	:	1.3000 sec
ACQTM	:	4096.0000 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	6.80 usec
OBNUC	:	1H
OBFRQ	:	399.65 MHz
OBSET	:	134645.61 Hz
RGAIN	:	13
SCANS	:	8 times
SLVNT	:	CDCL3
SPINNING	:	12 Hz
TEMP	:	20.4 C

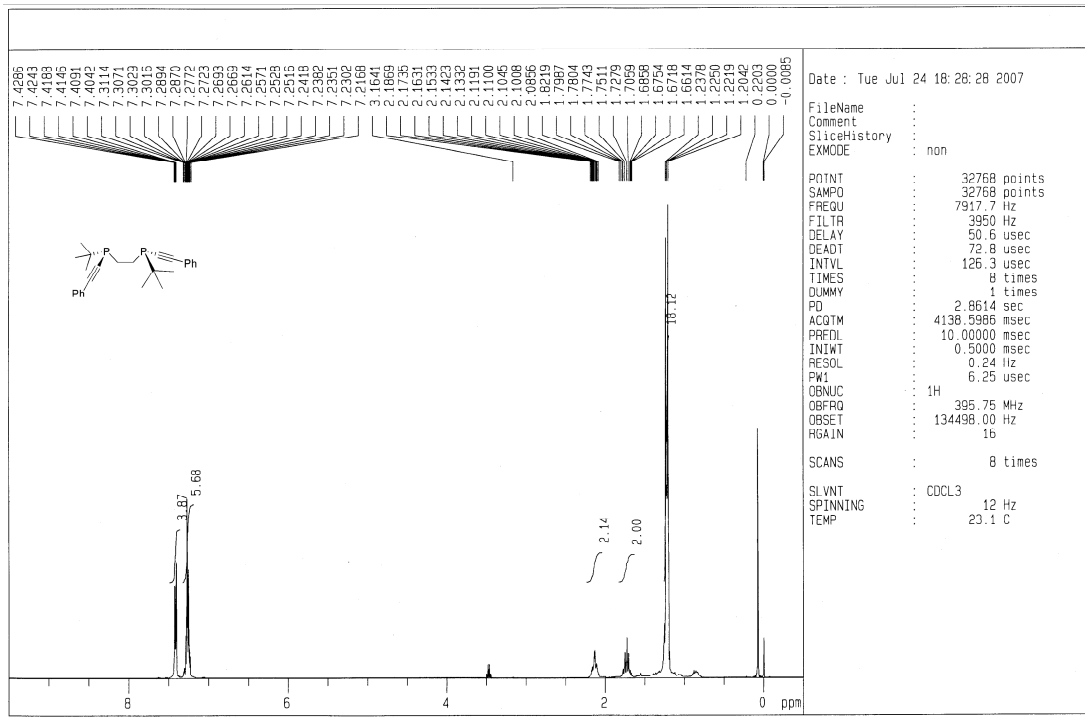

96.317
96.276
96.235
77.313
77.000
76.679
74.468
73.652
29.834
29.480
25.095
25.079
25.062
16.473
16.144
BH3
BH3
P
P
H
H
150
100
50
ppm
Date : Thu Mar 22 15:36:16 2007
FileName : 070322sai_lig_h_13c.nmr
Comment : Lig_H_13C
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 27100.3 Hz
FILTR : 13550 Hz
DELAY : 14.8 usec
DEADT : 19.6 usec
INTVL : 36.9 usec
TIMES : 512 times
DUMMY : 4 times
PD : 0.3000 sec
ACQTM : 1209.1393 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.83 Hz
PW1 : 5.00 usec
OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
RGAIN : 26
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134363.18 Hz
IRRPW : 50.0 usec
IRRNS : 0
SCANS : 512 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 21.6 C

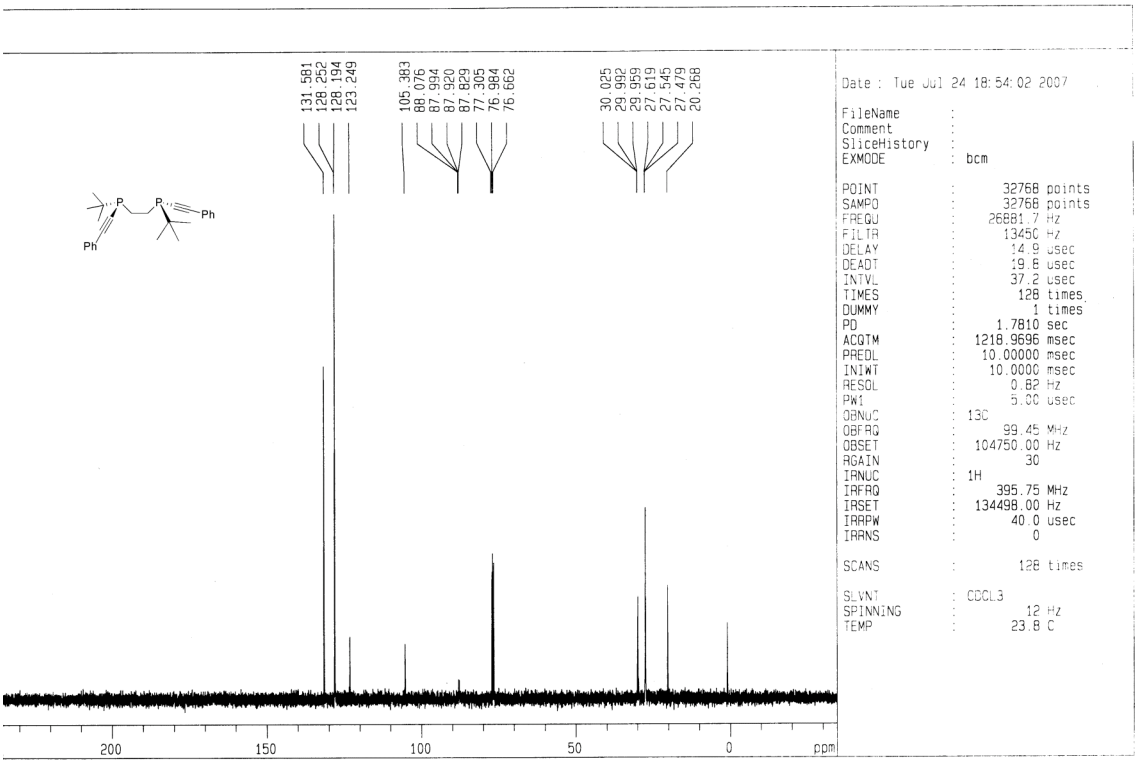

31.00
30.70
BH3
BH3
P
P
H
H
150
100
50
ppm
Date : Thu Mar 22 16:27:49 2007
FileName : 070322sai_lig_h_31p.nmr
Comment : Lig_H_31P
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 97087.4 Hz
FILTR : 48550 Hz
DELAY : 4.1 usec
DEADT : 10.0 usec
INTVL : 10.3 usec
TIMES : 5000 times
DUMMY : 1 times
PD : 1.0000 sec
ACQTM : 337.5104 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 2.96 Hz
PW1 : 4.50 usec
OBNUC : 31P
OBFRQ : 161.70 MHz
OBSET : 135320.00 Hz
RGAIN : 23
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134300.00 Hz
IRRPW : 50.0 usec
IRRNS : 0
SCANS : 65 times
SLVNT : CDCL3
SPINNING : 11 Hz
TEMP : 21.3 C

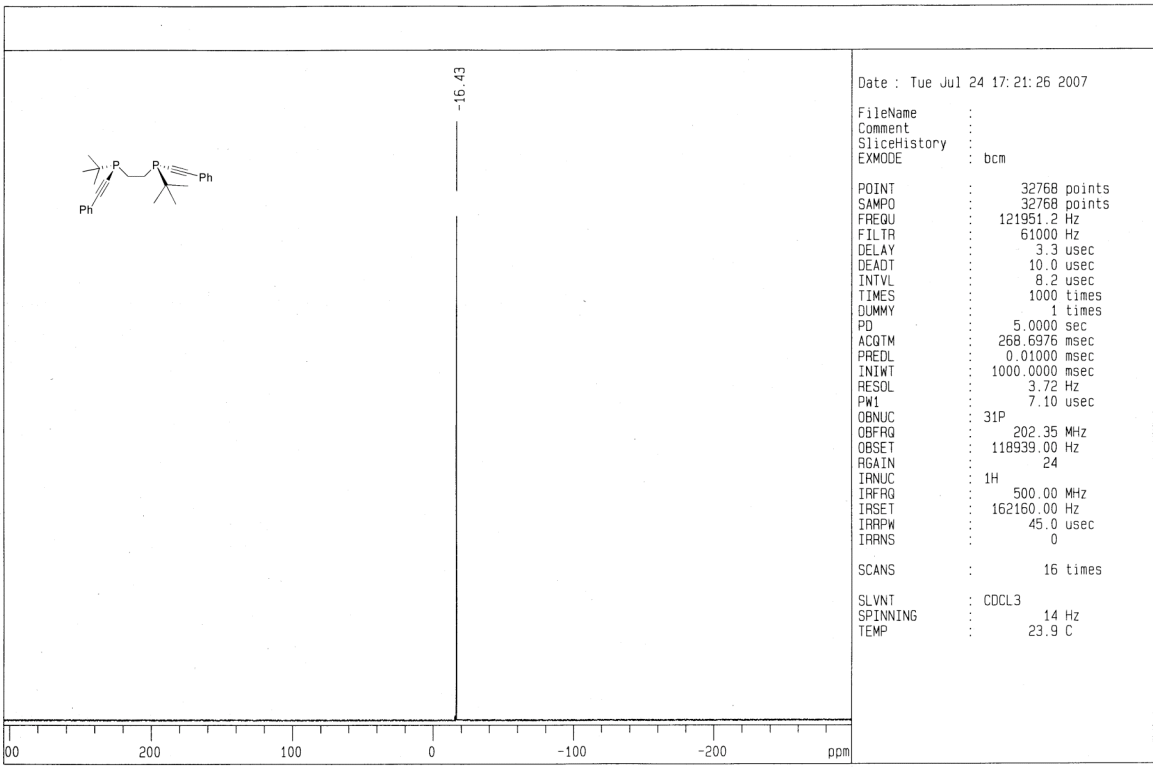

BH3
BH3
P
P
Me
Me
2.0592
2.0018
1.2672
1.2482
1.2293
0.8580
0.6986
0.3982
0.2363
0.0000
18.00
10.01
6.12
6
4
2
0 ppm
Date : Mon Mar 19 12:16:53 2007
FileName : 070319sai_lig_me_1h.nmr
Comment : Lig_Me_1H
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 8000.0 Hz
FILTR : 4000 Hz
DELAY : 50.0 usec
DEADT : 71.6 usec
INTVL : 125.0 usec
TIMES : 4 times
DUMMY : 1 times
PD : 1.3000 sec
ACQTM : 4096.0000 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.24 Hz
PW1 : 6.80 usec
OBNUC : 1H
OBFRQ : 399.65 MHz
OBSET : 134645.61 Hz
RGAIN : 13
SCANS : 4 times
SLVNT : CDCL3
SPINNING : 14 Hz
TEMP : 20.4 C

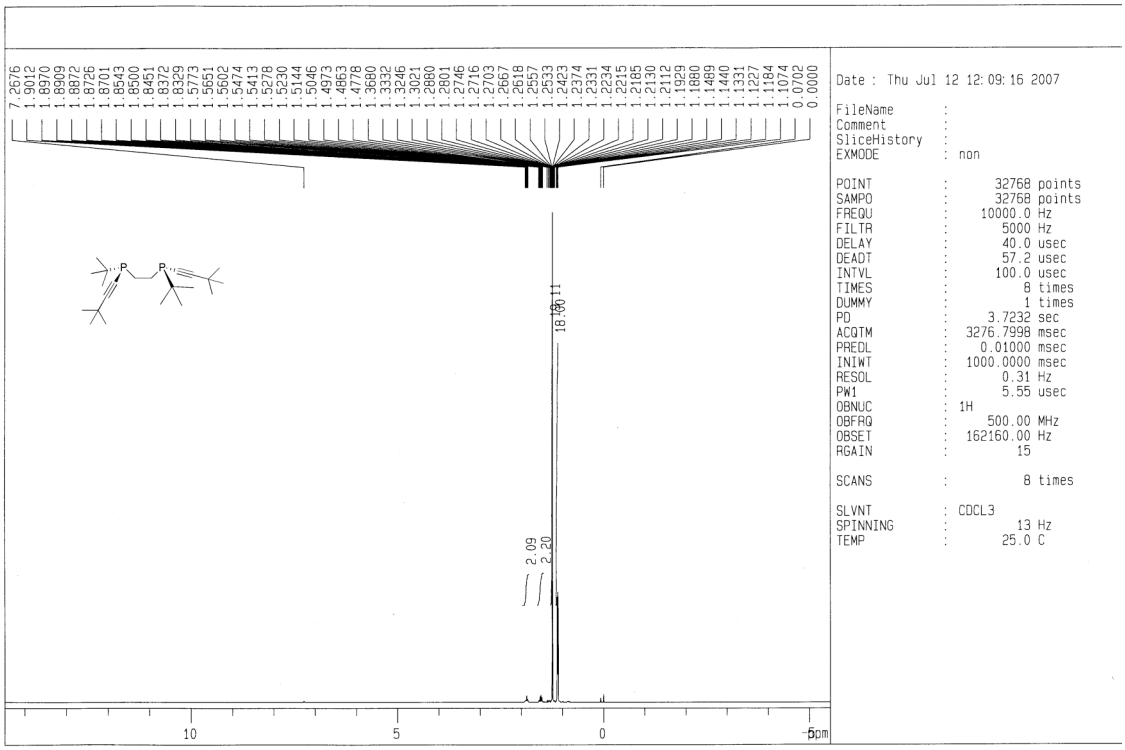

BH3
BH3
P
P
Me
Me
106.593
106.527
106.470
77.346
77.025
76.704
68.674
67.728
29.863
29.513
25.219
17.033
16.687
5.169
150
100
50
ppm
Date : Mon Mar 19 12:31:00 2007
FileName : 070319sai_lig_me_13c.nm
Comment : Lig_Me_13C
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 27100.3 Hz
FILTR : 13550 Hz
DELAY : 14.8 usec
DEADT : 19.6 usec
INTVL : 36.9 usec
TIMES : 512 times
DUMMY : 4 times
PD : 0.1000 sec
ACQTM : 1209.1393 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.83 Hz
PW1 : 5.00 usec
OBNUC : 13C
OBFRQ : 100.40 MHz
OBSET : 135500.00 Hz
RGAIN : 28
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134363.18 Hz
IRRPW : 50.0 usec
IRRNS : 0
SCANS : 512 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 21.1 C

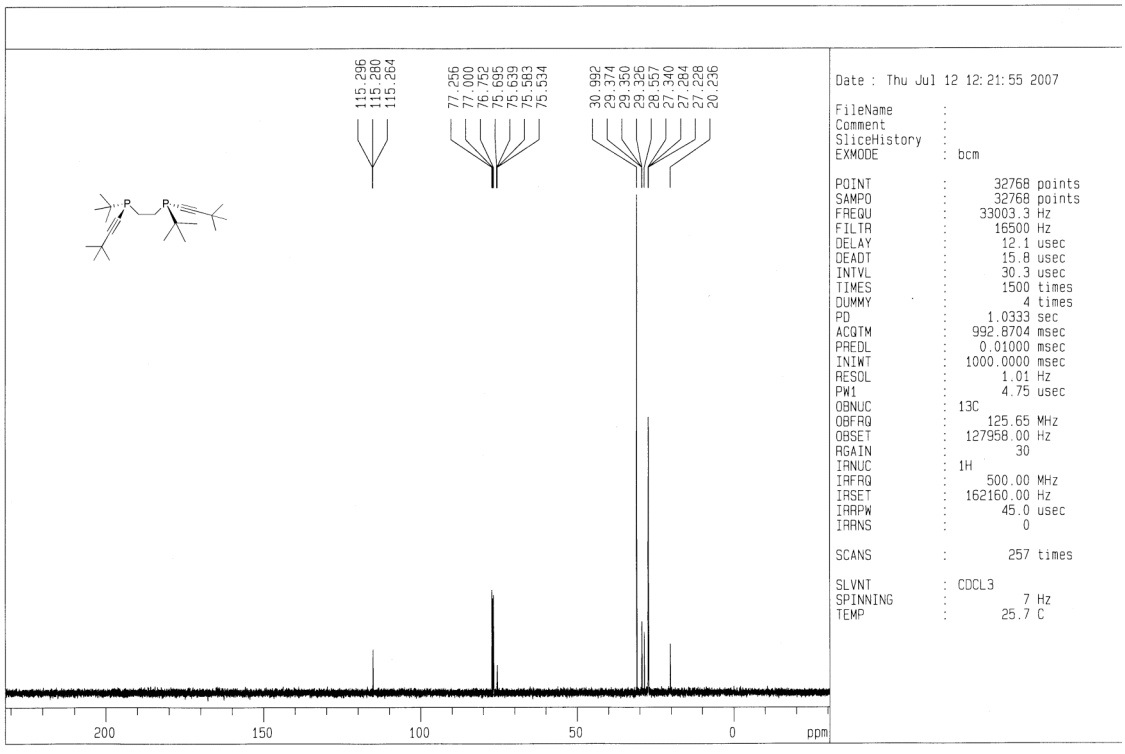

29.25
27.94
BH3
BH3
Me
Me
150
100
50
ppm
Date : Mon Mar 19 12:36:21 2007
FileName : 070319sai_lig_me_31p.nm
Comment : Lig_Me_31P
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 97087.4 Hz
FILTR : 48550 Hz
DELAY : 4.1 usec
DEADT : 10.0 usec
INTVL : 10.3 usec
TIMES : 5000 times
DUMMY : 1 times
PD : 1.0000 sec
ACQTM : 337.5104 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 2.96 Hz
PW1 : 4.50 usec
OBNUC : 31P
OBFRQ : 161.70 MHz
OBSET : 135320.00 Hz
RGAIN : 23
IRNUC : 1H
IRFRQ : 399.65 MHz
IRSET : 134300.00 Hz
IRRPW : 50.0 usec
IRRNS : 0
SCANS : 126 times
SLVNT : CDCL3
SPINNING : 14 Hz
TEMP : 20.5 C

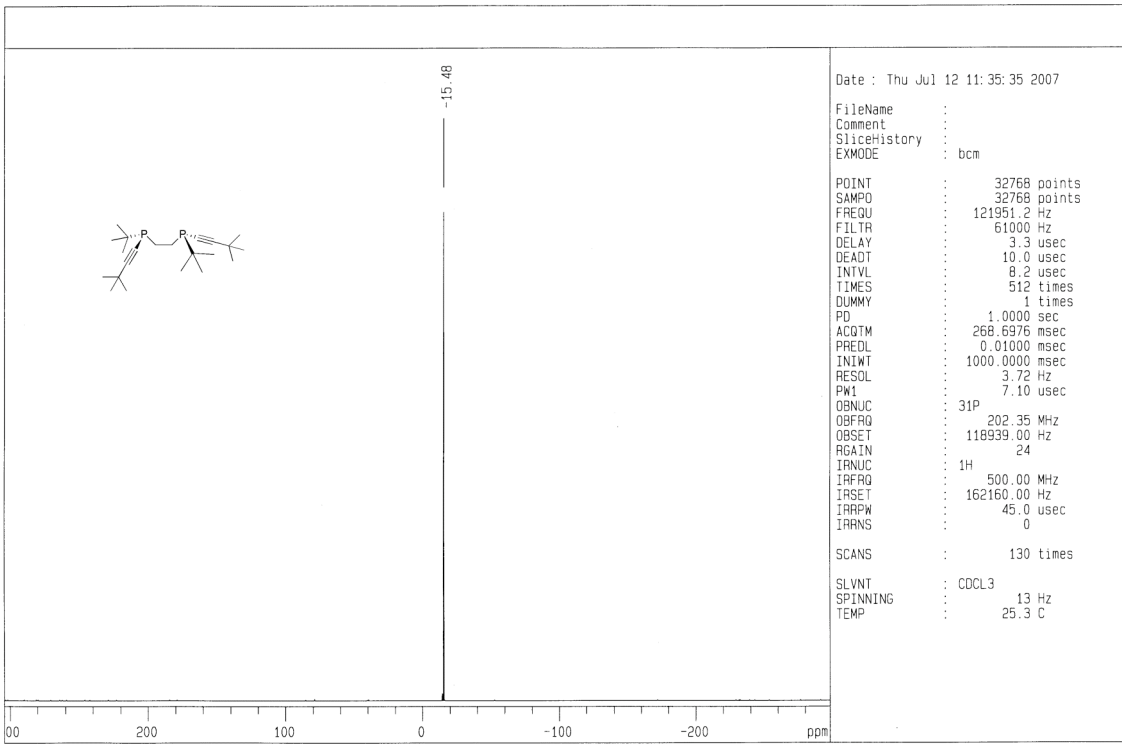

7.4266
7.4243
7.4188
7.4146
7.4091
7.4042
7.3114
7.3071
7.3023
7.3015
7.2894
7.2870
7.2772
7.2723
7.2693
7.2663
7.2614
7.2571
7.2528
7.2515
7.2418
7.2382
7.2351
7.2302
7.2168
3.1641
2.1869
2.1735
2.1631
2.1533
2.1423
2.1332
2.1191
2.1100
2.1045
2.1008
2.0856
1.8219
1.7987
1.7804
1.7743
1.7511
1.7279
1.7059
1.6858
1.6754
1.6718
1.6614
1.2378
1.2250
1.2219
1.2042
1.2003
0.0000
-0.0065
Ph
Ph
P
P
3.87
5.68
2.14
2.00
18.12
8
6
4
2
0 ppm
Date : Tue Jul 24 18:28:28 2007
FileName :
Comment :
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7917.7 Hz
FILTR : 3950 Hz
DELAY : 50.6 usec
DEADT : 72.8 usec
INTVL : 126.3 usec
TIMES : 8 times
DUMMY : 1 times
PD : 2.8614 sec
ACQTM : 4138.5986 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 0.24 Hz
PW1 : 6.25 usec
OBNUC : 1H
OBFRQ : 395.75 MHz
OBSET : 134498.00 Hz
RGAIN : 16
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 12 Hz
TEMP : 23.1 C


131.581
128.252
128.194
123.249
105.383
88.076
87.994
87.920
87.829
77.305
76.984
76.662
30.025
29.992
29.959
27.619
27.545
27.479
20.268
Ph
Ph
200
150
100
50
0
ppm
Date : Tue Jul 24 18:54:02 2007
FileName :
Comment :
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 26881.7 Hz
FILTR : 13450 Hz
DELAY : 14.9 usec
DEADT : 19.6 usec
INTVL : 37.2 usec
TIMES : 128 times
DUMMY : 1 times
PD : 1.7810 sec
ACQTM : 1218.9696 msec
PREDL : 10.00000 msec
INIWT : 10.0000 msec
RESOL : 0.82 Hz
PW1 : 5.00 usec
OBNUC : 13C
OBFRQ : 99.45 MHz
OBSET : 104750.00 Hz
RGAIN : 30
IRNUC : 1H
IRFRQ : 395.75 MHz
IRSET : 134498.00 Hz
IRRPW : 40.0 usec
IRRNS : 0
SCANS : 128 times
SLVNT : CDCL3
SPINNING : 12 Hz
TEMP : 23.8 C


-16.43
Ph
Ph
00
200
100
0
-100
-200
ppm
Date : Tue Jul 24 17:21:26 2007
FileName :
Comment :
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 121951.2 Hz
FILTR : 61000 Hz
DELAY : 3.3 usec
DEADT : 10.0 usec
INTVL : 8.2 usec
TIMES : 1000 times
DUMMY : 1 times
PD : 5.0000 sec
ACQTM : 268.6976 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 3.72 Hz
PW1 : 7.10 usec
OBNUC : 31P
OBFRQ : 202.35 MHz
OBSET : 118939.00 Hz
RGAIN : 24
IRNUC : 1H
IRFRQ : 500.00 MHz
IRSET : 162160.00 Hz
IRRPW : 45.0 usec
IRRNS : 0
SCANS : 16 times
SLVNT : CDCL3
SPINNING : 14 Hz
TEMP : 23.9 C

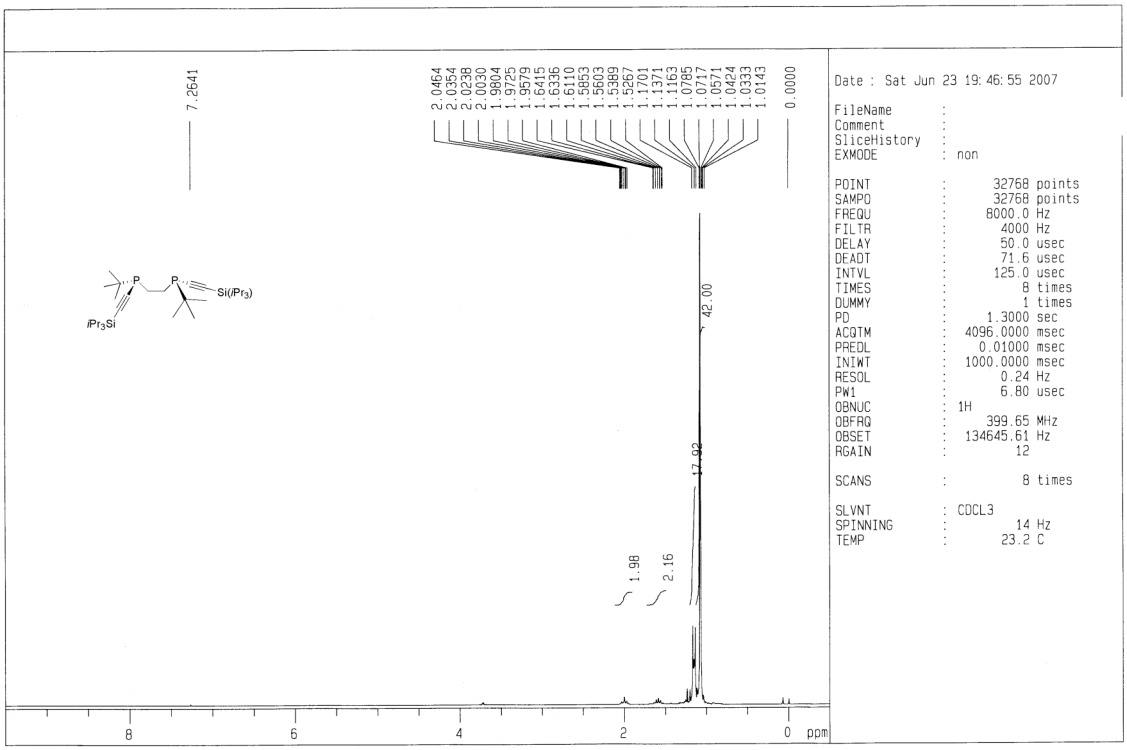

7.2676
1.9012
1.8970
1.8809
1.8672
1.8726
1.8701
1.8543
1.8500
1.8451
1.8372
1.8329
1.5773
1.5651
1.5602
1.5474
1.5413
1.5278
1.5230
1.5144
1.5046
1.4973
1.4853
1.4778
1.3680
1.3332
1.3246
1.3021
1.2880
1.2801
1.2746
1.2716
1.2703
1.2667
1.2618
1.2557
1.2533
1.2423
1.2374
1.2331
1.2234
1.2215
1.2185
1.2130
1.2112
1.1929
1.1880
1.1489
1.1440
1.1331
1.1227
1.1184
1.1074
0.0702
0.0000
18.00
18.11
2.09
2.20
10
5
0
-5
ppm
Date : Thu Jul 12 12:09:16 2007
FileName :
Comment :
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 57.2 usec
INTVL : 100.0 usec
TIMES : 8 times
DUMMY : 1 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 5.55 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
RGAIN : 15
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 13 Hz
TEMP : 25.0 C


115.296
115.280
115.264
77.256
77.000
76.752
75.695
75.639
75.583
75.534
30.992
29.374
29.350
29.326
28.557
27.340
27.284
27.228
20.236
200
150
100
50
0
ppm
Date : Thu Jul 12 12:21:55 2007
FileName :
Comment :
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 33003.3 Hz
FILTR : 16500 Hz
DELAY : 12.1 usec
DEADT : 15.8 usec
INTVL : 30.3 usec
TIMES : 1500 times
DUMMY : 4 times
PD : 1.0333 sec
ACQTM : 992.8704 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 1.01 Hz
PW1 : 4.75 usec
OBNUC : 13C
OBFRQ : 125.65 MHz
OBSET : 127958.00 Hz
RGAIN : 30
IRNUC : 1H
IRFRQ : 500.00 MHz
IRSET : 162160.00 Hz
IRPW : 45.0 usec
IRNS : 0
SCANS : 257 times
SLVNT : CDCL3
SPINNING : 7 Hz
TEMP : 25.7 C



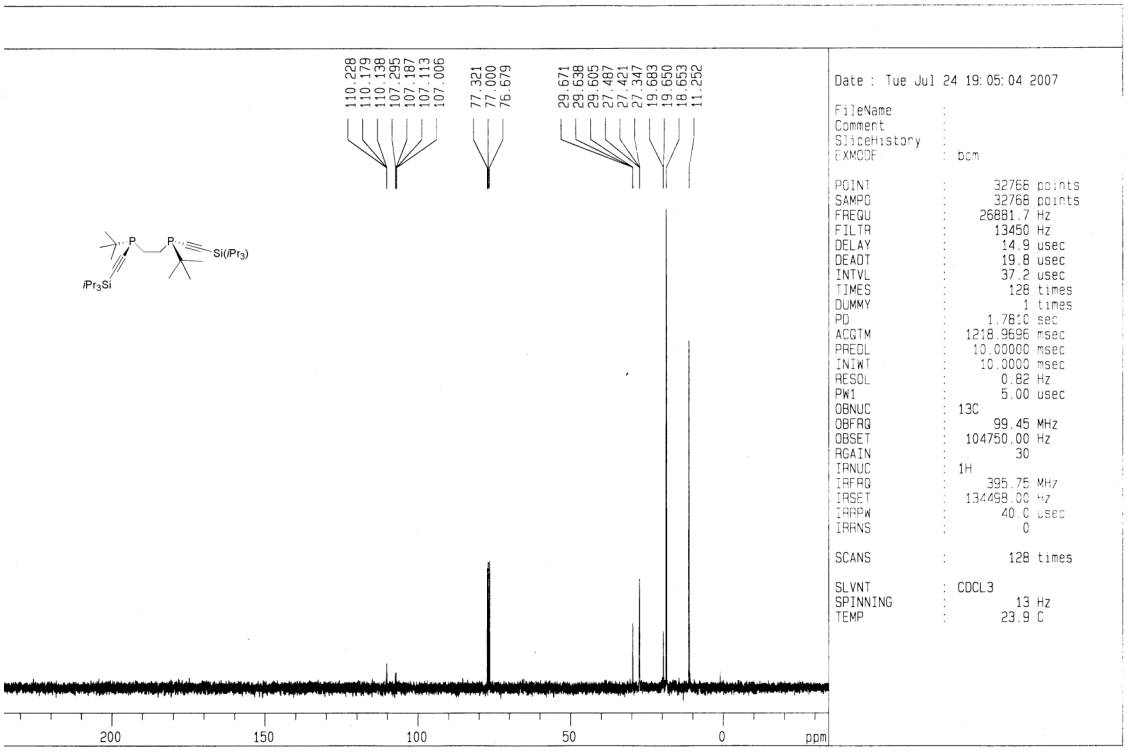
Date : Thu Jul 12 11:35:35 2007

FileName	:	
Comment	:	
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	121951.2 Hz
FILTR	:	61000 Hz
DELAY	:	3.3 usec
DEADT	:	10.0 usec
INTVL	:	8.2 usec
TIMES	:	512 times
DUMMY	:	1 times
PD	:	1.0000 sec
ACQTM	:	268.6976 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	3.72 Hz
PW1	:	7.10 usec
OBNUC	:	31P
OBFRQ	:	202.35 MHz
OBSET	:	118939.00 Hz
RGAIN	:	24
IRNUC	:	1H
IRFRQ	:	500.00 MHz
IRSET	:	162160.00 Hz
IRRPW	:	45.0 usec
IRRNS	:	0
SCANS	:	130 times
SLVNT	:	CDCL3
SPINNING	:	13 Hz
TEMP	:	25.3 C



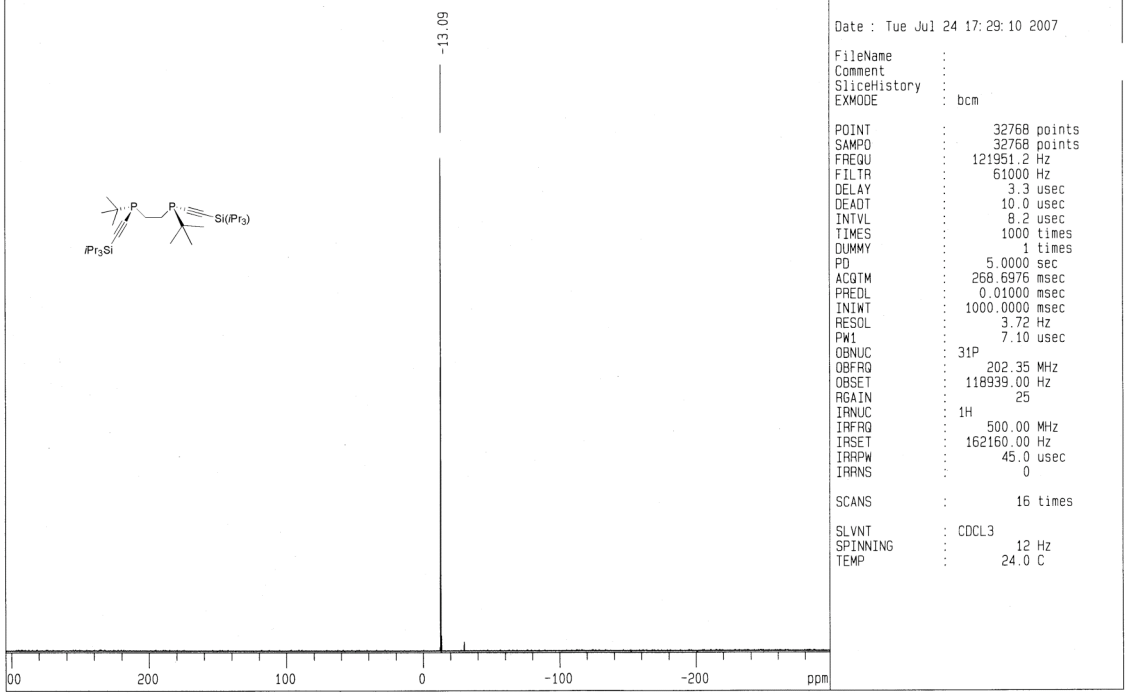
Date : Sat Jun 23 19:46:55 2007

FileName	:	
Comment	:	
SliceHistory	:	
EXMODE	:	non
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	8000.0 Hz
FILTR	:	4000 Hz
DELAY	:	50.0 usec
DEADT	:	71.6 usec
INTVL	:	125.0 usec
TIMES	:	8 times
DUMMY	:	1 times
PD	:	1.3000 sec
ACQTM	:	4096.0000 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	0.24 Hz
PW1	:	6.80 usec
OBNUC	:	1H
OBFRQ	:	399.65 MHz
OBSET	:	134645.61 Hz
RGAIN	:	12
SCANS	:	8 times
SLVNT	:	CDCL3
SPINNING	:	14 Hz
TEMP	:	23.2 C



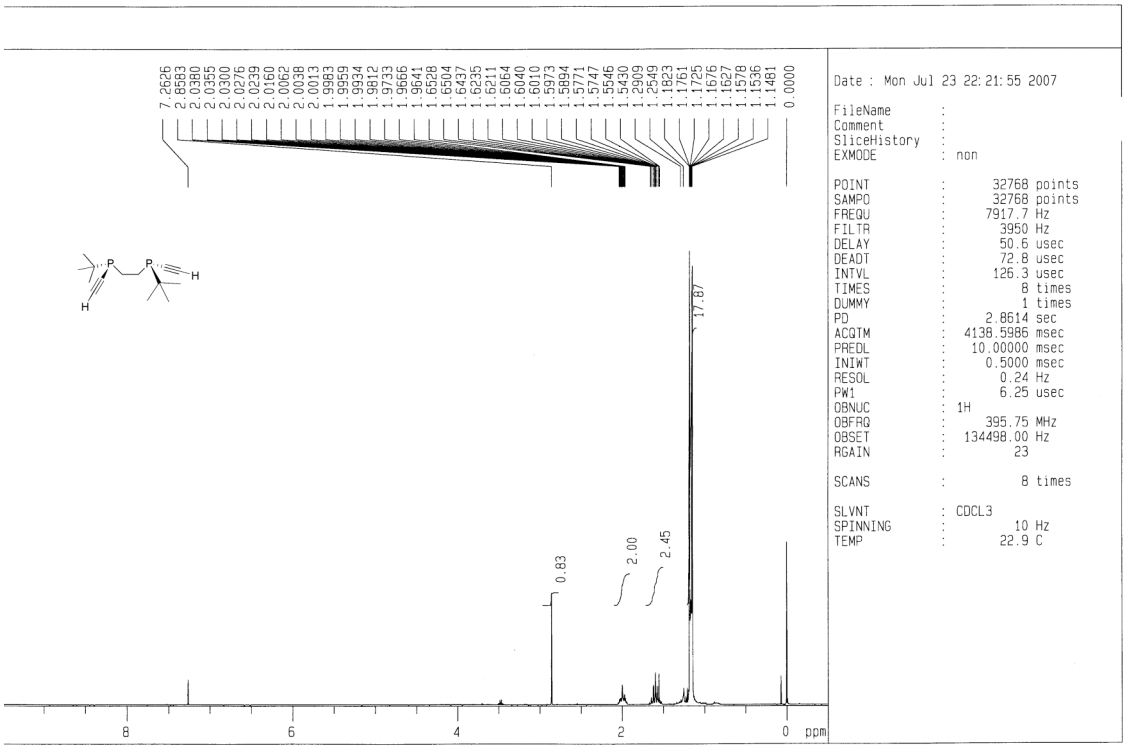
Date : Tue Jul 24 19:05:04 2007

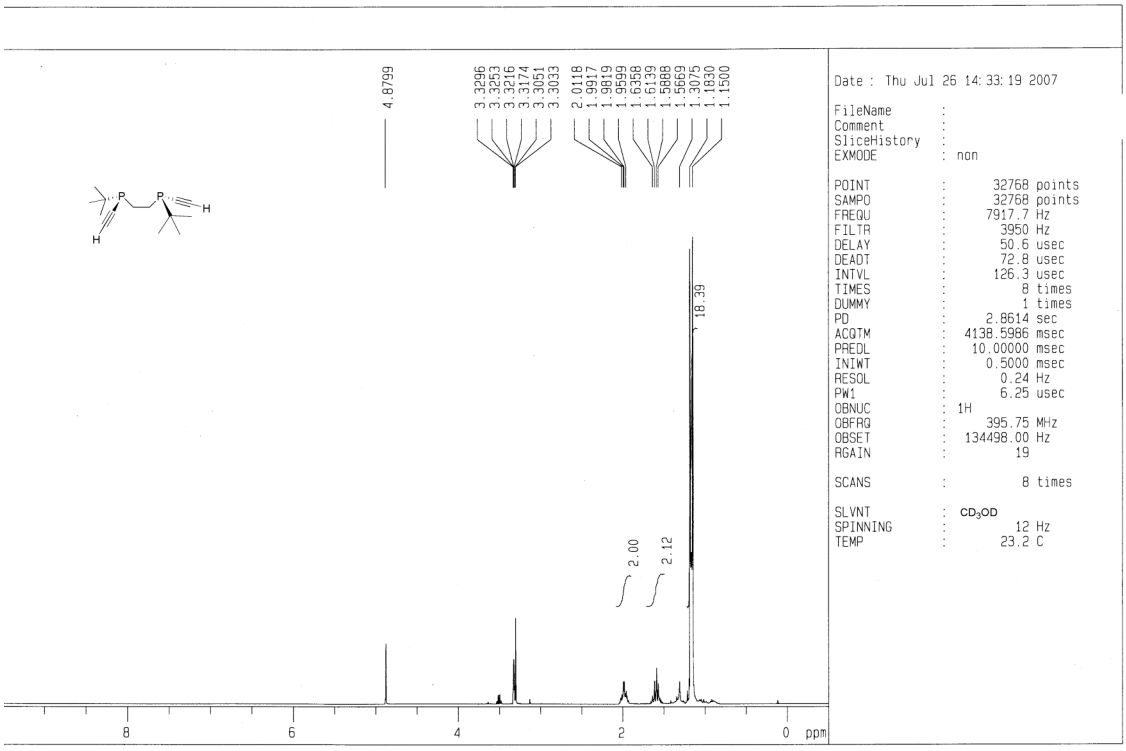
FileName	:	
Comment	:	
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	26881.7 Hz
FILTR	:	13450 Hz
DELAY	:	14.9 usec
DEADT	:	19.8 usec
INTVL	:	37.2 usec
TIMES	:	128 times
DUMMY	:	1 times
PD	:	1.7810 sec
ACQTM	:	1218.9696 msec
PREDL	:	10.00000 msec
INIWT	:	10.0000 msec
RESOL	:	0.82 Hz
PW1	:	5.00 usec
OBNUC	:	13C
OBFRQ	:	99.45 MHz
OBSET	:	104750.00 Hz
RGAIN	:	30
IRNUC	:	1H
IRFRQ	:	395.75 MHz
IRSET	:	134498.00 Hz
IRRPW	:	40.0 usec
IRRNS	:	0
SCANS	:	128 times
SLVNT	:	CDCL3
SPINNING	:	13 Hz
TEMP	:	23.9 C

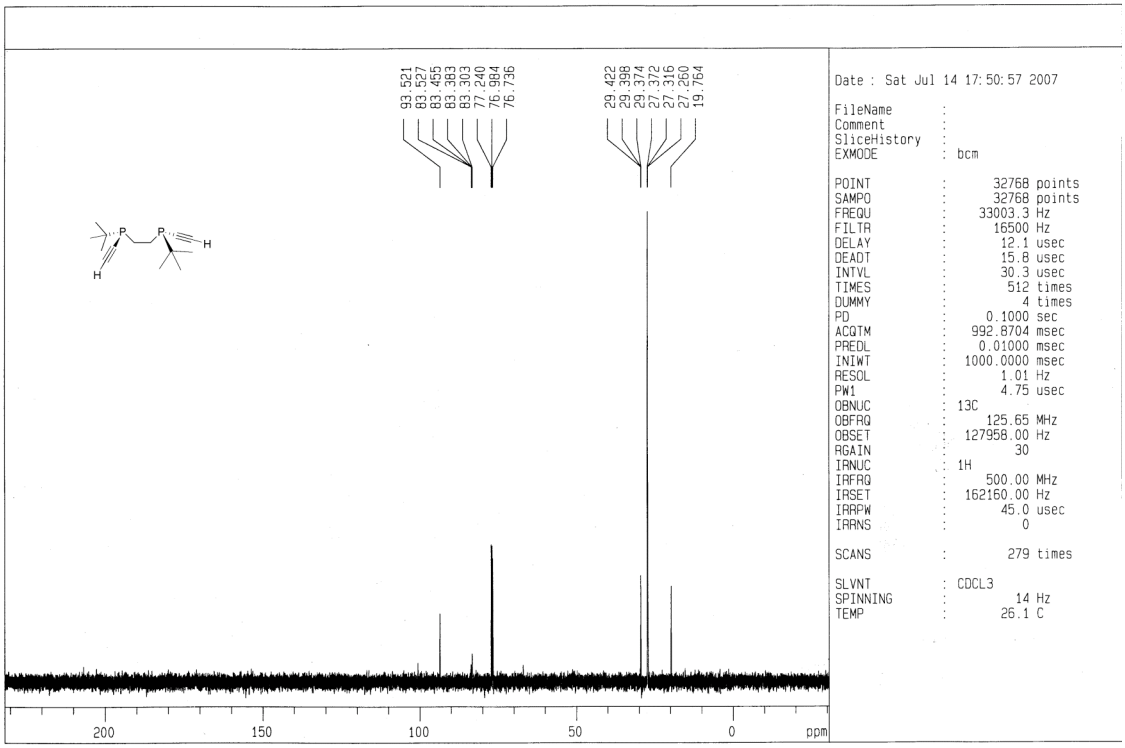


Date : Tue Jul 24 17:29:10 2007

FileName	:	
Comment	:	
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	121951.2 Hz
FILTR	:	61000 Hz
DELAY	:	3.3 usec
DEADT	:	10.0 usec
INTVL	:	8.2 usec
TIMES	:	1000 times
DUMMY	:	1 times
PD	:	5.0000 sec
ACQTM	:	268.6976 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	3.72 Hz
PW1	:	7.10 usec
OBNUC	:	31P
OBFRQ	:	202.35 MHz
OBSET	:	118939.00 Hz
RGAIN	:	25
IRNUC	:	1H
IRFRQ	:	500.00 MHz
IRSET	:	162160.00 Hz
IRRPW	:	45.0 usec
IRRNS	:	0
SCANS	:	16 times
SLVNT	:	CDCL3
SPINNING	:	12 Hz
TEMP	:	24.0 C

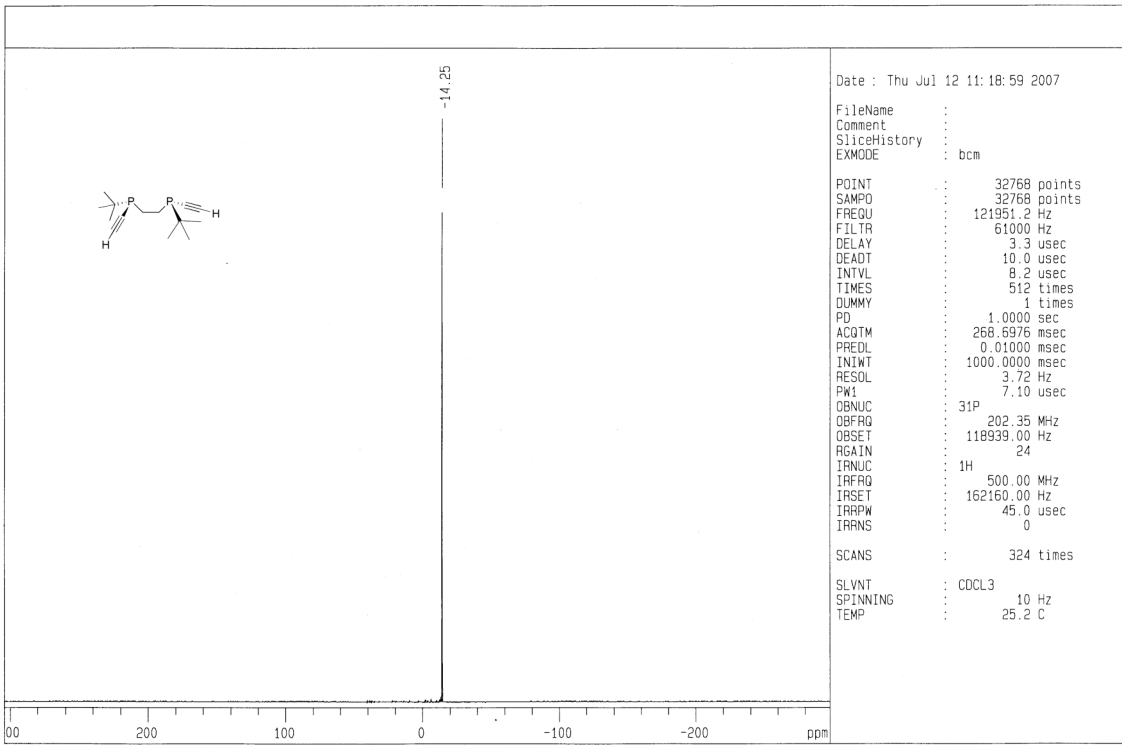

7.2626
2.8563
2.0380
2.0355
2.0300
2.0276
2.0239
2.0160
2.0062
2.0038
2.0013
1.9983
1.9959
1.9934
1.9812
1.9733
1.9666
1.9641
1.6528
1.6504
1.6437
1.6235
1.6211
1.6064
1.6040
1.6010
1.5973
1.5894
1.5771
1.5747
1.5546
1.5430
1.2909
1.2549
1.1823
1.1761
1.1725
1.1676
1.1627
1.1578
1.1536
1.1481
0.0000
17.87
2.00
2.45
0.83
8
6
4
2
0
ppm
Date : Mon Jul 23 22:21:55 2007
FileName :
Comment :
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7917.7 Hz
FILTR : 3950 Hz
DELAY : 50.6 usec
DEADT : 72.8 usec
INTVL : 126.3 usec
TIMES : 8 times
DUMMY : 1 times
PD : 2.8614 sec
ACQTM : 4138.5986 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 0.24 Hz
PW1 : 6.25 usec
OBNUC : 1H
OBFRQ : 395.75 MHz
OBSET : 134498.00 Hz
RGAIN : 23
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 10 Hz
TEMP : 22.9 C


4.8799
3.3296
3.3253
3.3216
3.3174
3.3051
3.3033
2.0118
1.9917
1.9819
1.9599
1.6358
1.6139
1.5888
1.5669
1.3075
1.1830
1.1500
18.39
2.00
2.12
8
6
4
2
0
ppm
Date : Thu Jul 26 14:33:19 2007
FileName :
Comment :
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 7917.7 Hz
FILTR : 3950 Hz
DELAY : 50.6 usec
DEADT : 72.8 usec
INTVL : 126.3 usec
TIMES : 8 times
DUMMY : 1 times
PD : 2.8614 sec
ACQTM : 4138.5986 msec
PREDL : 10.00000 msec
INIWT : 0.5000 msec
RESOL : 0.24 Hz
PW1 : 6.25 usec
OBNUC : 1H
OBFRQ : 395.75 MHz
OBSET : 134498.00 Hz
RGAIN : 19
SCANS : 8 times
SLVNT : CD3OD
SPINNING : 12 Hz
TEMP : 23.2 C



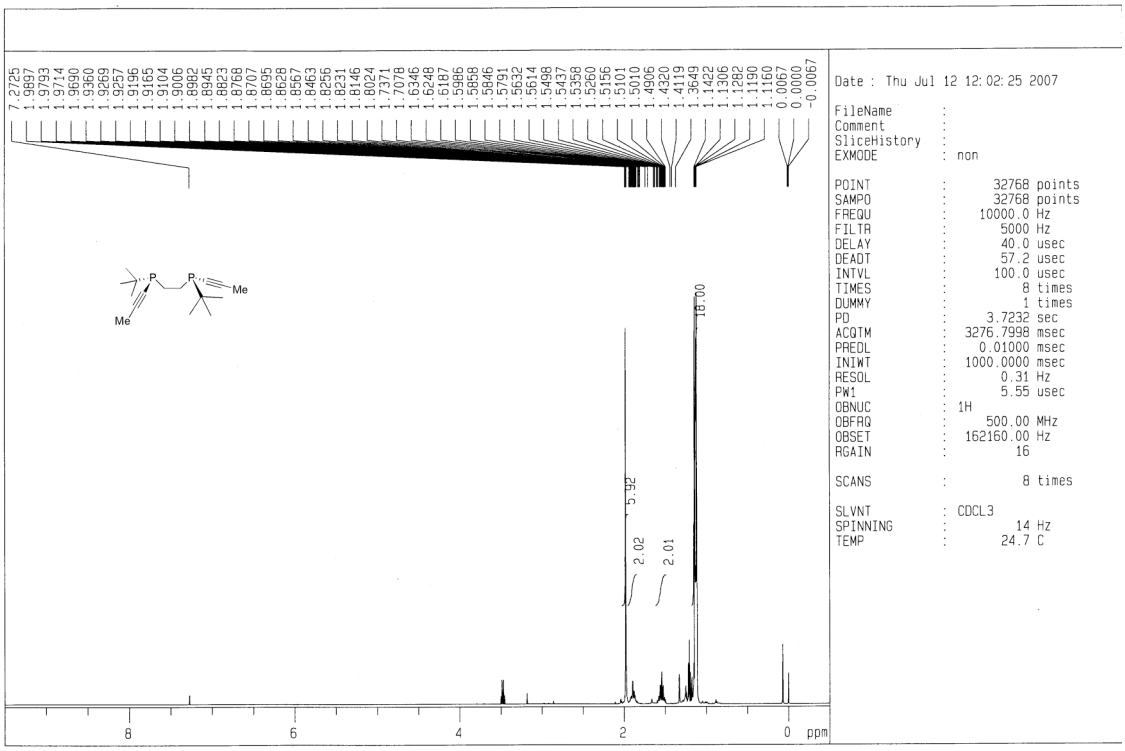
Date : Sat Jul 14 17:50:57 2007

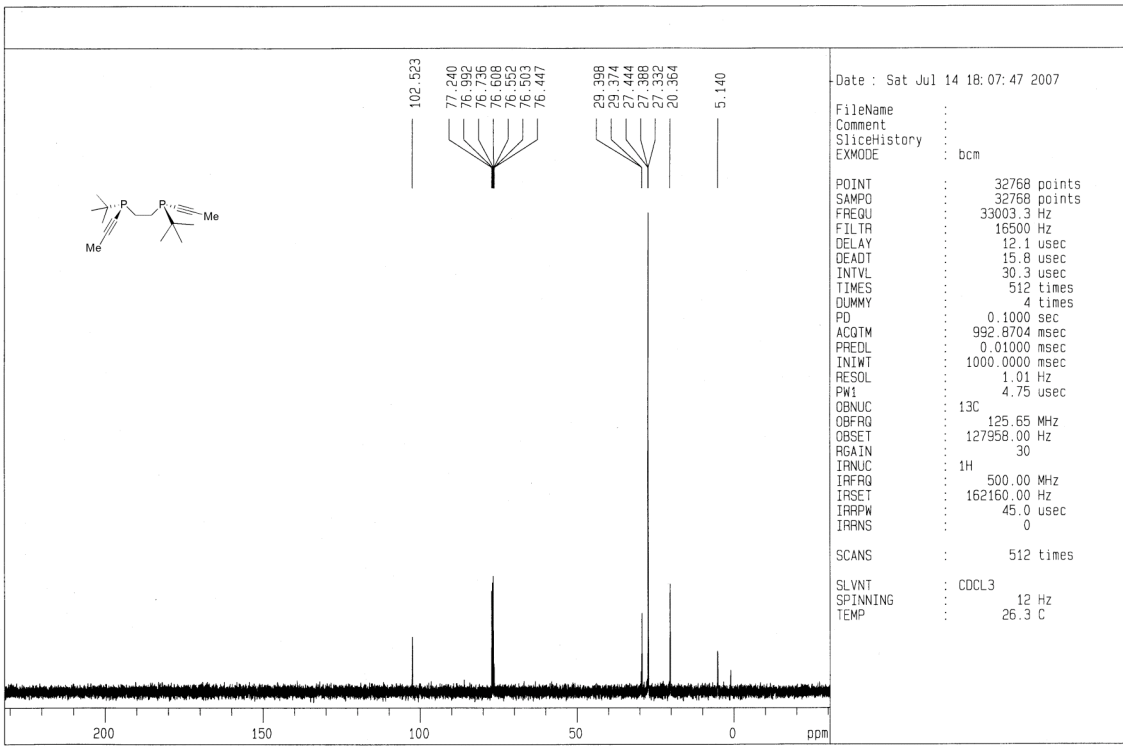
FileName	:	
Comment	:	
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	33003.3 Hz
FILTR	:	16500 Hz
DELAY	:	12.1 usec
DEADT	:	15.8 usec
INTVL	:	30.3 usec
TIMES	:	512 times
DUMMY	:	4 times
PD	:	0.1000 sec
ACQTM	:	992.8704 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	1.01 Hz
PW1	:	4.75 usec
OBNUC	:	13C
OBFRQ	:	125.65 MHz
OBSET	:	127958.00 Hz
RGAIN	:	30
IRNUC	:	1H
IRFRQ	:	500.00 MHz
IRSET	:	162160.00 Hz
IRRPW	:	45.0 usec
IRRNS	:	0
SCANS	:	279 times
SLVNT	:	CDCL3
SPINNING	:	14 Hz
TEMP	:	26.1 C

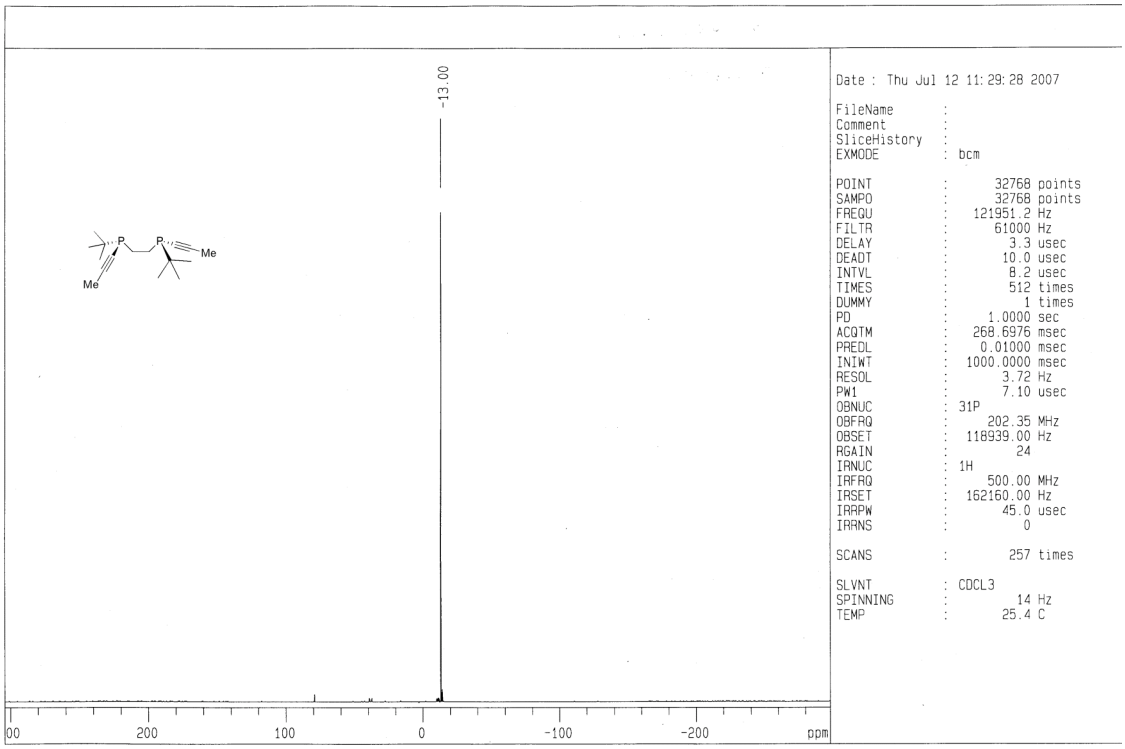


Date : Thu Jul 12 11:18:59 2007

FileName	:	
Comment	:	
SliceHistory	:	
EXMODE	:	bcm
POINT	:	32768 points
SAMPO	:	32768 points
FREQU	:	121951.2 Hz
FILTR	:	61000 Hz
DELAY	:	3.3 usec
DEADT	:	10.0 usec
INTVL	:	8.2 usec
TIMES	:	512 times
DUMMY	:	1 times
PD	:	1.0000 sec
ACQTM	:	268.6976 msec
PREDL	:	0.01000 msec
INIWT	:	1000.0000 msec
RESOL	:	3.72 Hz
PW1	:	7.10 usec
OBNUC	:	31P
OBFRQ	:	202.35 MHz
OBSET	:	118939.00 Hz
RGAIN	:	24
IRNUC	:	1H
IRFRQ	:	500.00 MHz
IRSET	:	162160.00 Hz
IRRPW	:	45.0 usec
IRRNS	:	0
SCANS	:	324 times
SLVNT	:	CDCL3
SPINNING	:	10 Hz
TEMP	:	25.2 C


7.2725
1.9857
1.9793
1.9714
1.9690
1.9360
1.9269
1.9257
1.9196
1.9165
1.9104
1.9006
1.8982
1.8945
1.8823
1.8768
1.8707
1.8695
1.8628
1.8567
1.8463
1.8256
1.8231
1.8146
1.8024
1.7371
1.7078
1.6346
1.6248
1.6187
1.5986
1.5858
1.5846
1.5791
1.5632
1.5614
1.5498
1.5437
1.5358
1.5260
1.5156
1.5101
1.5010
1.4906
1.4320
1.4119
1.3649
1.1422
1.1306
1.1282
1.1190
1.1160
0.0067
0.0000
-0.0067
Me
Me
18.00
5.92
2.02
2.01
8
6
4
2
0
ppm
Date : Thu Jul 12 12:02:25 2007
FileName :
Comment :
SliceHistory :
EXMODE : non
POINT : 32768 points
SAMPO : 32768 points
FREQU : 10000.0 Hz
FILTR : 5000 Hz
DELAY : 40.0 usec
DEADT : 57.2 usec
INTVL : 100.0 usec
TIMES : 8 times
DUMMY : 1 times
PD : 3.7232 sec
ACQTM : 3276.7998 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 0.31 Hz
PW1 : 5.55 usec
OBNUC : 1H
OBFRQ : 500.00 MHz
OBSET : 162160.00 Hz
RGAIN : 16
SCANS : 8 times
SLVNT : CDCL3
SPINNING : 14 Hz
TEMP : 24.7 C


102.523
77.240
76.992
76.736
76.608
76.552
76.503
76.447
29.398
29.374
27.444
27.388
27.332
20.364
5.140
Me
Me
200
150
100
50
0
ppm
Date : Sat Jul 14 18:07:47 2007
FileName :
Comment :
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 33003.3 Hz
FILTR : 16500 Hz
DELAY : 12.1 usec
DEADT : 15.8 usec
INTVL : 30.3 usec
TIMES : 512 times
DUMMY : 4 times
PD : 0.1000 sec
ACQTM : 992.8704 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 1.01 Hz
PW1 : 4.75 usec
OBNUC : 13C
OBFRQ : 125.65 MHz
OBSET : 127958.00 Hz
RGAIN : 30
IRNUC : 1H
IRFRQ : 500.00 MHz
IRSET : 162160.00 Hz
IRRPW : 45.0 usec
IRRNS : 0
SCANS : 512 times
SLVNT : CDCL3
SPINNING : 12 Hz
TEMP : 26.3 C


-13.00
Me
Me
00
200
100
0
-100
-200
ppm
Date : Thu Jul 12 11:29:28 2007
FileName :
Comment :
SliceHistory :
EXMODE : bcm
POINT : 32768 points
SAMPO : 32768 points
FREQU : 121951.2 Hz
FILTR : 61000 Hz
DELAY : 3.3 usec
DEADT : 10.0 usec
INTVL : 8.2 usec
TIMES : 512 times
DUMMY : 1 times
PD : 1.0000 sec
ACQTM : 268.6976 msec
PREDL : 0.01000 msec
INIWT : 1000.0000 msec
RESOL : 3.72 Hz
PW1 : 7.10 usec
OBNUC : 31P
OBFRQ : 202.35 MHz
OBSET : 118939.00 Hz
RGAIN : 24
IRNUC : 1H
IRFRQ : 500.00 MHz
IRSET : 162160.00 Hz
IRPW : 45.0 usec
IRNS : 0
SCANS : 257 times
SLVNT : CDCL3
SPINNING : 14 Hz
TEMP : 25.4 C