Electronic Supplementary Information

Tri-insertion with Dearomatization of Terminal Arylalkynes at Carborane Based Frustrated Lewis Pair Template

*Jian Zhang and Zuowei Xie**

Department of Chemistry and State Key Laboratory of Synthetic Chemistry, The Chinese University of Hong Kong, Shatin, New Territories, Hong Kong, China

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General Procedures. Unless otherwise noted, all experiments were performed under an atmosphere of dry dinitrogen or argon with the rigid exclusion of air and moisture using standard Schlenk or cannula techniques or in a glovebox. CH_2Cl_2 were refluxed over CaH_2 for several days and distilled immediately prior to use. Other organic solvents were refluxed over sodium benzophenone ketyl for several days and freshly distilled prior to use. Compounds 1-PPh_2-1,2-$C_2B_{10}H_{11}$ (**1**),[1] and Ph_2BCl[2] were prepared according to literature methods. All other chemicals were purchased from either Aldrich or Acros Chemical Co. and used as received unless otherwise noted. Infrared spectra were obtained from KBr pellets on a Perkin-Elmer 1600 Fourier transform spectrometer. The ^1H and ^{13}C NMR spectra were recorded on a Bruker DPX 400 spectrometer at 400 and 100 MHz, respectively. The ^{11}B and ^{31}P NMR spectra were recorded on a Bruker DPX 400 spectrometer or a Bruker DPX 400Q spectrometer at 128 MHz and 162 MHz, respectively. All chemical shifts were reported in δ units with references to residual protons or carbons of the deuterated solvents for proton or carbon chemical shifts, to external $BF_3 \cdot OEt_2$ (0.0 ppm) for boron chemical shifts, and to external 85% H_3PO_4 (0.0 ppm) for phosphorus chemical shifts. Mass spectra were recorded on a Thermo Finnigan MAT 95 XL spectrometer. Elemental analyses were performed by MEDAC Ltd., Brunel University, Middlesex, U.K. or Shanghai Institute of Organic Chemistry, the Chinese Academy of Sciences, Shanghai, China.

Preparation of 1-PPh_2-2-BPh_2-1,2-$C_2B_{10}H_{10}$ (2). To a toluene (40 mL) solution of 1-PPh_2-1,2-$C_2B_{10}H_{11}$ (**1**; 1.642 g, 5.0 mmol) was slowly added *n*-BuLi (3.3 mL, 1.6 M, 5.3 mmol in hexane) at room temperature, and the suspension was stirred overnight. To the resultant suspension was slowly added Ph_2BCl (1.062 g, 5.3 mmol), and the suspension was stirred at room temperature overnight. After removal of the precipitate, the solvent was removed under vacuum. The resultant pale yellow residue was thoroughly washed with cold Et_2O to give **2** as a white powder (1.31 g, 53%). Single crystals suitable for X-ray analyses were grown from *n*-hexane layering over a saturated toluene solution. ^1H NMR (400 MHz, CD_2Cl_2): δ 7.56 (m, 2H, C_6H_5), 7.42-7.39 (m, 12H, C_6H_5), 7.21-7.20 (m, 6H, C_6H_5). ^{13}C NMR (100 MHz, CD_2Cl_2): δ 146.6 (br, BC_6H_5), 134.1 (d, J_{PC} = 10.9 Hz, PC_6H_5), 133.0 (d, $^4J_{PC}$ = 2.4 Hz, PC_6H_5), 132.7 (d, J_{PC} = 11.0 Hz, PC_6H_5), 129.5 (d, J_{PC} = 10.2 Hz, PC_6H_5), 128.0 (d, $^1J_{PC}$ = 33.0 Hz, PC_6H_5), 127.9 (BC_6H_5), 127.0 (d, $^5J_{PC}$ = 1.5 Hz, BC_6H_5), 85.8 (br, cage *C*-B), 73.3 (d, $^1J_{PC}$ = 24.1 Hz, cage *C*-P). ^{11}B NMR (128 MHz, CD_2Cl_2): δ 15.5 (s, 1B, *B*Ph_2), 0.6 (d, J_{BH} = 145 Hz, 1B), –2.2 (d, J_{BH} = 149 Hz, 1B), –6.3 (d, J_{BH} = 142 Hz, 2B), –9.6 (not resolved, 6B). ^{31}P NMR (162 MHz, CD_2Cl_2): δ 27.9. IR (KBr, cm^{-1}): ν_{max} 2568 (B–H). Anal. Calcd for $C_{26}H_{30}B_{11}P$ (**2**): C, 63.42; H, 6.14. Found: C, 63.57; H, 6.19.

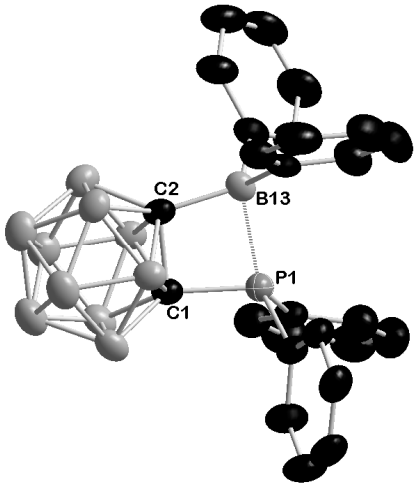

Figure S1. Molecular Structure of **2**

Preparation of 1,2-[$Ph_2PC(Me_2NC_6H_4)$=C(H)BPh_2]-1,2-$C_2B_{10}H_{10}$ (3-Me). To a toluene (2 mL) solution of **2** (99 mg, 0.20 mmol) was added *p*-$Me_2NC_6H_4C{\equiv}CH$ (31 mg, 0.21 mmol), and the mixture was stirred at room temperature for 1 h. After removal of the solvent, the pale yellow residue was thoroughly washed with *n*-hexane to afford **3-Me** as a yellow powder (120 mg, 94%). Single crystals suitable for X-ray analyses were grown from *n*-hexane layering over a saturated THF solution. ^{1}H NMR (400 MHz, CD_2Cl_2): δ 9.29 (d, $^3J_{PH}$ = 53 Hz, 1H, BC*H*=C), 7.84 (dd, J_1 = 7.7 Hz, J_2 = 12.5 Hz, 4H, PC_6H_5), 7.63 (t, J = 7.4 Hz, 2H, PC_6H_5), 7.44-7.39 (m, 8H, PC_6H_5 + BC_6H_5), 7.13 (t, J = 7.4 Hz, 4H, BC_6H_5), 7.04 (t, J = 7.2 Hz, 2H, BC_6H_5), 6.91 (d, J = 8.0 Hz, 2H, C_6H_4), 6.42 (d, J = 8.7 Hz, 2H, C_6H_4), 2.83 (s, 6H, CH_3). ^{13}C NMR (100 MHz, CD_2Cl_2): δ 191.4 (br, B*C*H=C), 155.9 (br, BC_6H_5), 150.1 (C_6H_4), 135.7 (d, $^2J_{PC}$ = 9.7 Hz, PC_6H_5), 135.1 (d, $^4J_{PC}$ = 2.9 Hz, PC_6H_5), 134.4 (BC_6H_5), 129.6 (d, $^3J_{PC}$ = 12.9 Hz, PC_6H_5), 128.7 (d, $^3J_{PC}$ = 5.4 Hz, C_6H_4), 127.0 (BC_6H_5), 126.6 (d, $^2J_{PC}$ = 17.9 Hz, C_6H_4), 124.7 (BC_6H_5), 118.1 (d, $^1J_{PC}$ = 87 Hz, PC_6H_5), 117.8 (d, $^1J_{PC}$ = 78 Hz, BCH=*C*), 112.1 (C_6H_4), 90.9 (br, cage *C*B), 67.8 (d, $^1J_{PC}$ = 61 Hz, cage *C*P), 40.3 (NCH_3). ^{11}B NMR (128 MHz, CD_2Cl_2): δ 2.1 (d, J_{BH} = 115 Hz, 1B), –2.3 (d, J_{BH} = 127 Hz, 1B), –7.7 (d, J_{BH} = 142 Hz, 5B), –10.2 (not resolved, 3B), –11.4 (s, 1B). ^{31}P NMR (162 MHz, CD_2Cl_2): δ 11.5 (d, $^3J_{PH}$ = 53 Hz). IR (KBr, cm^{-1}): ν_{max} 2572 (B–H). HRMS (ESI): *m*/*z* calcd for $C_{36}H_{41}{}^{11}B_9{}^{10}B_2NNaP$ $[M+Na]^+$: 660.3984. Found: 660.3972.

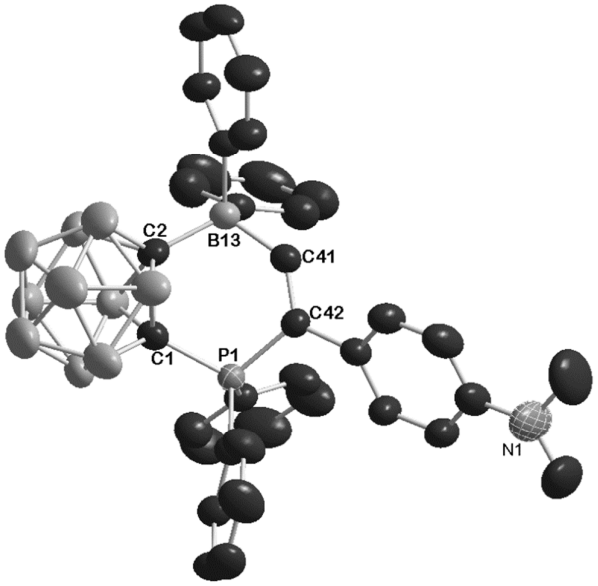

Figure S2. Molecular Structure of **3-Me**

Preparation of 1,2-[$Ph_2PC(Et_2NC_6H_4)$=C(H)BPh_2]-1,2-$C_2B_{10}H_{10}$ (3-Et). To a toluene (2 mL) solution of **2** (99 mg, 0.20 mmol) was added *p*-$Et_2NC_6H_4C{\equiv}CH$ (36 mg, 0.21 mmol), and the mixture was stirred at room temperature for 1 h. After removal of the solvent, the pale yellow residue was thoroughly washed with *n*-hexane to afford **3-Et** as a yellow powder (120 mg, 90%). ^{1}H NMR (400 MHz, CD_2Cl_2): δ 9.27 (d, $^3J_{PH}$ = 54 Hz, 1H, BC*H*=C), 7.85 (dd, J_1 = 7.5 Hz, J_2 = 12.5 Hz, 4H, PC_6H_5), 7.63 (dd, J_1 = 6.8 Hz, J_2 = 8.2 Hz, 2H, PC_6H_5), 7.44-7.40 (m, 8H, PC_6H_5 + BC_6H_5), 7.13 (t, J = 7.3 Hz, 4H, BC_6H_5), 7.04 (t, J = 7.2 Hz, 2H, BC_6H_5),6.87 (dd, J_1 = 1.0 Hz, J_2 = 8.8 Hz, 2H, C_6H_4), 6.38 (d, J = 8.7 Hz, 2H, C_6H_4), 3.24 (q, J = 7.0 Hz, 4H, CH_2), 1.05 (t, J = 7.0 Hz, 6H, CH_3). ^{13}C NMR (100 MHz, CD_2Cl_2): δ 190.7 (br, B*C*H=C), 155.6 (br, BC_6H_5), 147.5 (C_6H_4), 135.7 (d, $^2J_{PC}$ = 9.8 Hz, PC_6H_5), 135.1 (d, $^4J_{PC}$ = 2.9 Hz, PC_6H_5), 134.4 (BC_6H_5), 129.6 (d, $^3J_{PC}$ = 12.8 Hz, PC_6H_5), 129.0 (d, $^3J_{PC}$ = 5.4 Hz, C_6H_4), 127.1 (BC_6H_5), 125.6 (d, $^2J_{PC}$ = 18.1 Hz, C_6H_4), 124.7 (BC_6H_5), 118.3 (d, $^1J_{PC}$ = 87 Hz, PC_6H_5), 117.9 (d, $^1J_{PC}$ = 77 Hz, BCH=*C*), 111.6 (C_6H_4), 91.1 (br, cage *C*B), 67.9 (d, $^1J_{PC}$ = 60 Hz, cage *C*P), 44.6 (N*C*H$_2$), 12.6 (*C*H$_3$). ^{11}B NMR (128 MHz, CD_2Cl_2): δ 2.1 (d, J_{BH} = 125 Hz, 1B), –2.2 (d, J_{BH} = 125 Hz, 1B), –7.7 (d, J_{BH} = 126 Hz, 5B), –10.0 (not resolved, 3B), –11.5 (s, 1B). ^{31}P NMR (162 MHz, CD_2Cl_2): δ 11.4 (d, $^3J_{PH}$ = 54 Hz). IR (KBr, cm^{-1}): ν_{max} 2588 (B–H). HRMS (ESI): *m*/*z* calcd for $C_{38}H_{46}{}^{11}B_9{}^{10}B_2NP$ $[M+H]^+$: 666.4478. Found: 666.4485.

Preparation of 1,2-[$Ph_2PC(Ph_2NC_6H_4)$=C(H)BPh_2]-1,2-$C_2B_{10}H_{10}$ (3-Ph). To a toluene (2 mL) solution of **2** (198 mg, 0.40 mmol) was added *p*-$Ph_2NC_6H_4C{\equiv}CH$ (113 mg, 0.42 mmol), and the mixture was stirred at room temperature for 2 d. After removal of the solvent, the pale yellow residue was washed with Et_2O and dried under vacuum to afford **3-Ph** as a yellow powder (260 mg, 85%). Single crystals suitable for X-ray analyses were grown from slow evaporation of a saturated Et_2O solution. ^{1}H NMR (400 MHz, CD_2Cl_2): δ 9.41 (d, $^3J_{PH}$ = 53 Hz, 1H, BC*H*=C), 7.81 (dd, J_1 = 7.9 Hz, J_2 = 12.5 Hz, 4H, PC_6H_5), 7.67 (t, J = 7.2 Hz, 2H, PC_6H_5), 7.45-7.40 (m, 8H, PC_6H_5 + BC_6H_5), 7.22 (t, J = 7.8 Hz, 4H, NC_6H_5), 7.13 (t, J = 7.3 Hz, 4H, BC_6H_5), 7.05 (t, J = 6.5 Hz, 2H, BC_6H_5), 7.01 (t, J = 7.2 Hz, 2H, NC_6H_5), 6.91 (d, J = 8.0 Hz, 4H, NC_6H_5), 6.88 (d, J = 8.8 Hz, 2H, C_6H_4), 6.74 (d, J = 8.4 Hz, 2H, C_6H_4). ^{13}C NMR (100 MHz, CD_2Cl_2): δ 193.6 (br, B*C*H=C), 155.7 (br, BC_6H_5), 147.73 (C_6H_4), 147.56 (NC_6H_5), 135.8 (d, $^2J_{PC}$ = 9.7 Hz, PC_6H_5), 135.2 (d, $^4J_{PC}$ = 2.9 Hz, PC_6H_5), 134.4 (BC_6H_5), 132.9 (d, $^2J_{PC}$ = 17.8 Hz, C_6H_4), 129.64 (NC_6H_5), 129.63 (d, $^3J_{PC}$ = 13.0 Hz, PC_6H_5), 129.0 (d, $^3J_{PC}$ = 5.3 Hz, C_6H_4), 127.1 (BC_6H_5), 124.80 (BC_6H_5), 124.74 (NC_6H_5), 123.7 (NC_6H_5), 123.4 (C_6H_4), 118.3 (d, $^1J_{PC}$ = 79 Hz, BCH=*C*), 117.8 (d, $^1J_{PC}$ = 88 Hz, PC_6H_5), 90.7 (br, cage *C*B), 67.3 (d, $^1J_{PC}$ = 61 Hz, cage *C*P). ^{11}B NMR (128 MHz, CD_2Cl_2): δ 2.1 (not resolved, 1B), –2.1 (not resolved, 1B), –7.7 (not resolved, 5B), –9.9 (not resolved, 2B), –11.3 (s+d, not resolved, 2B). ^{31}P NMR (162 MHz, CD_2Cl_2): δ 12.2 (d, $^3J_{PH}$ = 53 Hz). IR (KBr, cm^{-1}): ν_{max} 2566 (B–H). HRMS (ESI): *m*/*z* calcd for $C_{46}H_{45}{}^{11}B_9{}^{10}B_2NNaP$ $[M+Na]^+$: 784.4301. Found: 784.4294.

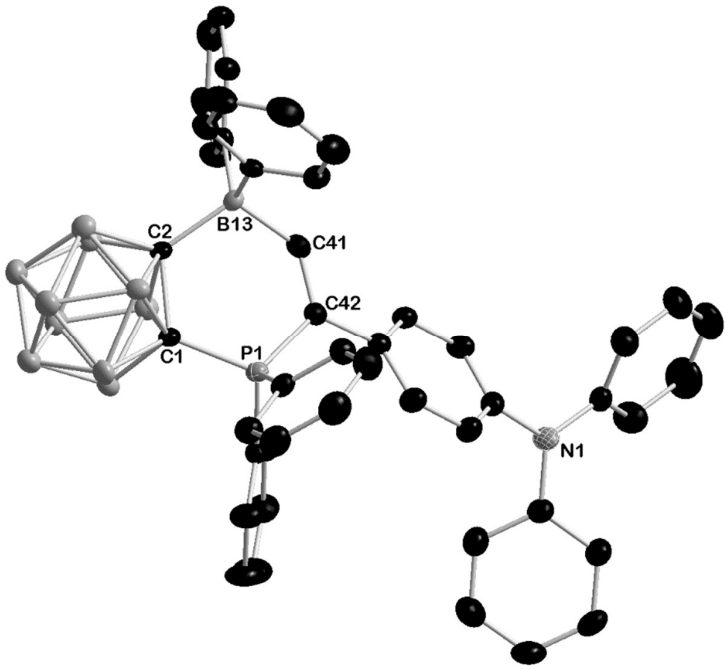

Figure S3. Molecular Structure of **3-Ph**

Preparation of 4-Me$_3$. To a toluene (1 mL) suspension of **3-Me** (64 mg, 0.10 mmol) was added *p*-$Me_2NC_6H_4C{\equiv}CH$ (58 mg, 0.40 mmol), and the suspension was heated at 70 °C with stirring in a sealed flask for 14 d. After removal of the solvent, the red residue was thoroughly washed with *n*-hexane to afford **4-Me$_3$** as a red powder (56 mg, 60%). Single crystals suitable for X-ray analysis were grown by slow evaporation of a saturated THF solution. ^{1}H NMR (400 MHz, CD_2Cl_2): δ 7.75-7.69 (m, 4H, $PC_6\mathit{H}_5$), 7.48-7.40 (m, 4H + 1H, $PC_6\mathit{H}_5$ + $BCC_6\mathit{H}_5$), 7.38 (d, J = 8.9 Hz, 2H, $BC(Ph)CH{=}CC_6\mathit{H}_4$), 7.33 (t, J = 7.3 Hz, 2H, $PC_6\mathit{H}_5$), 7.18-7.09 (m, 3H + 1H, $BCC_6\mathit{H}_5$ + $BC(Ph)C\mathit{H}{=}CCH$), 7.07-7.03 (m, 2H + 1H, $BCH{=}CC_6\mathit{H}_4$ + $BCC_6\mathit{H}_5$), 6.80-6.75 (m, 5H, $BC(Ph)CH{=}CCHC_6\mathit{H}_5$), 6.64 (d, J = 8.8 Hz, 2H, $BC(Ph)CH{=}CC_6\mathit{H}_4$), 6.30 (d, J = 8.9 Hz, 2H, $BCH{=}CC_6\mathit{H}_4$), 5.97 (d, J = 2.9 Hz, 1H, $BC\mathit{H}{=}C$), 5.71 (d, J = 9.8 Hz, 1H, $BC(Ph)CC\mathit{H}{=}CH$), 5.58, (d, J = 9.6 Hz, 1H, $BC(Ph)CCH{=}C\mathit{H}$), 4.19 (s, 2H, $BC(Ph)CC\mathit{H}C\mathit{H}{=}C$), 4.07 (s, 1H, $BC(Ph)CH{=}CC\mathit{H}$), 2.98 (s, 6H, $BCH{=}CC_6H_4NC\mathit{H}_3$), 2.90 (s, 6H, $BC(Ph)CH{=}CC_6H_4NC\mathit{H}_3$), 2.20 (s, 6H, $BC(Ph)CCH{=}CHCNC\mathit{H}_3$). ^{13}C NMR (100 MHz, CD_2Cl_2): δ 194.2 ($BCH{=}\mathit{C}$), 151.8 ($BCH{=}C\mathit{C}_6H_4$), 150.1 ($BC(Ph)CH{=}C\mathit{C}_6H_4$), 146.5 ($BC(Ph)CCHCH{=}\mathit{C}$), 144.7 ($BC\mathit{C}_6H_5$), 140.6 ($BC(Ph)CH{=}CCH\mathit{C}_6H_5$), 137.6 ($BC(Ph)C\mathit{C}H{=}CH$), 135.71 (d, $^2J_{PC}$ = 26.5 Hz, $P\mathit{C}_6H_5$), 135.67 (br, $B\mathit{C}H{=}C$), 135.43 (d, $^2J_{PC}$ = 25.6 Hz, $P\mathit{C}_6H_5$), 134.98 (d, J_{PC} = 5.6 Hz, $BC\mathit{C}_6H_5$), 134.68 (d, $^1J_{PC}$ = 15.6 Hz, $P\mathit{C}_6H_5$), 134.55 (d, $^1J_{PC}$ = 15.1 Hz, $P\mathit{C}_6H_5$), 134.1 ($BC(Ph)CH{=}\mathit{C}CH$), 131.3 ($BCH{=}C\mathit{C}_6H_4$), 131.00 ($BC\mathit{C}_6H_5$), 130.98 (d, $^4J_{PC}$ = 7.8 Hz, $P\mathit{C}_6H_5$), 129.9 ($BC(Ph)CH{=}CCH\mathit{C}_6H_5$), 129.5 ($BC(Ph)CH{=}C\mathit{C}_6H_4$), 129.11 (d, $^3J_{PC}$ = 9.7 Hz, $P\mathit{C}_6H_5$), 129.02 (d, $^3J_{PC}$ = 8.8 Hz, $P\mathit{C}_6H_5$), 128.4 ($BC(Ph)\mathit{C}H{=}CH$), 127.68 ($BC\mathit{C}_6H_5$), 127.4 ($BC(Ph)CH{=}CCH\mathit{C}_6H_5$), 126.3 ($BC(Ph)CH{=}C\mathit{C}_6H_4$), 126.01 ($BC(Ph)CH{=}CCH\mathit{C}_6H_5$), 125.85, 125.3 ($BC\mathit{C}_6H_5$), 124.8 ($BCH{=}C\mathit{C}_6H_4$), 122.9 ($BC(Ph)CCH{=}C\mathit{H}$), 112.7 ($BC(Ph)CH{=}C\mathit{C}_6H_4$), 110.9 ($BCH{=}C\mathit{C}_6H_4$), 104.5 ($BC(Ph)CCH\mathit{C}H{=}C$), 83.2 (d, $^1J_{PC}$ = 69 Hz, cage $\mathit{C}P$), 80.5 (d, $^2J_{PC}$ = 17 Hz, cage $\mathit{C}B$), 60.8 ($BC(Ph)\mathit{C}$), 60.3 ($B\mathit{C}(Ph)C$), 48.2 ($BC(Ph)CH{=}C\mathit{C}H$), 46.1 ($BC(Ph)C\mathit{C}HCH{=}C$), 40.7 ($BC(Ph)CH{=}CC_6H_4N\mathit{C}H_3$), 40.36 ($BC(Ph)CCH{=}CHCN\mathit{C}H_3$), 40.30 ($BCH{=}CC_6H_4N\mathit{C}H_3$). ^{11}B NMR (128 MHz, CD_2Cl_2): δ 0.6 (not resolved, 3B), –7.9 (not resolved, 8B). ^{31}P NMR (162 MHz, CD_2Cl_2): δ 17.8. IR (KBr, cm^{-1}): ν_{max} 2569 (B–H). Anal. Calcd for $C_{56}H_{63}B_{11}N_3P$ (**4-Me$_3$**): C, 72.48; H, 6.84; N, 4.53. Found: C, 72.74; H, 7.01; N, 4.84.

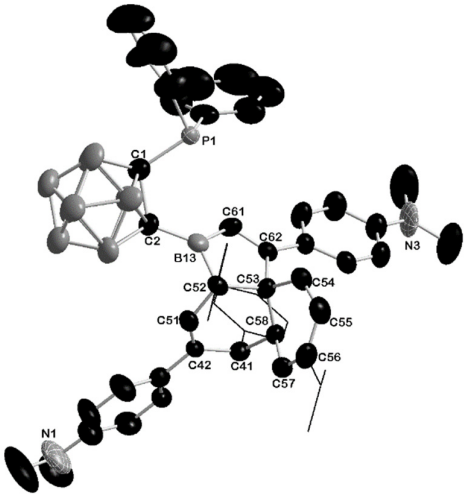

Figure S4. Molecular structure of **4-Me$_3$**. Two phenyls and one Me_2N group are shown in wireframe format for clarity.

Preparation of 4-Et$_3$. To a toluene (1 mL) suspension of **3-Et** (67 mg, 0.10 mmol) was added *p*-$Et_2NC_6H_4C{\equiv}CH$ (69 mg, 0.40 mmol), and the suspension was heated at 70 °C with stirring in a sealed flask for 14 d. After removal of the solvent, the red residue was thoroughly washed with *n*-hexane to afford **4-Et$_3$** as a red powder (51 mg, 50%). Single crystals suitable for X-ray analysis were grown by slow evaporation of a saturated THF solution. ^{1}H NMR (400 MHz, CD_2Cl_2): δ 7.77-7.70 (m, 4H, $PC_6\mathit{H}_5$), 7.48-7.40 (m, 4H + 1H, $PC_6\mathit{H}_5$ + $BCC_6\mathit{H}_5$), 7.34-7.31 (m, 2H + 2H, $PC_6\mathit{H}_5$ + $BC(Ph)CH{=}CC_6\mathit{H}_4$), 7.17-7.08 (m, 3H + 1H, $BCC_6\mathit{H}_5$ + BC(Ph)C*H*=C), 7.05-7.01 (m, 2H + 1H, $BCH{=}CC_6\mathit{H}_4$ + $BCC_6\mathit{H}_5$), 6.80-6.72 (m, 5H, BC(Ph)Ch=CCH$C_6\mathit{H}_5$), 6.57 (d, J = 8.9 Hz, 2H, $BC(Ph)CH{=}CC_6\mathit{H}_4$), 6.25 (d, J = 9.0 Hz, 2H, $BCH{=}CC_6\mathit{H}_4$), 5.94 (d, J = 3.0 Hz, 1H, BC*H*=C), 5.70 (d, J = 9.7 Hz, 1H, BC(Ph)CC*H*=CHC), 5.49 (dd, J_1 = 1.4 Hz, J_2 = 9.8 Hz, 1H, BC(Ph)CCH=C*H*C), 4.21 (br, 1H, BC(Ph)CC*H*CH=C), 4.08 (br, 1H, BC(Ph)CCHC*H*=C), 4.01 (br, 1H, BC(Ph)CH=CC*H*(Ph)), 3.46-3.31 (m, 4H, $BCH{=}CC_6H_4NC\mathit{H}_2$), 3.32 (q, J = 7.4 Hz, 4H, $BC(Ph)CH{=}CC_6H_4NC\mathit{H}_2$), 2.69-2.60 (m, 2H, BC(Ph)CCH=CHCNCH_2), 2.55-2.46 (m, 2H, BC(Ph)CCH=CHCNCH_2), 1.19 (t, J = 7.0 Hz, 6H, $BCH{=}CC_6H_4NCH_2C\mathit{H}_3$), 1.12 (t, J = 7.0 Hz, $BC(Ph)CH{=}CC_6H_4NCH_2CH_3$), 0.91 (t, J = 6.5 Hz, 6H, $BC(Ph)CCH{=}CHCNCH_2C\mathit{H}_3$). ^{13}C NMR (100 MHz, CD_2Cl_2): δ 194.0 (d, $^2J_{PC}$ = 3.5 Hz BCH=*C*), 149.0 ($BCH{=}C\mathit{C}_6H_4$), 147.1 ($BC(Ph)CH{=}C\mathit{C}_6H_4$), 144.8 ($BC\mathit{C}_6H_5$), 143.8 (BC(Ph)CCH=CH*C*), 140.8 ($BC(Ph)CH{=}CCH\mathit{C}_6H_5$), 137.8 (BC(Ph)C*C*H=CHC), 135.72 (d, $^2J_{PC}$ = 27 Hz, $P\mathit{C}_6H_5$), 135.43 (d, $^2J_{PC}$ = 25 Hz, $P\mathit{C}_6H_5$), 134.86 (B*C*H=C), 134.84 (d, J_{PC} = 5.1 Hz, $BC\mathit{C}_6H_5$), 134.73 (d, $^1J_{PC}$ = 15.7 Hz, $P\mathit{C}_6H_5$), 134.61 (d, $^1J_{PC}$ = 15.2 Hz, $P\mathit{C}_6H_5$), 134.1 (BC(Ph)CH=*C*), 131.6 ($BCH{=}C\mathit{C}_6H_4$), 130.93 ($BC\mathit{C}_6H_5$), 130.92 (d, $^4J_{PC}$ = 9.6 Hz, $P\mathit{C}_6H_5$), 129.9 ($BC(Ph)CH{=}CCH\mathit{C}_6H_5$), 129.05 (d, $^3J_{PC}$ = 9.6 Hz, $P\mathit{C}_6H_5$), 128.97 (d, $^3J_{PC}$ = 8.6 Hz, $P\mathit{C}_6H_5$), 128.5 ($BC(Ph)CH{=}C\mathit{C}_6H_4$), 127.9 (BC(Ph)*C*H=C), 127.51 ($BC\mathit{C}_6H_5$), 127.28

(BC(Ph)CH=CCH*C*$_6$H$_5$), 126.5 (BC(Ph)CH=C*C*$_6$H$_4$), 125.74 (BC(Ph)CH=CCH*C*$_6$H$_5$), 125.61, 125.0 (BC*C*$_6$H$_5$), 123.8 (BCH=C*C*$_6$H$_4$), 122.7 (BC(Ph)CCH=*C*HC), 111.6 (BC(Ph)CH=C*C*$_6$H$_4$), 110.3 (BCH=C*C*$_6$H$_4$), 100.7 (BC(Ph)CCH*C*H=C), 83.2 (d, $^1J_{PC}$ = 69 Hz, cage *C*P), 80.6 (d, $^2J_{PC}$ = 24 Hz, cage *C*B), 60.9 (BC(Ph)*C*CH=CHC), 60.2 (B*C*(Ph)CCH=CHC), 48.4 (BC(Ph)CH=C*C*H(Ph)), 46.2 (BC(Ph)C*C*HCH=C), 44.9 (BCH=CC$_6$H$_4$N*C*H$_2$), 44.6 (BC(Ph)CH=CC$_6$H$_4$N*C*H$_2$), 43.1 (BC(Ph)CCH=CHCN*C*H$_2$CH$_3$), 13.5 (BC(Ph)CCH=CHCNCH$_2$*C*H$_3$), 12.87, 12.84 (*C*H$_3$). ^{11}B NMR (128 MHz, CD_2Cl_2): δ 0.5 (not resolved, 3B), –7.9 (not resolved, 8B). ^{31}P NMR (162 MHz, CD_2Cl_2): δ 17.5. IR (KBr, cm^{-1}): ν_{max} 2582 (B–H). Anal. Calcd for $C_{62}H_{75}B_{11}N_3P$ (**4-Et$_3$**): C, 73.57; H, 7.47; N, 4.15. Found: C, 73.01; H, 7.95; N, 4.42.

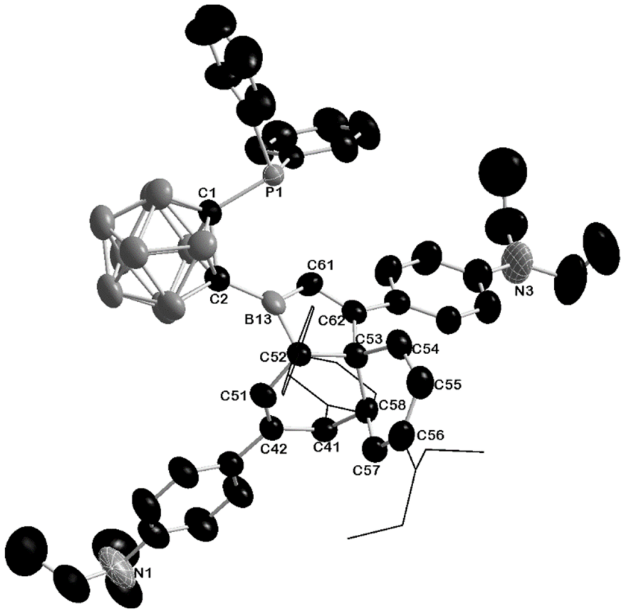

Figure S5. Molecular Structure of **4-Et$_3$**. Two phenyls and one Et$_2$N group are shown in wireframe format for clarity.

Preparation of 4-MeEt$_2$. To a toluene (1 mL) suspension of **3-Me** (64 mg, 0.10 mmol) was added *p*-Et$_2$NC$_6$H$_4$C≡CH (69 mg, 0.40 mmol), and the suspension was heated at 70 °C with stirring in a sealed flask for 14 d. After removal of the solvent, the red residue was thoroughly washed with *n*-hexane to afford **4-MeEt$_2$** as a pink powder (50 mg, 51%). ^{1}H NMR (400 MHz, CD_2Cl_2): δ 7.77-7.69 (m, 4H, PC$_6$*H*$_5$), 7.48-7.40 (m, 4H + 1H, PC$_6$*H*$_5$ + BCC$_6$*H*$_5$), 7.38-7.32 (m, 2H + 2H, BC(Ph)CH=CC$_6$*H*$_4$ + PC$_6$*H*$_5$), 7.15-7.08 (m, 1H + 3H, BC(Ph)C*H*=C + BCC$_6$*H*$_5$), 7.05-7.02 (m, 1H + 2H, BCC$_6$*H*$_5$ + BCH=CC$_6$*H*$_4$), 6.81-6.74 (m, 5H, BC(Ph)CH=CCHC$_6$*H*$_5$), 6.64 (d, J = 9.0 Hz, 2H, BC(Ph)CH=CC$_6$*H*$_4$), 6.26 (d, J = 9.2 Hz, BCH=CC$_6$*H*$_4$), 5.94 (d, J_{PH} = 3.0 Hz, 1H, BC*H*=C), 5.70 (d, J = 9.7 Hz, 1H, BC(Ph)CC*H*=CH), 5.50 (dd, J_1 = 9.8 Hz, J_2 = 1.7 Hz, 1H, BC(Ph)CCH=C*H*), 4.22

(brs, 1H, BC(Ph)CC*H*CH=C), 4.09 (brs, 1H, BC(Ph)CCHC*H*=C), 4.02 (d, J = 1.4 Hz, 1H, BC(Ph)CH=CC*H*), 3.46-3.31 (m, 4H, BCH=CC$_6$H$_4$NC*H*$_2$), 2.90 (s, 6H, NCH$_3$), 2.69-2.60 (m, 2H, BC(Ph)CCH=CHCNC*H*$_2$), 2.56-2.47 (m, 2H, BC(Ph)CCH=CHCNC*H*$_2$), 1.20 (t, J = 7.0 Hz, 6H, BCH=CC$_6$H$_4$NCH$_2$C*H*$_3$), 0.91 (t, J = 6.9 Hz, 6H, BC(Ph)CCH=CHCNCH$_2$C*H*$_3$). ^{13}C NMR (100 MHz, CD$_2$Cl$_2$): δ 194.2 (d, J_{PC} = 4.2 Hz, BCH=*C*), 150.0 (BC(Ph)CH=C*C*$_6$H$_4$), 149.1 (BCH=C*C*$_6$H$_4$), 144.7 (BC*C*$_6$H$_5$), 143.8 (BC(Ph)CCH=CH*C*NEt$_2$), 140.7 (BC(Ph)CH=CCH*C*$_6$H$_5$), 137.7 (BC(Ph)C*C*H=CH), 135.70 (d, $^2J_{PC}$ = 27.5 Hz, P*C*$_6$H$_5$), 135.44 (d, $^2J_{PC}$ = 25.9 Hz, P*C*$_6$H$_5$), 134.9 (B*C*H=C), 134.82 (d, J_{PC} = 4.2 Hz, BC*C*$_6$H$_5$), 134.72 (d, $^1J_{PC}$ = 15.8 Hz, P*C*$_6$H$_5$), 134.61 (d, $^1J_{PC}$ = 15.2 Hz, P*C*$_6$H$_5$), 134.0 (BC(Ph)CH=*C*CH(Ph)), 131.6 (BCH=C*C*$_6$H$_4$), 130.94 (d, $^4J_{PC}$ = 9.2 Hz,P*C*$_6$H$_5$), 130.89 (BC*C*$_6$H$_5$), 129.92 (BC(Ph)CH=CCH*C*$_6$H$_5$), 129.69 (BC(Ph)CH=C*C*$_6$H$_4$), 129.07 (d, $^3J_{PC}$ = 9.3 Hz, P*C*$_6$H$_5$), 128.99 (d, $^3J_{PC}$ = 8.6 Hz, P*C*$_6$H$_5$), 128.7 (BC(Ph)*C*H=C), 127.55 (BC*C*$_6$H$_5$), 127.28 (BC(Ph)CH=CCH*C*$_6$H$_5$), 126.3 (BC(Ph)CH=C*C*$_6$H$_4$), 125.77 (BC(Ph)CH=CCH*C*$_6$H$_5$), 125.65, 125.1 (BC*C*$_6$H$_5$), 123.8 (BCH=C*C*$_6$H$_4$), 122.7 (BC(Ph)CCH=*C*H), 112.6 (BC(Ph)CH=C*C*$_6$H$_4$), 110.4 (BCH=C*C*$_6$H$_4$), 100.6 (BC(Ph)CCH*C*H=C), 83.2 (d, $^1J_{PC}$ = 69 Hz, cage *C*P), 80.6 (d, $^2J_{PC}$ = 23 Hz, cage *C*B), 60.9 (BC(Ph)C), 60.2 (B*C*(Ph)C), 48.5 (BC(Ph)CH=C*C*H(Ph)), 46.2 (BC(Ph)C*C*HCH=C), 44.9 (BCH=CC$_6$H$_4$N*C*H$_2$CH$_3$), 43.1 (BC(Ph)CCH=CHCN*C*H$_2$CH$_3$), 40.6 (N*C*H$_3$), 13.5 (BC(Ph)CCH=CHCNCH$_2$*C*H$_3$), 12.9 (BCH=CC$_6$H$_4$NCH$_2$*C*H$_3$). ^{11}B NMR (128 MHz, CD$_2$Cl$_2$): δ 0.8 (not resolved, 4B), –7.7 (not resolved, 7B). ^{31}P NMR (162 MHz, CD$_2$Cl$_2$): δ 17.5. IR (KBr, cm^{-1}): ν_{max} 2566 (B–H). Anal. Calcd for C$_{60}$H$_{71}$B$_{11}$N$_3$P (**4-MeEt$_2$**): C, 73.23; H, 7.27; N, 4.27. Found: C, 72.87; H, 7.07; N, 4.68.

Preparation of 4-EtMe$_2$. To a toluene (1 mL) suspension of **3-Et** (67 mg, 0.10 mmol) was added *p*-Me$_2$NC$_6$H$_4$C≡CH (58 mg, 0.40 mmol), and the suspension was heated with stirring at 70 °C in a sealed flask for 14 d. After removal of the volatile materials, the red residue was thoroughly washed with *n*-hexane to afford **4-EtMe$_2$** as a yellow powder (49 mg, 51%). Single crystals suitable for X-ray analysis were grown by slow evaporation of a saturated THF solution. ^{1}H NMR (400 MHz, CD$_2$Cl$_2$): δ 7.76-7.70 (m, 4H, PC$_6$*H*$_5$), 7.48-7.41 (m, 4H + 1H, PC$_6$*H*$_5$ + BCC$_6$*H*$_5$), 7.36-7.31 (m, 2H + 2H, BC(Ph)CH=CC$_6$*H*$_4$ + PC$_6$*H*$_5$), 7.17-7.10 (m, 1H + 3H, BC(Ph)C*H*=C + BCC$_6$*H*$_5$), 7.08-7.03 (m, 1H + 2H, BCC$_6$*H*$_5$ + BCH=CC$_6$H$_4$), 6.80-6.77 (m, 5H, BC(Ph)CH=CCHC$_6$*H*$_5$), 6.58 (d, J = 9.0 Hz, 2H, BC(Ph)CH=CC$_6$*H*$_4$), 6.30 (d, J = 9.1 Hz, 2H, BCH=CC$_6$H$_4$), 5.97 (d, J_{PH} = 3.1 Hz, 1H, BC*H*=C), 5.72 (d, J = 9.7 Hz, 1H, BC(Ph)CC*H*=CH), 5.58 (dd, J_1 = 9.5 Hz, J_2 = 1.6 Hz, 1H, BC(Ph)CCH=C*H*), 4.20 (brs, 1H + 1H, BC(Ph)CC*H*CH=C + BC(Ph)CCHC*H*=C), 4.07 (brs, 1H, BC(Ph)CH=CC*H*), 3.32 (q, J = 7.4 Hz, 4H, NC*H*$_2$CH$_3$), 2.99 (s, 6H, BCH=CC$_6$H$_4$NC*H*$_3$), 2.20 (s, 6H, BC(Ph)CCH=CHCNC*H*$_2$), 1.13 (t, J = 7.0 Hz, 6H, NCH$_2$C*H*$_3$). ^{13}C NMR (100 MHz, CD$_2$Cl$_2$): δ 193.9 (d, J_{PC} = 3.9 Hz, BCH=*C*), 151.7 (BCH=C*C*$_6$H$_4$), 147.2 (BC(Ph)CH=C*C*$_6$H$_4$), 146.5

(BC(Ph)CCH=CH*C*NMe$_2$), 144.8 (BC*C*$_6$H$_5$), 140.6 (BC(Ph)CH=CCH*C*$_6$H$_5$), 137.6 (BC(Ph)C*C*H=CH), 135.70 (d, $^2J_{PC}$ = 27 Hz, P*C*$_6$H$_5$), 135.40 (d, $^2J_{PC}$ = 27 Hz, P*C*$_6$H$_5$), 135.6 (B*C*H=C), 135.0 (d, J_{PC} = 5.6 Hz, BC*C*$_6$H$_5$), 134.66 (d, $^1J_{PC}$ = 15.6 Hz, P*C*$_6$H$_5$), 134.52 (d, $^1J_{PC}$ = 15.6 Hz, P*C*$_6$H$_5$), 134.18 (BC(Ph)CH=*C*), 131.27 (BCH=C*C*$_6$H$_4$), 130.96 (d, $^4J_{PC}$ = 8.5 Hz, P*C*$_6$H$_5$), 130.92 (BC*C*$_6$H$_5$), 129.9 (BC(Ph)CH=CCH*C*$_6$H$_5$), 129.09 (d, $^3J_{PC}$ = 9.7 Hz, P*C*$_6$H$_5$), 129.00 (d, $^3J_{PC}$ = 8.6 Hz, P*C*$_6$H$_5$), 128.2 (BC(Ph)CH=C*C*$_6$H$_4$), 127.62 (BC(Ph)*C*H=C + BC*C*$_6$H$_5$), 127.36 (BC(Ph)CH=CCH*C*$_6$H$_5$), 126.5 (BC(Ph)CH=C*C*$_6$H$_4$), 125.97 (BC(Ph)CH=CCH*C*$_6$H$_5$), 125.80, 125.2 (BC*C*$_6$H$_5$), 124.8 (BCH=C*C*$_6$H$_4$), 122.9 (BC(Ph)CCH=*C*H), 111.7 (BC(Ph)CH=C*C*$_6$H$_4$), 110.8 (BCH=C*C*$_6$H$_4$), 104.6 (BC(Ph)CCH*C*H=C), 83.2 (d, $^1J_{PC}$ = 69 Hz, cage *C*P), 80.5 (d, $^2J_{PC}$ = 22 Hz, cage *C*B), 60.7 (BC(Ph)*C*), 60.3 (B*C*(Ph)C), 48.1 (BC(Ph)CH=C*C*H(Ph)), 46.1 (BC(Ph)C*C*HCH=C), 44.6 (N*C*H$_2$CH$_3$), 40.35, 40.29 (N*C*H$_3$), 12.9 (CH$_2$*C*H$_3$). ^{11}B NMR (128 MHz, CD_2Cl_2): δ 0.6 (not resolved, 4B), –7.8 (not resolved, 7B). ^{31}P NMR (162 MHz, CD_2Cl_2): δ 17.9. IR (KBr, cm^{-1}): ν_{max} 2565 (B–H). Anal. Calcd for $C_{58}H_{67}B_{11}N_3P$ (**4-EtMe$_2$**): C, 72.86; H, 7.06; N, 4.40. Found: C, 72.57; H, 7.39; N, 4.70.

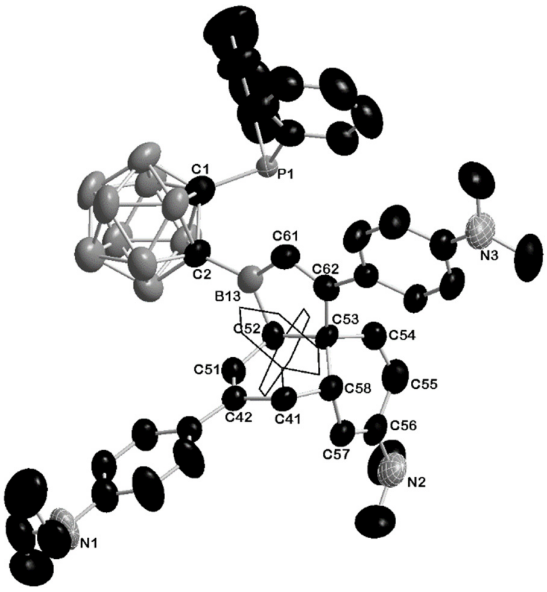

Figure S6. Molecular structure of **4-EtMe$_2$**. Two phenyl groups are shown in wireframe format for clarity.

Preparation of 4-PhMe$_2$. To a toluene (1 mL) suspension of **3-Ph** (76 mg, 0.10 mmol) was added *p*-Me$_2$NC$_6$H$_4$C≡CH (58 mg, 0.40 mmol), and the suspension was heated with stirring at 60 °C in a sealed flask for 4 d. After removal of the volatile materials, the pink residue was thoroughly washed with *n*-hexane to afford **4-PhMe$_2$** as a yellow powder (85 mg, 81%). ^{1}H NMR (400 MHz, CD_2Cl_2): δ 7.75-7.69 (m, 4H, PC$_6$*H*$_5$), 7.51-7.42 (m, 4H, PC$_6$*H*$_5$), 7.39-7.37 (m, 1H + 2H, BCC$_6$*H*$_5$ +

BC(Ph)CH=CC$_6$*H*$_4$), 7.34-7.31 (m, 2H, PC$_6$*H*$_5$), 7.26-7.22 (m, 4H + 1H, NC$_6$*H*$_5$ + BC(Ph)C*H*=C), 7.19-7.09 (m, 3H, BCC$_6$*H*$_5$), 7.07-7.03 (m, 4H + 2H + 1H, NC$_6$*H*$_5$ + BCH=CC$_6$*H*$_4$ + BCC$_6$*H*$_5$), 7.01 (t, J = 7.4 Hz, 2H, NC$_6$*H*$_5$), 6.94 (d, J = 8.7 Hz, 2H, BC(Ph)CH=CC$_6$*H*$_4$), 6.81-6.76 (m, 5H, BC(Ph)CH=CCHC$_6$*H*$_5$), 6.30 (d, J = 9.0 Hz, 2H, BC(Ph)CH=CC$_6$*H*$_4$), 5.97 (d, J_{PH} = 3.0 Hz, 1H, BC*H*=C), 5.70 (d, J = 9.7 Hz, 1H, BC(Ph)CC*H*=CH), 5.58 (dd, J_1 = 9.7 Hz, J_2 = 1.3 Hz, 1H, BC(Ph)CCH=C*H*), 4.21 (brs, 1H, BC(Ph)CC*H*CH=C), 4.19 (br, 1H, BC(Ph)CCHC*H*=C), 4.07 (brs, 1H, BC(Ph)CH=CC*H*), 2.98 (s, 6H, BCH=CC$_6$H$_4$NC*H*$_3$), 2.20 (s, 6H, BC(Ph)CCH=CHCNC*H*$_3$). ^{13}C NMR (100 MHz, CD$_2$Cl$_2$): δ 194.6 (BCH=*C*), 151.8 (BCH=C*C*$_6$H$_4$), 148.0 (N*C*$_6$H$_5$), 146.9 (BC(Ph)CH=C*C*$_6$H$_4$), 146.6 (BC(Ph)CCH=CH*C*), 144.5 (BC*C*$_6$H$_5$), 140.2 (BC(Ph)CH=CCH*C*$_6$H$_5$), 137.4 (BC(Ph)C*C*H=CH), 135.67 (d, $^2J_{PC}$ = 26.9 Hz, P*C*$_6$H$_5$), 135.67 (br, B*C*H=C), 135.51 (BC(Ph)CH=C*C*$_6$H$_4$), 135.38 (d, $^2J_{PC}$ = 25.1 Hz, P*C*$_6$H$_5$), 135.0 (d, J_{PC} = 5.9 Hz, BC*C*$_6$H$_5$), 134.56 (d, $^1J_{PC}$ = 15.7 Hz, P*C*$_6$H$_5$), 134.45 (d, $^1J_{PC}$ = 15.0 Hz, P*C*$_6$H$_5$), 133.8 (BC(Ph)CH=*C*), 131.37 (BCH=C*C*$_6$H$_4$), 131.30 (BC(Ph)*C*H=C), 131.00 (d, $^4J_{PC}$ = 7.4 Hz, P*C*$_6$H$_5$), 130.87 (BC*C*$_6$H$_5$), 129.83 (BC(Ph)CH=CCH*C*$_6$H$_5$), 129.62 (N*C*$_6$H$_5$), 129.11 (d, $^3J_{PC}$ = 9.9 Hz, P*C*$_6$H$_5$), 129.02 (d, $^3J_{PC}$ = 9.1 Hz, P*C*$_6$H$_5$), 127.67 (BC*C*$_6$H$_5$), 127.44 (BC(Ph)CH=CCH*C*$_6$H$_5$), 126.27 (BC(Ph)CH=C*C*$_6$H$_4$), 126.13 (BC(Ph)CH=CCH*C*$_6$H$_5$), 125.94, 125.3 (BC*C*$_6$H$_5$), 124.9 (N*C*$_6$H$_5$), 124.6 (BCH=C*C*$_6$H$_4$), 123.35 (N*C*$_6$H$_5$), 123.26 (BC(Ph)CH=C*C*$_6$H$_4$), 122.9 (BC(Ph)CCH=*C*H), 110.8 (BCH=C*C*$_6$H$_4$), 104.3 (BC(Ph)CCH*C*H=C), 83.2 (d, $^1J_{PC}$ = 69 Hz, cage CP), 80.3 (d, $^2J_{PC}$ = 21 Hz, cage *C*B), 60.8 (BC(Ph)*C*), 60.4 (B*C*(Ph)), 48.3 (BC(Ph)CH=C*C*HPh), 46.1 (BC(Ph)C*C*HCH=C), 40.34, 40.27 (N*C*H$_3$). ^{11}B NMR (128 MHz, CD$_2$Cl$_2$): δ 0.5 (not resolved, 4B), –7.7 (not resolved, 7B). ^{31}P NMR (162 MHz, CD$_2$Cl$_2$): δ 17.8. IR (KBr, cm^{-1}): ν_{max} 2568 (B–H). Anal. Calcd for C$_{66}$H$_{67}$B$_{11}$N$_3$P (**4-PhMe$_2$**): C, 75.34; H, 6.42; N, 3.99. Found: C, 75.51; H, 6.46; N, 4.33.

Preparation of 4-PhEt$_2$. To a toluene (1 mL) suspension of **3-Ph** (76 mg, 0.10 mmol) was added *p*-Et$_2$NC$_6$H$_4$C≡CH (69 mg, 0.40 mmol), and the suspension was heated with stirring at 60 °C in a sealed flask for 4 d. After removal of the volatile materials, the red residue was thoroughly washed with *n*-hexane to afford **4-PhEt$_2$** as a pink powder (85 mg, 77%). Single crystals suitable for X-ray analysis were grown from slow evaporation of a saturated THF solution. ^{1}H NMR (400 MHz, CD$_2$Cl$_2$): δ 7.78-7.70 (m, 4H, PC$_6$*H*$_5$), 7.52-7.40 (m, 4H, PC$_6$*H*$_5$), 7.40-7.37 (m, 1H + 2H, BCC$_6$*H*$_5$ + BC(Ph)CH=CC$_6$*H*$_4$), 7.35-7.32 (m, 2H, PC$_6$*H*$_5$), 7.26-7.22 (m, 4H + 1H, NC$_6$*H*$_5$ + BC(Ph)C*H*=C), 7.18-7.11 (m, 3H, BCC$_6$*H*$_5$), 7.08-7.03 (m, 4H + 2H, NC$_6$*H*$_5$ + BCH=CC$_6$*H*$_4$), 7.03-6.99 (m, 1H + 2H, BCC$_6$*H*$_5$ + NC$_6$*H*$_5$), 6.94 (d, J = 8.7 Hz, 2H, BC(Ph)CH=CC$_6$*H*$_4$), 6.83-6.76 (m, 5H, BC(Ph)CH=CCHC$_6$*H*$_5$), 6.26 (d, J = 9.0 Hz, 2H, BC(Ph)CH=CC$_6$*H*$_4$), 5.96 (d, J_{PH} = 2.9 Hz, 1H, BC*H*=C), 5.69 (d, J = 9.7 Hz, 1H, BC(Ph)CC*H*=CH), 5.51 (dd, J_1 = 9.8 Hz, J_2 = 1.5 Hz, 1H, BC(Ph)CCH=C*H*), 4.25 (brs, 1H, BC(Ph)CC*H*CH=C), 4.09 (brs, 1H, BC(Ph)CCHC*H*=C), 4.03 (brs,

1H, BC(Ph)CH=CC*H*), 3.46-3.32 (m, 4H, BCH=CC$_6$H$_4$NC*H*$_2$), 2.71-2.62 (m, 2H, BC(Ph)CCH=CHCNC*H*$_2$), 2.60-2.48 (m, 2H, BC(Ph)CCH=CHCNC*H*$_2$), 1.20 (t, *J* = 7.0 Hz, 6H, BCH=CC$_6$H$_4$NCH$_2$C*H*$_3$), 0.93 (t, 7.0 Hz, 6H, BC(Ph)CCH=CHCNCH$_2$C*H*$_3$). ^{13}C NMR (100 MHz, CD_2Cl_2): *δ* 194.6 (d, J_{PC} = 3.9 Hz, BCH=*C*), 149.2 (BCH=C*C*$_6$H$_4$), 148.0 (N*C*$_6$H$_5$), 146.8 (BC(Ph)CH=C*C*$_6$H$_4$), 144.5 (BC*C*$_6$H$_5$), 143.9 (BC(Ph)CCH=CH*C*), 140.4 (BC(Ph)CH=CCH*C*$_6$H$_5$), 137.7 (BC(Ph)C*C*H=CH), 135.83 (BC(Ph)CH=C*C*$_6$H$_4$), 135.70 (d, $^2J_{PC}$ = 27 Hz, P*C*$_6$H$_5$), 135.42 (d, $^2J_{PC}$ = 25.2 Hz, P*C*$_6$H$_5$), 135.01 (BC*H*=C), 134.85 (d, J_{PC} = 6.0 Hz, BC*C*$_6$H$_5$), 134.73 (d, $^1J_{PC}$ = 16.0 Hz, P*C*$_6$H$_5$), 134.56 (d, $^1J_{PC}$ = 15.2 Hz, P*C*$_6$H$_5$), 133.8 (BC(Ph)CH=*C*CH), 131.72 (BCH=C*C*$_6$H$_4$), 131.59 (BC(Ph)*C*H=CCH), 130.97 (d, $^4J_{PC}$ = 8.7 Hz, P*C*$_6$H$_5$), 130.8 (BC*C*$_6$H$_5$), 129.85 (BC(Ph)CH=CCH*C*$_6$H$_5$), 129.62 (N*C*$_6$H$_5$), 129.09 (d, $^3J_{PC}$ = 9.1 Hz, P*C*$_6$H$_5$), 129.00 (d, $^3J_{PC}$ = 8.2 Hz, P*C*$_6$H$_5$), 127.56 (BC*C*$_6$H$_5$), 127.38 (BC(Ph)CH=CCH*C*$_6$H$_5$), 126.3 (BC(Ph)CH=C*C*$_6$H$_4$), 125.92 (BC(Ph)CH=CCH*C*$_6$H$_5$), 125.77, 125.13 (BC*C*$_6$H$_5$), 124.9 (N*C*$_6$H$_5$), 123.7 (BCH=C*C*$_6$H$_4$), 123.32 (N*C*$_6$H$_5$), 123.26 (BC(Ph)CH=C*C*$_6$H$_4$), 122.8 (BC(Ph)CCH=*C*H), 110.4 (BCH=C*C*$_6$H$_4$), 100.4 (BC(Ph)CCH*C*H=C), 83.3 (d, $^1J_{PC}$ = 69 Hz, cage *C*P), 80.5 (d, $^2J_{PC}$ = 22 Hz, cage *C*B), 61.0 (BC(Ph)*C*), 60.4 (B*C*(Ph)C), 48.6 (BC(Ph)CH=C*C*H), 46.2 (BC(Ph)C*C*HCH=C), 44.9 (BCH=CC$_6$H$_4$NC*H*$_2$), 43.1 (BC(Ph)CCH=CHCN*C*H$_2$), 13.5 (BC(Ph)CCH=CHCNCH$_2$*C*H$_3$), 12.9 (BCH=CC$_6$H$_4$NCH$_2$*C*H$_3$). ^{11}B NMR (128 MHz, CD_2Cl_2): *δ* 0.5 (not resolved, 4B), –7.9 (not resolved, 7B). ^{31}P NMR (162 MHz, CD_2Cl_2): *δ* 17.6. IR (KBr, cm^{-1}): ν_{max} 2565 (B–H). Anal. Calcd for $C_{70}H_{75}B_{11}N_3P$ (**4-PhEt$_2$**): C, 75.86; H, 6.82; N, 3.79. Found: C, 75.65; H, 7.07; N, 3.94.

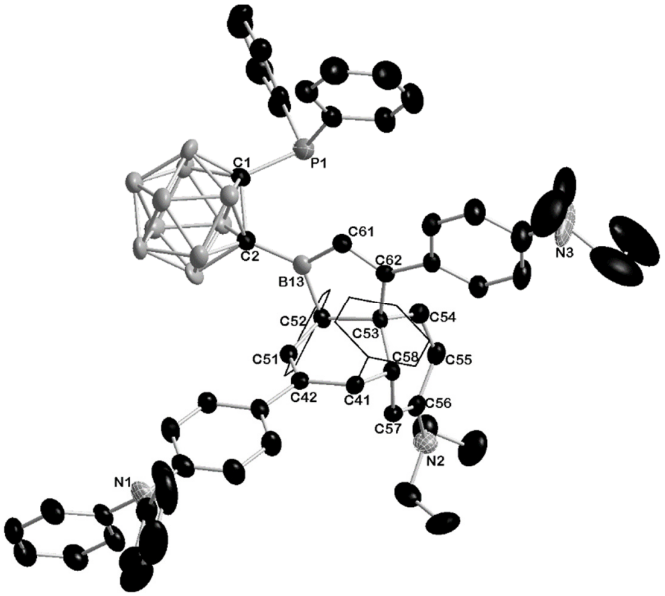

Figure S7. Molecular structure of **4-PhEt$_2$**. Two phenyl groups are shown in wireframe format for clarity.

Preparation of 1,2-{$Ph_2PCH_2OB(Ph)[C(C_6H_4NPh_2)=CHPh]$}-1,2-$C_2B_{10}H_{10}$ (5-Ph). To a toluene (0.5 mL) suspension of **3-Ph** (30 mg, 0.04 mmol) was added $(CH_2O)_n$ (6 mg, 0.80 mmol), and the suspension was heated at 70 °C in a closed tube for 7 d. The reaction was monitored by ^{31}P NMR till the signal of the starting material **3-Ph** disappeared. After removal of the volatile materials, the resulting white residue was thoroughly washed with cold Et_2O to afford **5-Ph** as a white powder (12 mg, 38%). Single crystals suitable for X-ray analysis were grown by slow evaporation of a saturated THF solution. ^1H NMR (400 MHz, CD_2Cl_2): δ 8.23-8.18 (m, 2H, PC_6H_5), 8.01-7.96 (m, 2H, PC_6H_5), 7.90-7.87 (m, 1H, PC_6H_5), 7.85-7.81 (m, 1H, PC_6H_5), 7.76-7.72 (m, 2H, PC_6H_5), 7.69-7.64 (m, 2H + 2H, PC_6H_5 + BC_6H_5), 7.24-7.17 (m, 2H + 1H + 4H, BC_6H_5 + BC=C*H* + NC_6H_5), 7.15-7.09 (m, 1H + 2H, BC_6H_5 + BC=CHC_6H_5), 7.04-6.99 (m, 1H + 4H, BC=CHC_6H_5 + NC_6H_5), 6.95-6.91 (m, 2H + 2H, NC_6H_5 + BC=CHC_6H_5), 6.77-6.71 (m, 4H, C_6H_4), 5.06 (dd, J_1 = 14.4 Hz, J_{PH} = 2.2 Hz, 1H, CH_2O), 4.84 (dd, J_1 = 14.4 Hz, J_{PH} = 3.6 Hz, 1H, CH_2O). ^{13}C NMR (100 MHz, CD_2Cl_2): δ 148.5 (NC_6H_5), 144.8, 142.6 (C_6H_4), 140.4 (BC=CHC_6H_5), 136.14 (d, $^4J_{PC}$ = 3.5 Hz, PC_6H_5), 136.11 (d, $^4J_{PC}$ = 3.9 Hz, PC_6H_5), 135.72 (d, $^2J_{PC}$ = 8.8 Hz, PC_6H_5), 135.10 (d, $^2J_{PC}$ = 8.9 Hz, PC_6H_5), 134.96 (BC=*C*H), 133.4 (BC_6H_5), 130.58 (C_6H_4), 130.45 (d, $^3J_{PC}$ = 12 Hz, PC_6H_5), 130.16 (d, $^3J_{PC}$ = 12 Hz, PC_6H_5), 129.89 (BC=CHC_6H_5), 129.4 (NC_6H_5), 127.8 (BC=CHC_6H_5), 127.0 (BC_6H_5), 125.68, 125.66 (C_6H_5), 124.8 (C_6H_4), 123.7, 122.4 (NC_6H_5), 117.06 (d, $^1J_{PC}$ = 84 Hz, PC_6H_5), 116.58 (d, $^1J_{PC}$ = 77 Hz) (PC_6H_5), 65.5 (d, $^1J_{PC}$ = 59 Hz, cage *C*P), 61.2 (d, $^1J_{PC}$ = 52 Hz, PCH_2O). ^{11}B NMR (128 MHz, CD_2Cl_2): δ 3.0 (not resolved, 2B), –2.8 (not resolved, 1B), –5.0 (not resolved, 2B), –7.9 (not resolved, 2B), –10.8 (not resolved, 4B). ^{31}P NMR (162 MHz, CD_2Cl_2): δ 3.7. IR (KBr, cm^{-1}): ν_{max} 2570 (B–H). HRMS (ESI): *m*/*z* calcd for $C_{47}H_{47}{}^{11}B_9{}^{10}B_2NNaOP$ $[M+Na]^+$: 814.4407. Found: *m*/*z* 814.4403.

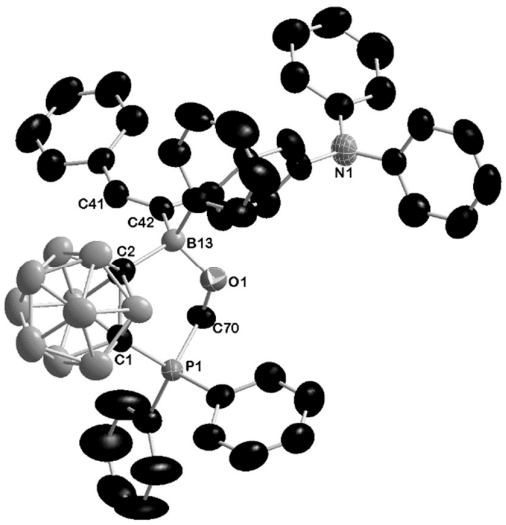

Figure S8. Molecular Structure of **5-Ph**

X-ray Structure Determination. X-ray data were collected at 293 K on a Bruker SMART 1000 CCD diffractometer using Mo-Kα radiation. An empirical absorption correction was applied using the SADABS program.[3] All structures were solved by direct methods and subsequent Fourier difference techniques and refined anisotropically for all non-hydrogen atoms by full-matrix least squares calculations on F^2 using the SHELXTL program package.[4] All hydrogen atoms were geometrically fixed using the riding model.

CCDC 2026881-2026888 for **2**, **3-Me**, **3-Ph**, **4-Et$_3$**, **4-EtMe$_2$**, **4-Me$_3$**, **4-PhEt$_2$** and **5-Ph** contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Table S1. Crystal Data and Summary of Data Collection and Refinement for **2**, **3-Me**, **3-Ph**·$(Et_2O)_2$, and **4-Et$_3$**.

compd. No.	**2**	**3-Me**	**3-Ph**·$(Et_2O)_2$	**4-Et$_3$**
formula	$C_{26}H_{30}B_{11}P$	$C_{36}H_{41}B_{11}NP$	$C_{54}H_{65}B_{11}NO_2P$	$C_{62}H_{75}B_{11}N_3P$
crystal size (mm)	0.50 x 0.40 x 0.30	0.50 x 0.40 x 0.30	0.50 x 0.40 x 0.30	0.50 x 0.40 x 0.30
fw	492.4	637.6	910.0	1012.1
crystal system	Monoclinic	Monoclinic	Triclinic	Triclinic
space group	$P2_1/c$	$P2_1/n$	P-1	P-1
a, Å	14.029(2)	11.5120(13)	13.061(2)	14.553(3)
b, Å	9.9593(19)	22.661(3)	13.363(3)	14.829(3)
c, Å	20.562(3)	14.8847(17)	17.006(3)	15.990(3)
α, deg	90	90	91.902	69.051(4)
β, deg	92.648(4)	95.426(3)	112.387(3)	79.173(4)
γ, deg	90	90	107.918(3)	70.065(4)
V, Å^3	2869.9(9)	3865.6(8)	2572.9(8)	3021.0(9)
Z	4	4	2	2
D_{calcd}, Mg/m^3	1.140	1.096	1.175	1.113
radiation (λ), Å	Mo Kα (0.71073)	Mo Kα (0.71073)	Mo Kα (0.71073)	Mo Kα (0.71073)
2θ max, deg	50.5	50.5	50.5	50.5
μ, mm^{-1}	0.112	0.097	0.096	0.086
F(000)	1024	1336	964	1076
no. of obsd reflns	5203	7000	9054	10919
no. of params refnd	343	442	622	695
goodness of fit	1.021	0.893	1.016	0.973
R1	0.0636	0.0666	0.0694	0.0874
wR2	0.1547	0.1593	0.1709	0.1997

Table S2. Crystal Data and Summary of Data Collection and Refinement for **4-EtMe$_2$**, **4-Me$_3$**, **4-PhEt$_2$**·(THF)$_2$, and **5-Ph**·THF.

compd. No.	**4-EtMe$_2$**	**4-Me$_3$**	**4-PhEt$_2$**·(THF)$_2$	**5-Ph**·THF
formula	$C_{58}H_{67}B_{11}N_3P$	$C_{56}H_{63}B_{11}N_3P$	$C_{78}H_{91}B_{11}N_3O_2P$	$C_{51}H_{55}B_{11}NO_2P$
crystal size (mm)	0.19 x 0.16 x 0.10	0.40 x 0.30 x 0.20	0.40 x 0.30 x 0.20	0.30 x 0.10 x 0.06
fw	956.0	928.0	1252.4	863.8
crystal system	Triclinic	Monoclinic	Triclinic	Monoclinic
space group	*P*-1	$P2_1/c$	*P*-1	$P2_1/c$
a, Å	11.524(3)	15.0697(18)	14.840(2)	12.189(6)
b, Å	14.772(4)	14.0752(18)	15.233(2)	21.845(11)
c, Å	20.643(6)	29.446(4)	17.153(2)	22.102(11)
α, deg	77.743(5)	90	78.440(3)	90
β, deg	77.242(5)	98.860(3)	77.680(3)	102.623(9)
γ, deg	71.242(5)	90	74.344(3)	90
V, Å^3	3207.3(16)	6171.3(13)	3605.5(8)	5743(5)
Z	2	4	2	4
D_{calcd}, Mg/m^3	0.990	0.999	1.154	0.999
radiation (λ), Å	Mo Kα (0.71073)	Mo Kα (0.71073)	Mo Kα (0.71073)	Mo Kα (0.71073)
2θ max, deg	50.5	50.5	50.5	50.5
μ, mm^{-1}	0.078	0.079	0.087	0.083
F(000)	1012	1960	1332	1816
no. of obsd reflns	11548	11162	13017	10373
no. of params refnd	658	641	856	595
goodness of fit	0.840	0.865	1.002	0.927
R1	0.0857	0.0829	0.0834	0.0949
*w*R2	0.2219	0.1917	0.1880	0.2313

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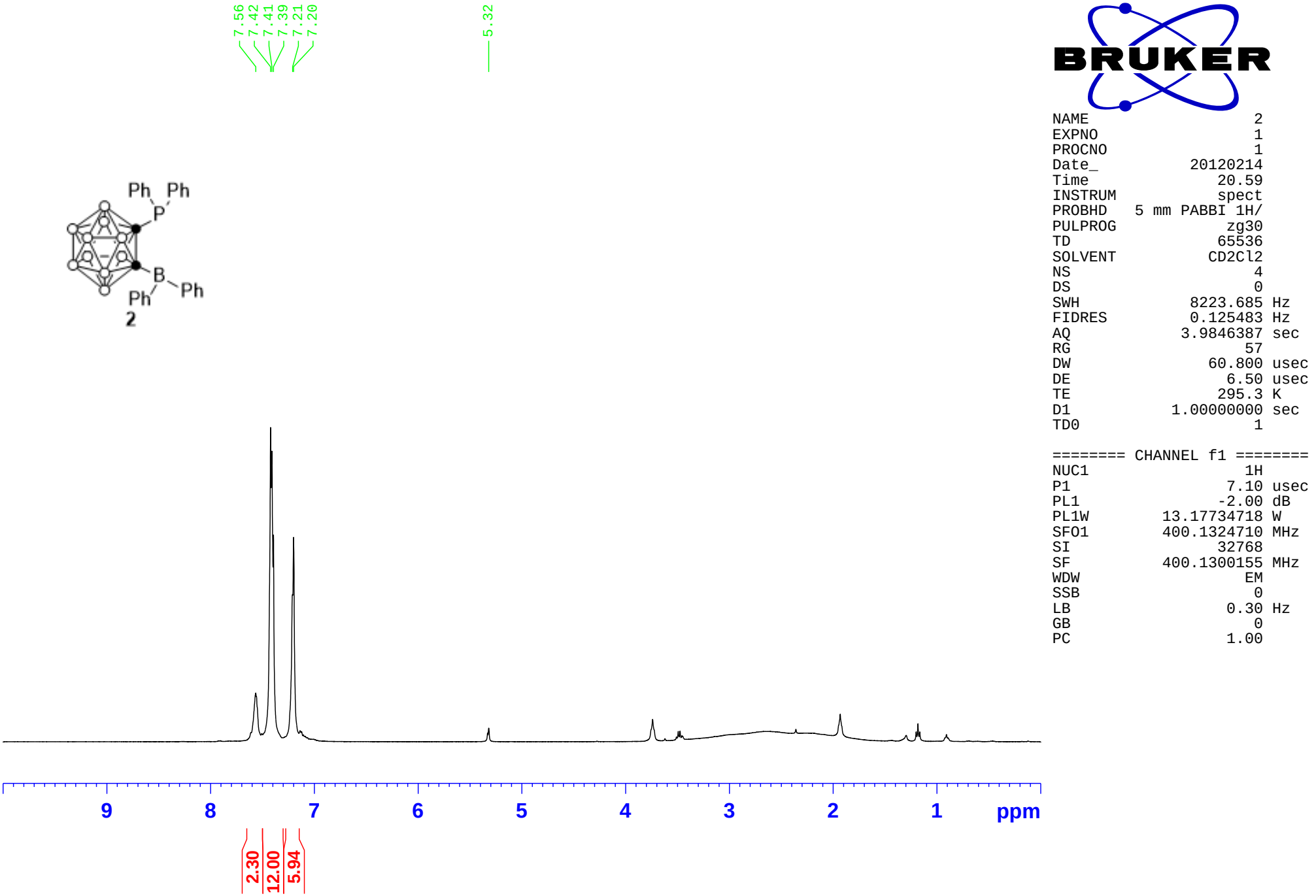
[4]. G. M. Sheldrick, SHELXTL 5.10 for Windows NT: Structure Determination Software Programs. Bruker Analytical X-ray Systems, Inc., Madison, Wisconsin, USA, **1997**.

Author Contributions

Z.X. directed and conceived this project. J.Z. conducted the experimental work. Both authors discussed the results and wrote the manuscript.



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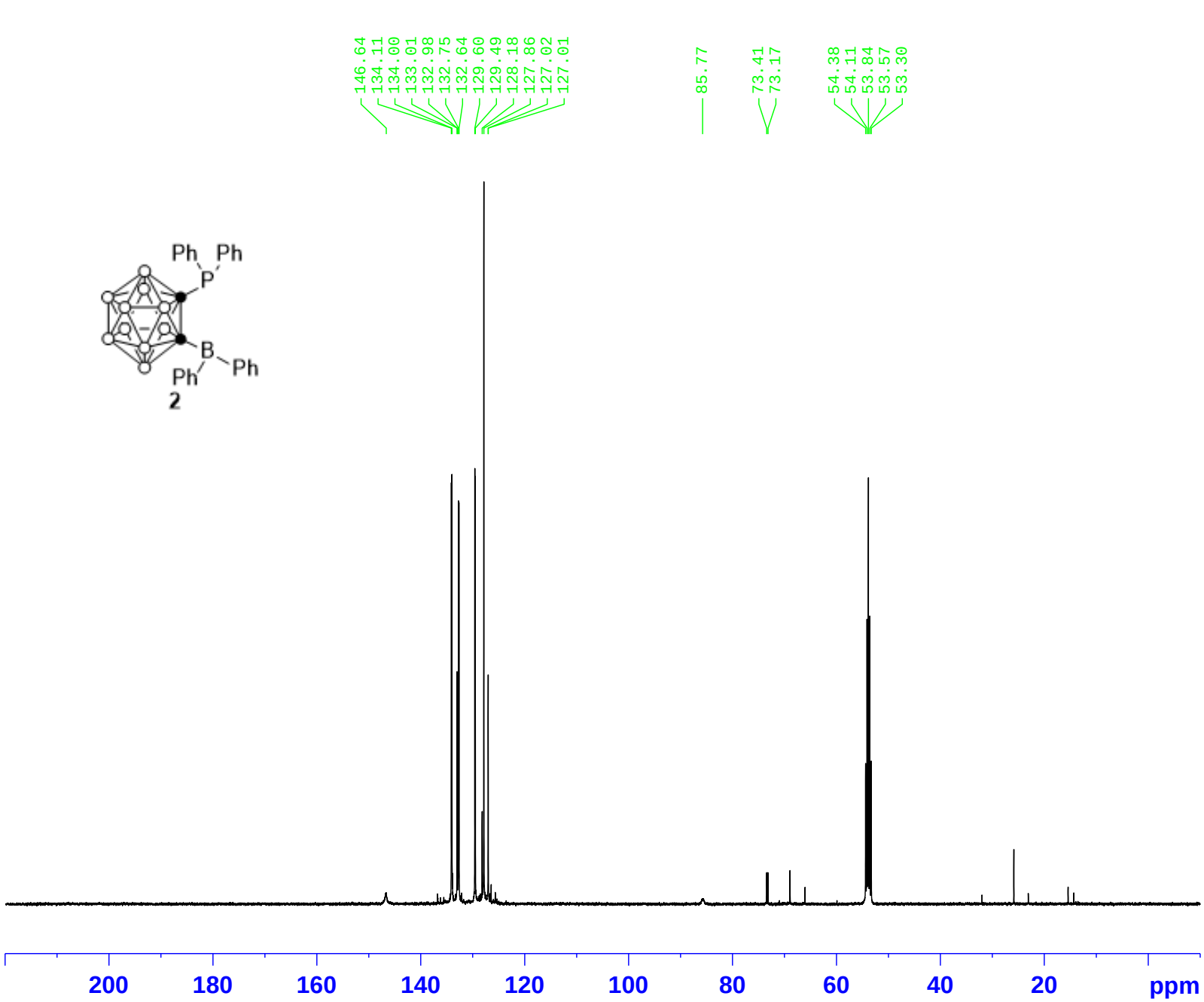

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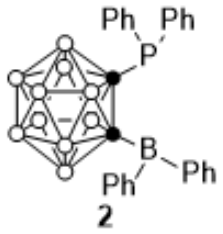
^{11}B



2

$^{11}B\{^{1}H\}$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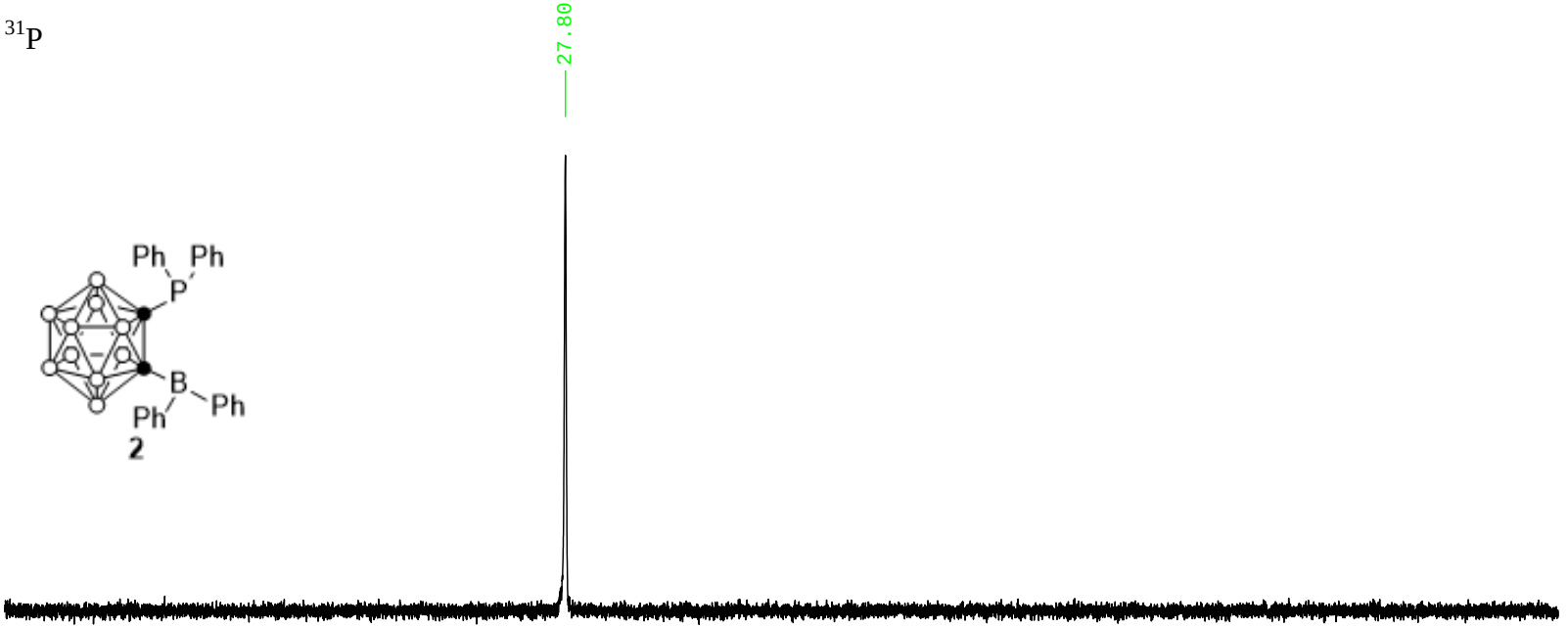
======== CHANNEL f1 ========
NUC1                11B
P1                10.00 usec
PL1               -4.00 dB
PL1W        63.09573364 W
SFO1        128.3776076 MHz

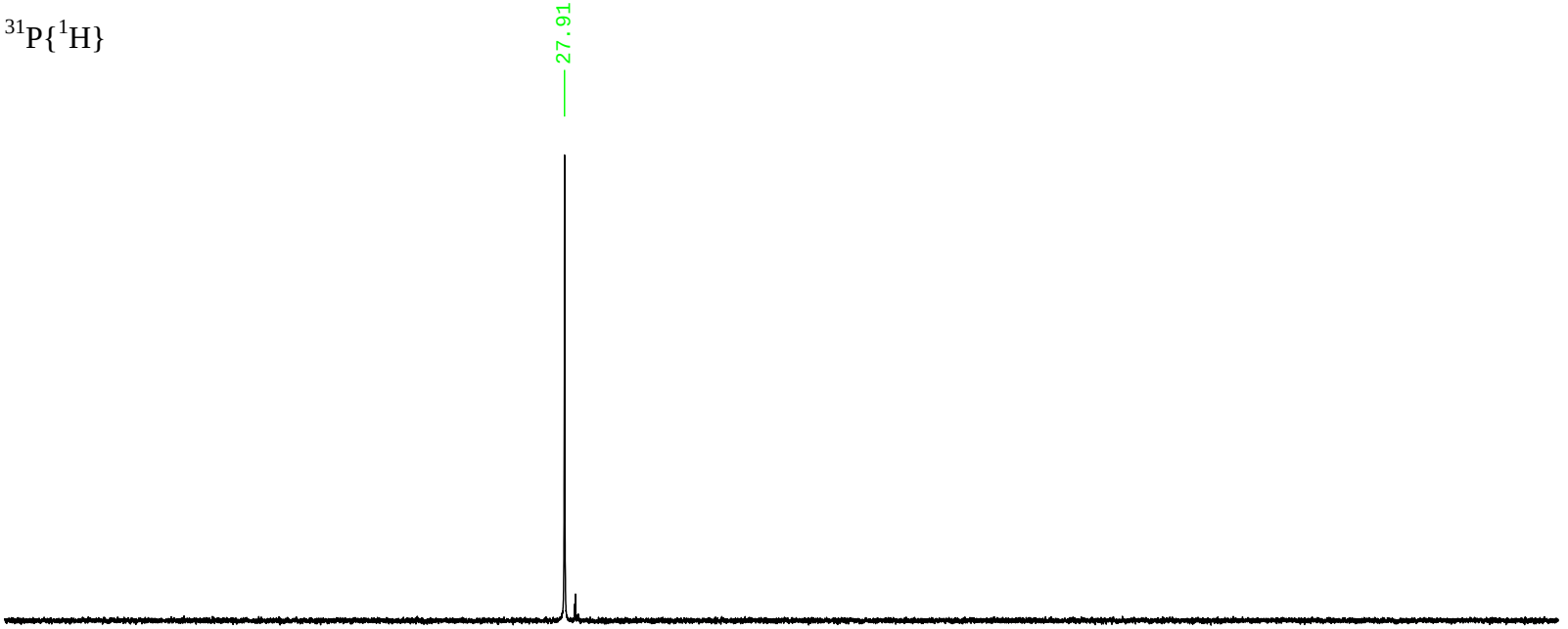
======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2            90.00 usec
PL2               -2.00 dB
PL12              20.06 dB
PL2W        13.17734718 W
PL12W        0.08200268 W
SFO2        400.1316005 MHz
SI                  2048
SF          128.3776497 MHz
WDW                 EM
SSB                  0
LB                2.00 Hz
GB                   0
PC                1.40
```

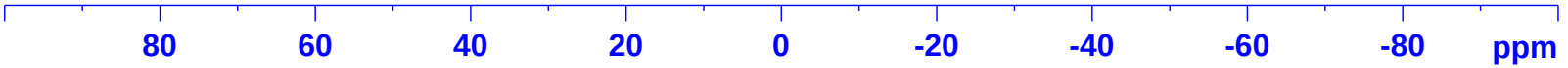
^{31}P



2

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





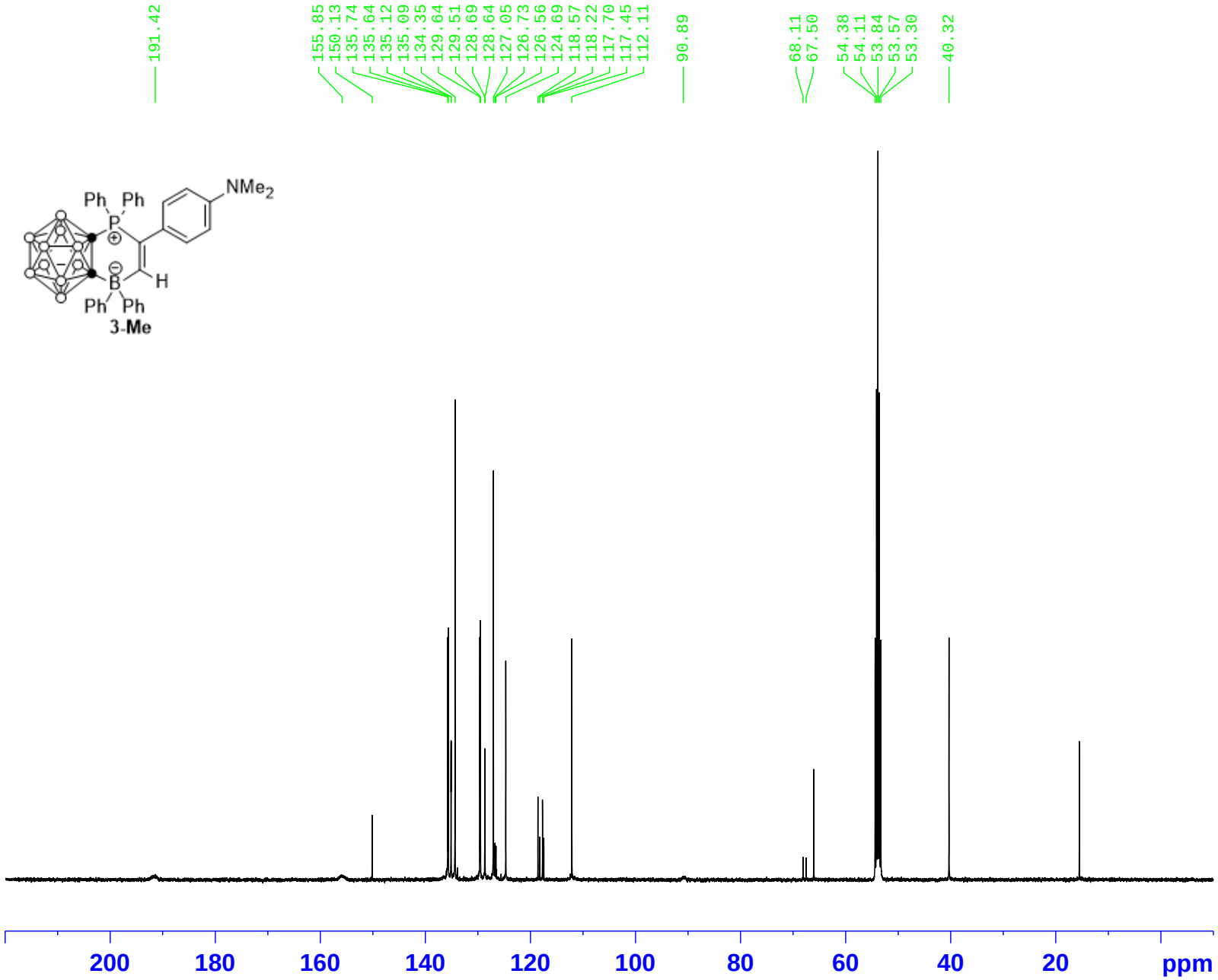

```
NAME                   2
EXPNO                 13
PROCNO                 1
Date_           20120413
Time               20.10
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG          zgdc30
TD                65536
SOLVENT           CD2Cl2
NS                    13
DS                     0
SWH            64102.563 Hz
FIDRES          0.978127 Hz
AQ             0.5112308 sec
RG                  2050
DW                 7.800 usec
DE                 6.50 usec
TE                299.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 31P
P1                 14.70 usec
PL1                 4.00 dB
PL1W        10.30000019 W
SFO1        161.9917814 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              90.00 usec
PL2                -1.00 dB
PL12               15.16 dB
PL2W        13.56617069 W
PL12W        0.32844096 W
SFO2        400.1916008 MHz
SI                 32768
SF          161.9997813 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

```
NAME                  3-Me
EXPNO                    1
PROCNO                   1
Date_             20120423
Time                 22.39
INSTRUM              spect
PROBHD    5 mm PABBI 1H/
PULPROG               zg30
TD                   65536
SOLVENT             CD2Cl2
NS                       16
DS                        0
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                     71.8
DW                  60.800 usec
DE                   6.50 usec
TE                  294.6 K
D1              1.00000000 sec
TD0                      1

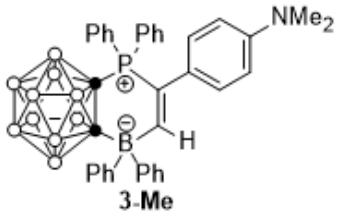
======== CHANNEL f1 ========
NUC1                    1H
P1                   7.10 usec
PL1                 -2.00 dB
PL1W          13.17734718 W
SFO1          400.1324710 MHz
SI                   32768
SF            400.1300154 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                    1.00
```



BRUKER

NAME	ch-1408-1-C
EXPNO	2
PROCNO	1
Date_	20120423
Time	22.41
INSTRUM	spect
PROBHD	5 mm PABBI 1H/
PULPROG	zgpg30
TD	65536
SOLVENT	CD2Cl2
NS	10434
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	294.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	14.50 usec
PL1	-4.00 dB
PL1W	90.22689819 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.00 dB
PL12	20.06 dB
PL13	22.00 dB
PL2W	13.17734718 W
PL12W	0.08200268 W
PL13W	0.05245997 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127298 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

^{11}B

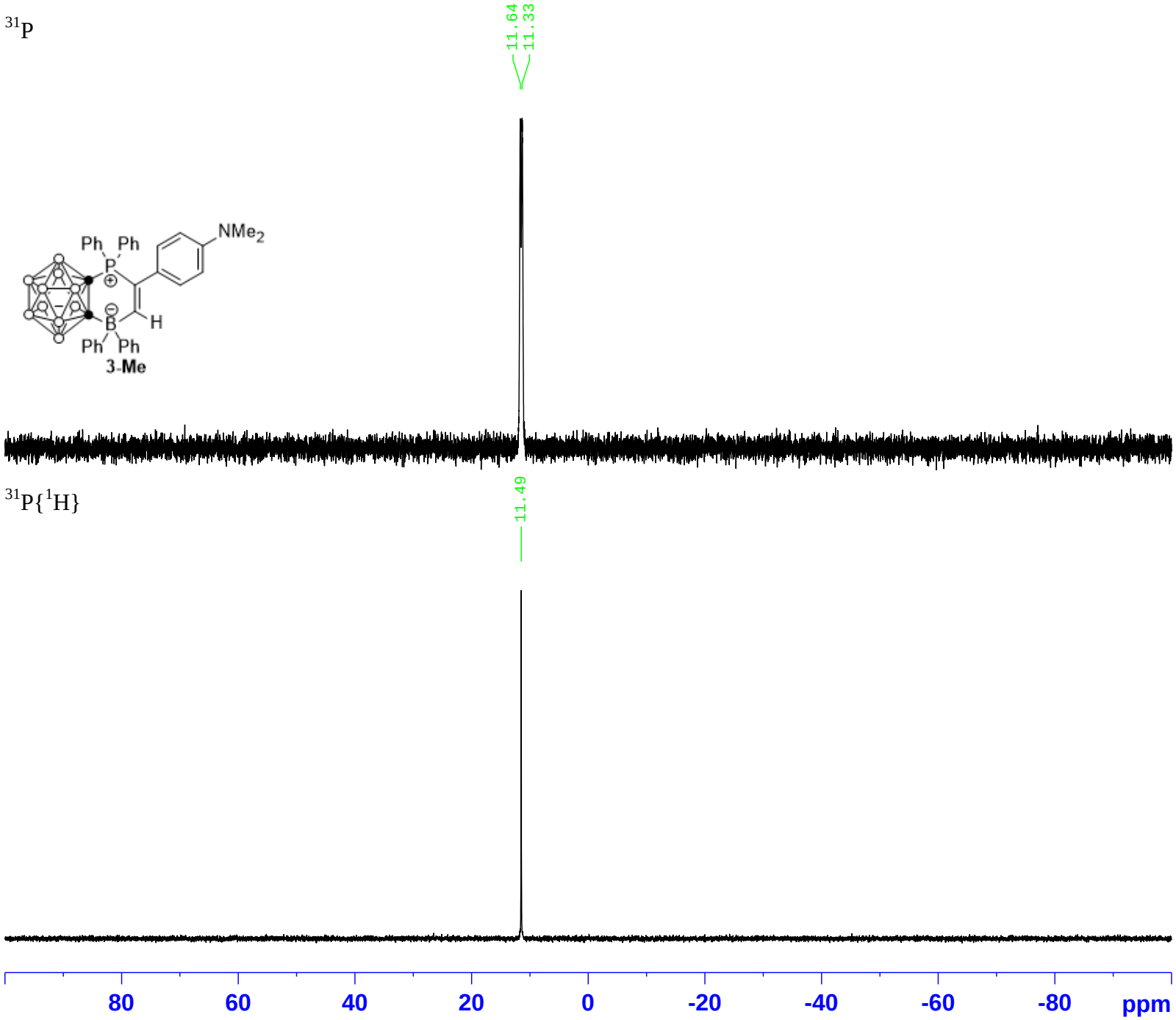


$^{11}B\{^{1}H\}$

```
NAME                  3-Me
EXPNO                   11
PROCNO                   1
Date_             20120421
Time                 16.26
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zgdc
TD                   65536
SOLVENT             CD2Cl2
NS                       8
DS                       0
SWH              25510.203 Hz
FIDRES            0.389255 Hz
AQ                1.2845556 sec
RG                     322
DW                  19.600 usec
DE                   6.50 usec
TE                  300.1 K
D1               5.00000000 sec
D11              0.03000000 sec
TD0                      1

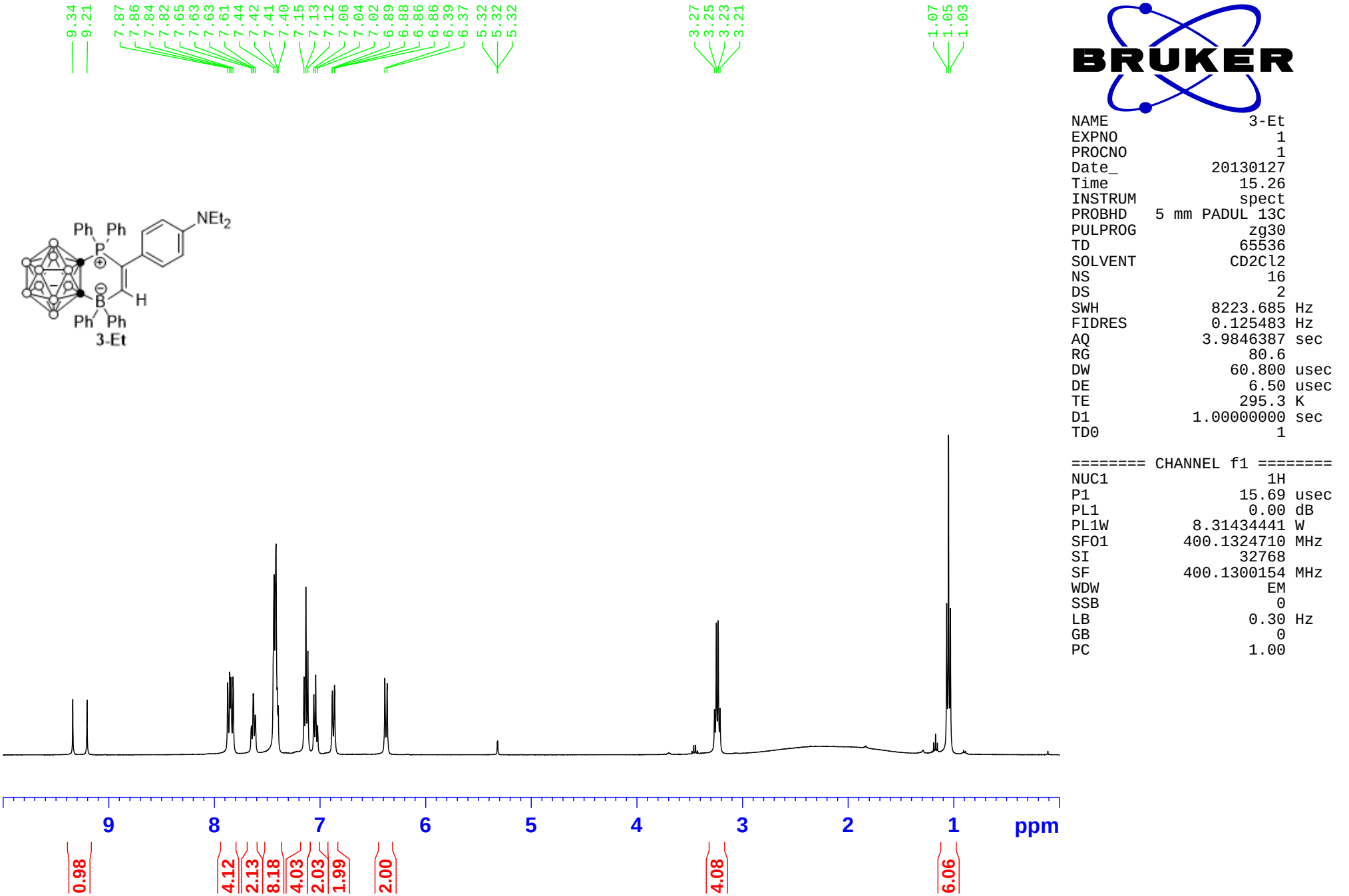
======== CHANNEL f1 ========
NUC1                   11B
P1                    7.60 usec
PL1                  -3.00 dB
PL1W          55.13059616 W
SFO1          128.3968556 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2                90.00 usec
PL2                  -1.00 dB
PL12                 15.16 dB
PL2W          13.56617069 W
PL12W          0.32844096 W
SFO2          400.1916008 MHz
SI                   32768
SF            128.3968897 MHz
WDW                    EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.40
```



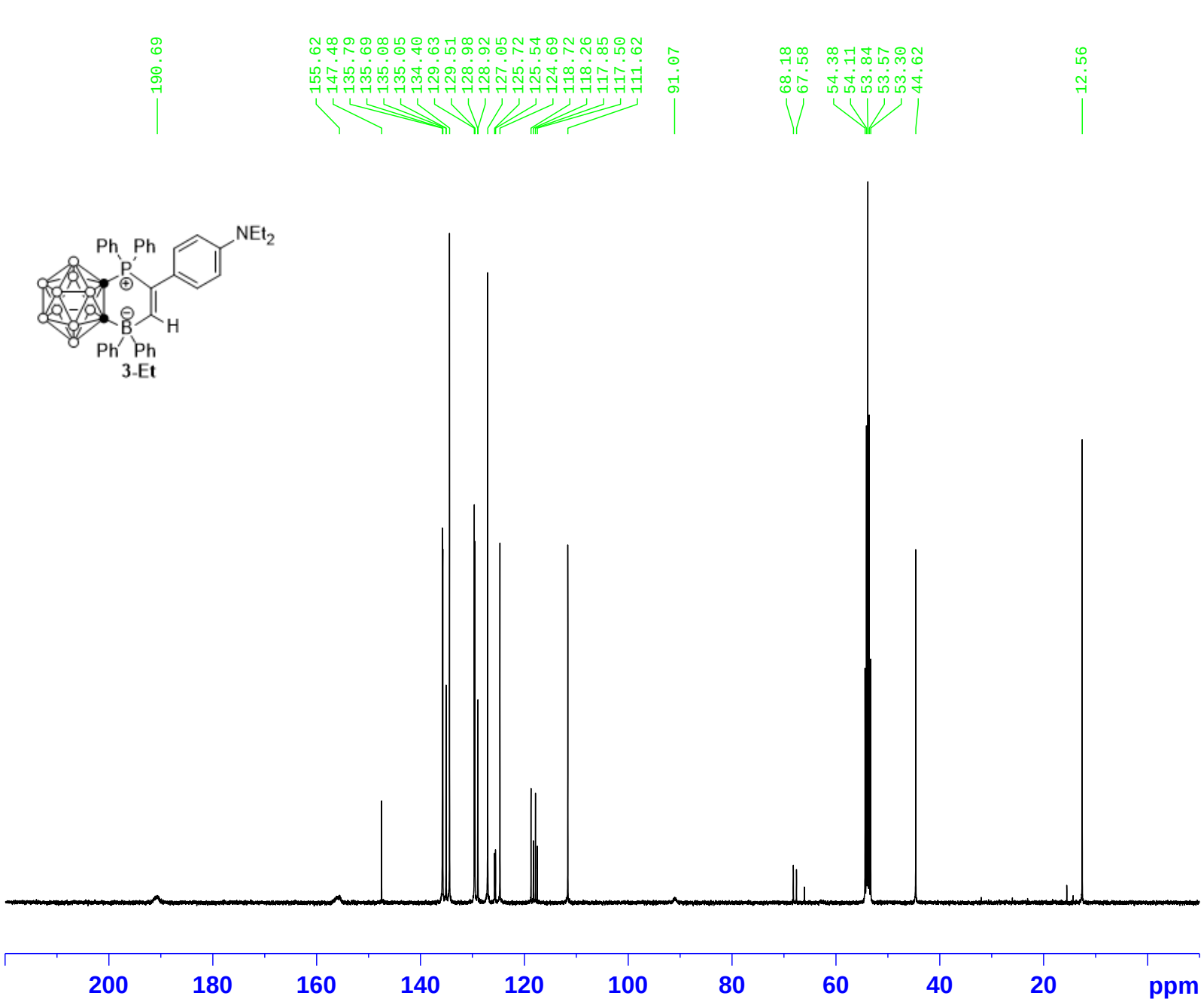
BRUKER

NAME	3-Me
EXPNO	13
PROCNO	1
Date_	20120421
Time	16.20
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgdc30
TD	65536
SOLVENT	CD2Cl2
NS	16
DS	0
SWH	64102.563 Hz
FIDRES	0.978127 Hz
AQ	0.5112308 sec
RG	2050
DW	7.800 usec
DE	6.50 usec
TE	300.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	31P
P1	14.70 usec
PL1	4.00 dB
PL1W	10.30000019 W
SFO1	161.9917814 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-1.00 dB
PL12	15.16 dB
PL2W	13.56617069 W
PL12W	0.32844096 W
SFO2	400.1916008 MHz
SI	32768
SF	161.9997813 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



BRUKER

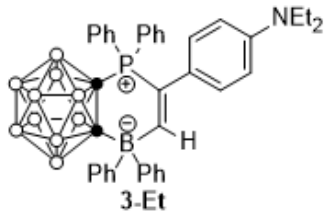
NAME	3-Et
EXPNO	1
PROCNO	1
Date_	20130127
Time	15.26
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CD2Cl2
NS	16
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	80.6
DW	60.800 usec
DE	6.50 usec
TE	295.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	15.69 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300154 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

NAME	3-Et
EXPNO	2
PROCNO	1
Date_	20130127
Time	15.29
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CD2Cl2
NS	5293
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	295.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.68 usec
PL1	-0.60 dB
PL1W	41.24164963 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	15.17 dB
PL13	15.92 dB
PL2W	8.31434441 W
PL12W	0.25282964 W
PL13W	0.21272963 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127278 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

^{11}B



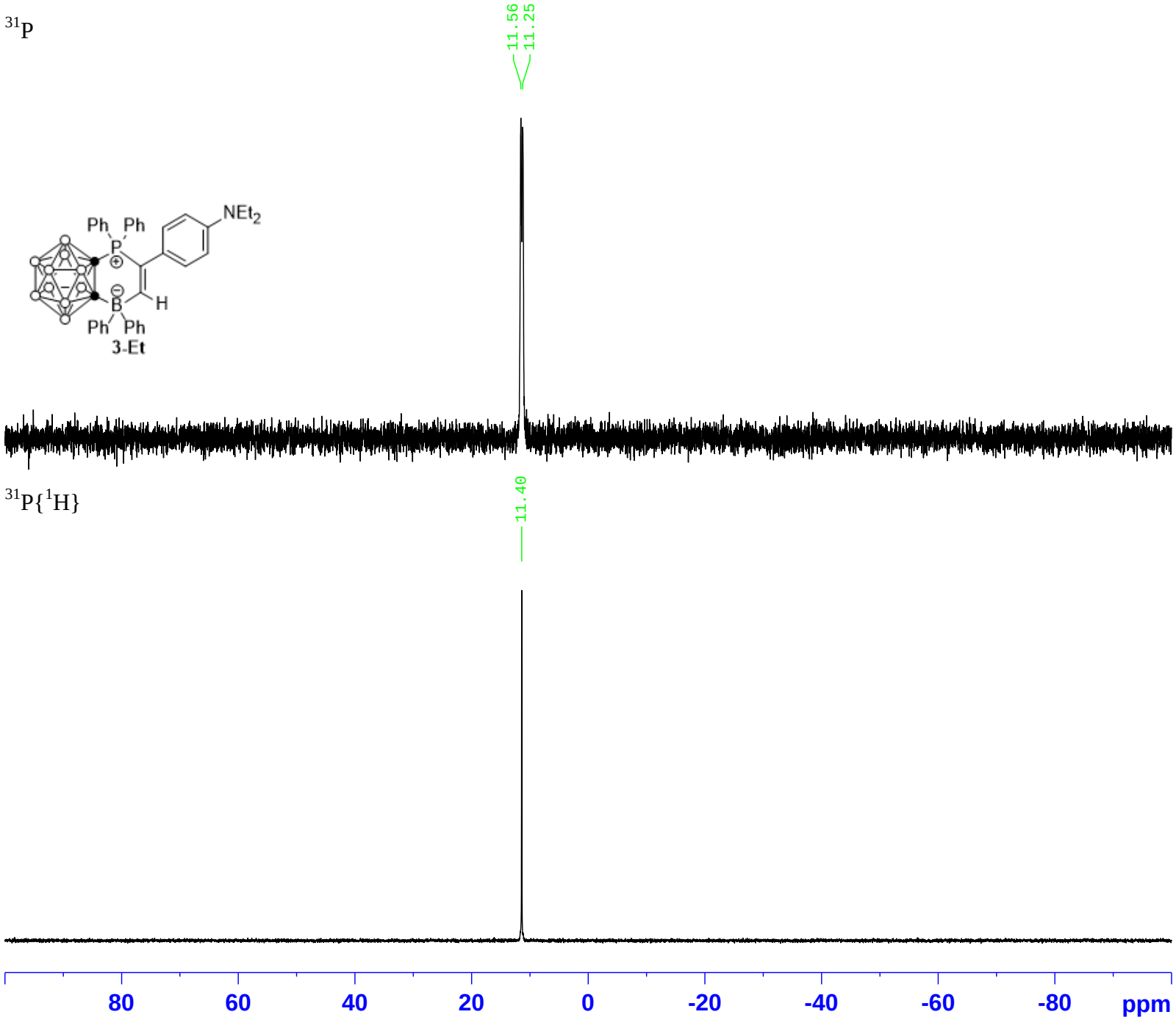
3-Et

$^{11}B\{^{1}H\}$


```
NAME                3-Et
EXPNO                  4
PROCNO                 1
Date_           20130128
Time                9.20
INSTRUM            spect
PROBHD   5 mm PABBO BB-
PULPROG             zgdc
TD                 65536
SOLVENT           CD2Cl2
NS                     8
DS                     0
SWH          25510.203 Hz
FIDRES        0.389255 Hz
AQ           1.2845556 sec
RG                   287
DW              19.600 usec
DE               6.50 usec
TE               296.8 K
D1          5.00000000 sec
D11         0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                11B
P1                 7.60 usec
PL1               -3.00 dB
PL1W       55.13059616 W
SFO1       128.3968556 MHz

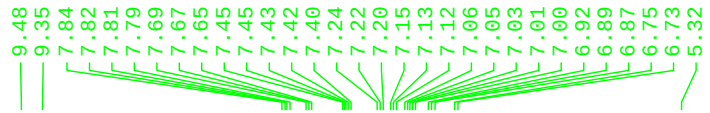
======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2            90.00 usec
PL2               -1.00 dB
PL12              15.16 dB
PL2W       13.56617069 W
PL12W       0.32844096 W
SFO2       400.1916008 MHz
SI                 32768
SF         128.3968897 MHz
WDW                EM
SSB                 0
LB               3.00 Hz
GB                  0
PC                1.40
```

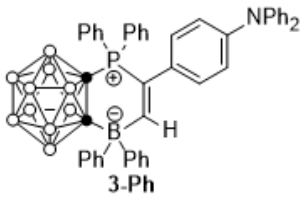


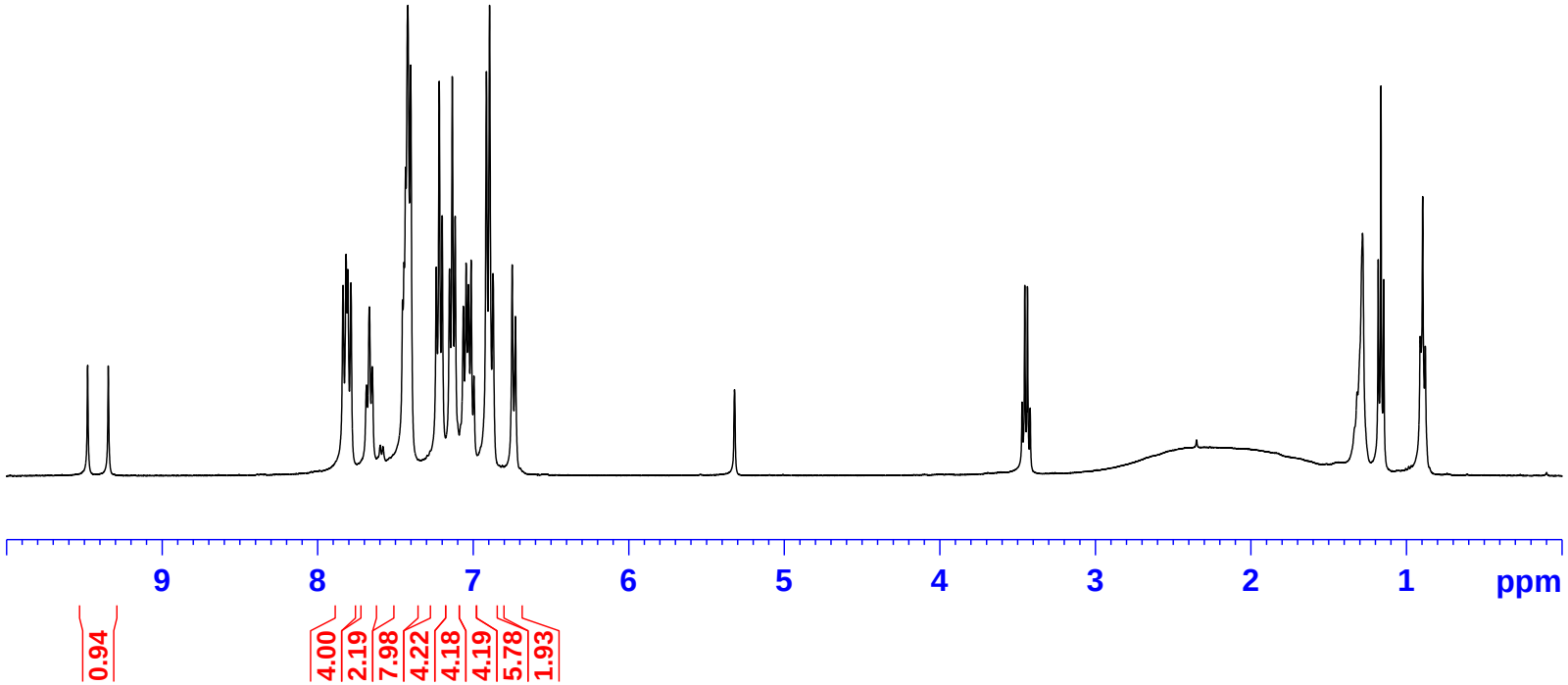

```
NAME                  3-Et
EXPNO                    6
PROCNO                   1
Date_             20130128
Time                  9.16
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG           zgdc30
TD                  65536
SOLVENT            CD2Cl2
NS                      20
DS                       0
SWH              96153.844 Hz
FIDRES            1.467191 Hz
AQ                0.3408372 sec
RG                    2050
DW                   5.200 usec
DE                   6.50 usec
TE                  297.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   31P
P1                   14.70 usec
PL1                   4.00 dB
PL1W         10.30000019 W
SFO1         161.9917814 MHz

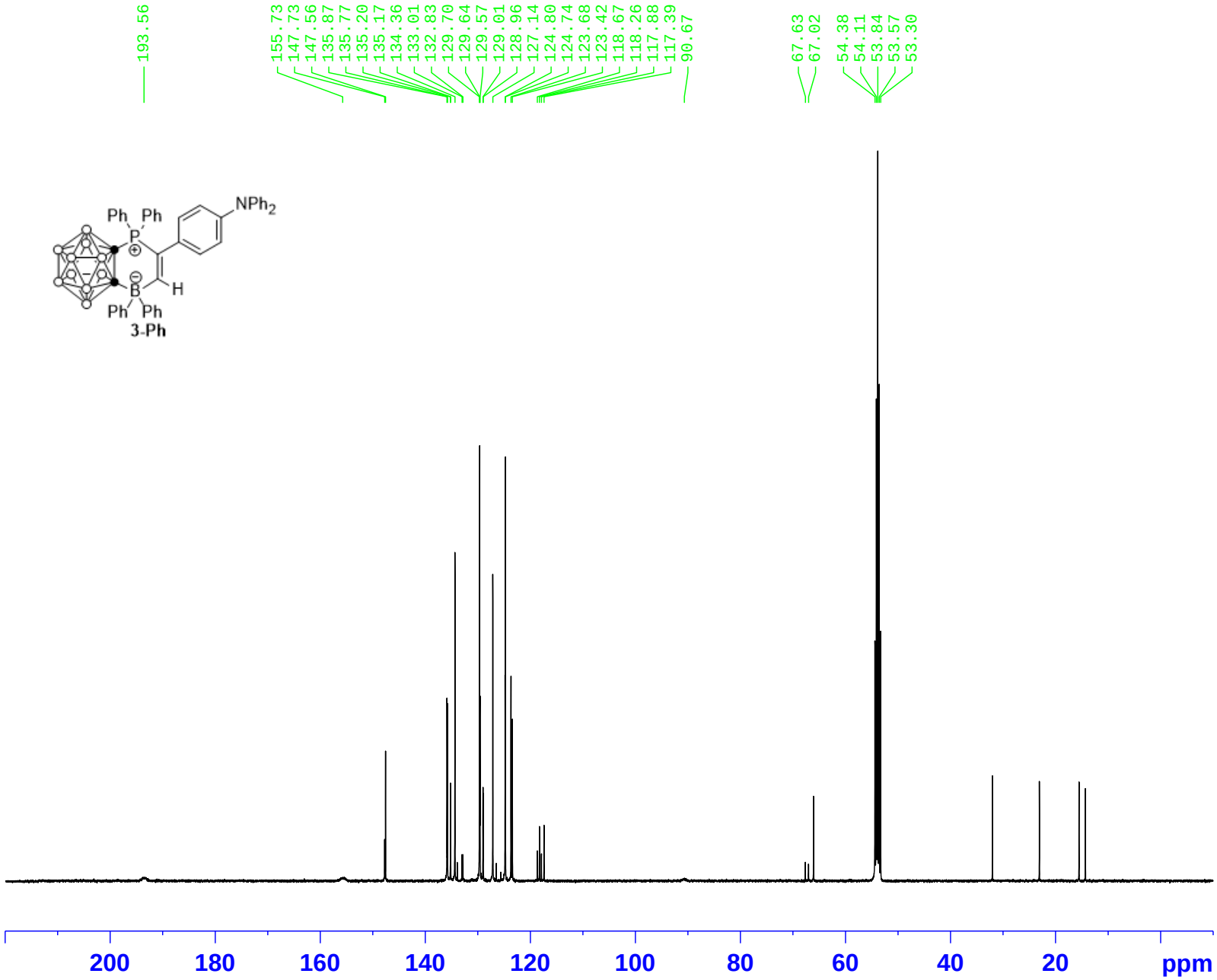
======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2                90.00 usec
PL2                  -1.00 dB
PL12                 15.16 dB
PL2W         13.56617069 W
PL12W         0.32844096 W
SFO2         400.1916008 MHz
SI                   32768
SF           161.9997813 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```







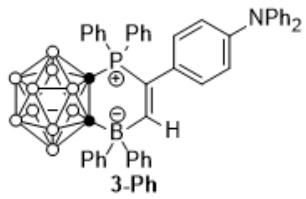
NAME	3-Ph
EXPNO	1
PROCNO	1
Date_	20121126
Time	21.38
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zg30
TD	65536
SOLVENT	CD2Cl2
NS	8
DS	2
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	50.8
DW	60.800 usec
DE	6.50 usec
TE	296.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
PL1	-1.00 dB
PL1W	13.56617069 W
SFO1	400.1924713 MHz
SI	32768
SF	400.1900197 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



BRUKER

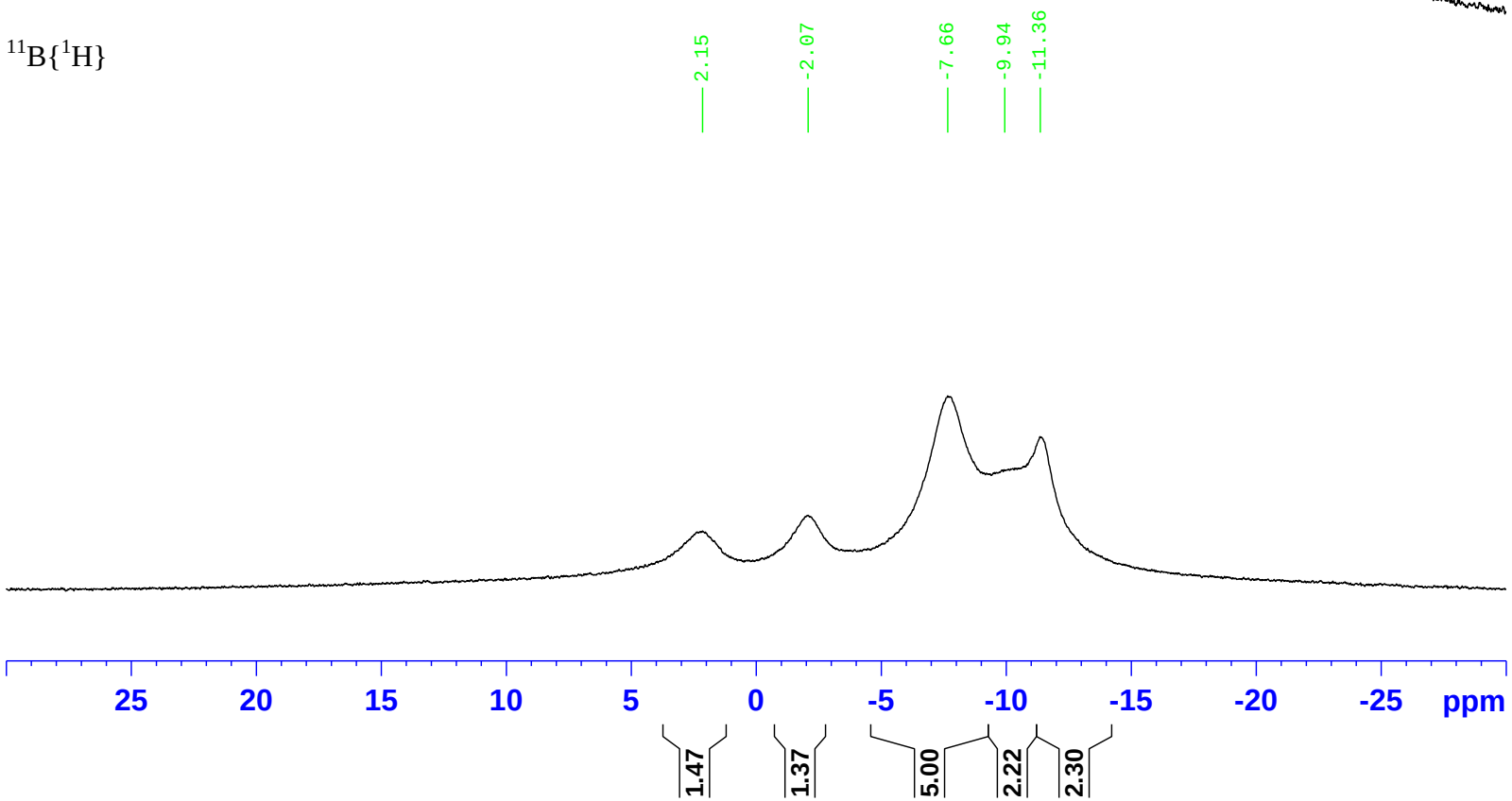
NAME	3-Ph
EXPNO	2
PROCNO	1
Date_	20121126
Time	21.41
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CD2Cl2
NS	11828
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	406
DW	20.800 usec
DE	6.50 usec
TE	296.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.90 usec
PL1	-2.00 dB
PL1W	55.33689499 W
SFO1	100.6379183 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-1.00 dB
PL12	15.16 dB
PL13	18.62 dB
PL2W	13.56617069 W
PL12W	0.32844096 W
PL13W	0.14806664 W
SFO2	400.1916008 MHz
SI	32768
SF	100.6278149 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

^{11}B



3-Ph

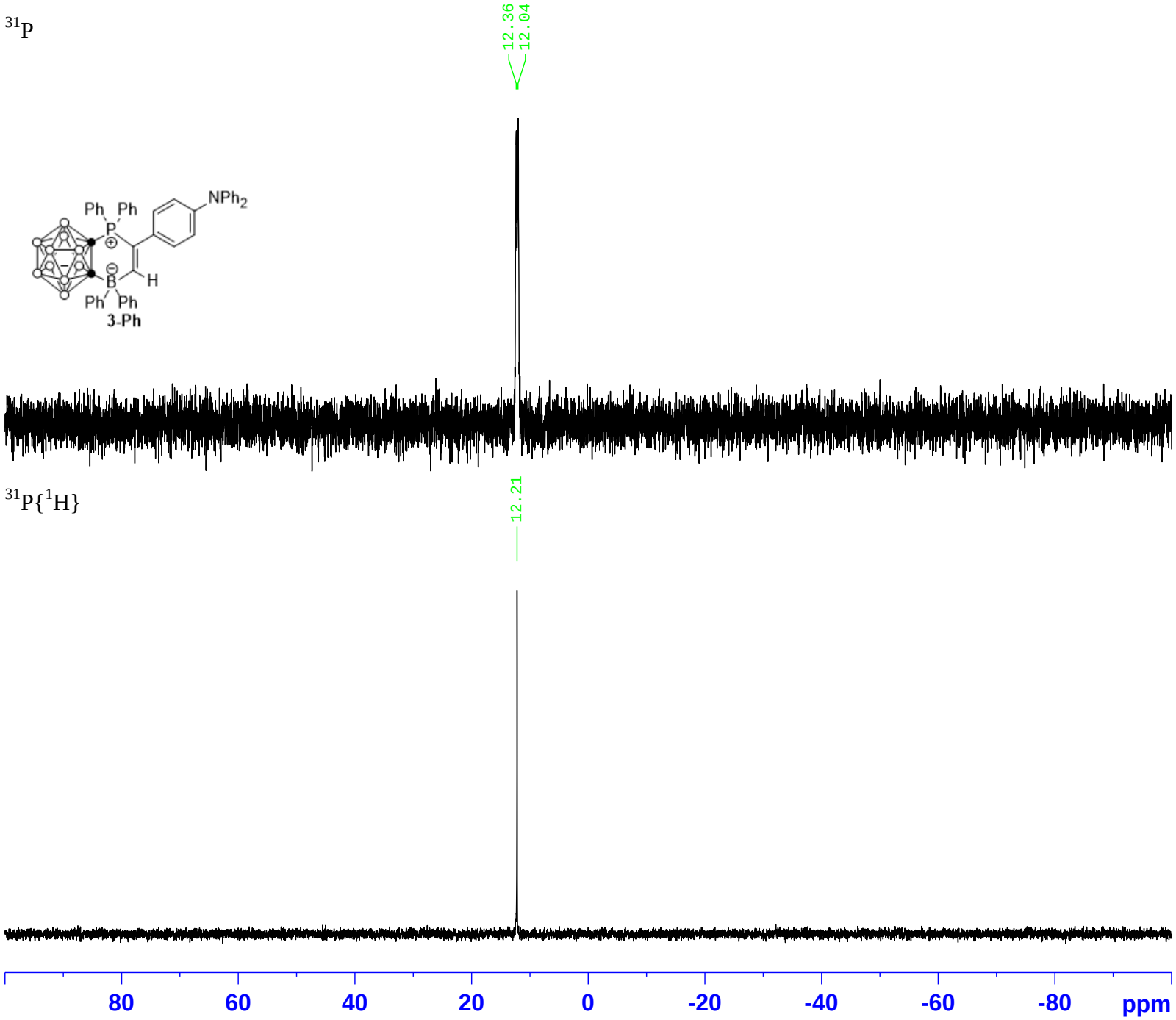
$^{11}B\{^{1}H\}$




```
NAME                  3-Ph
EXPNO                    4
PROCNO                   1
Date_             20121126
Time                 20.17
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zgdc
TD                   65536
SOLVENT           CD2Cl2
NS                       16
DS                        0
SWH              25510.203 Hz
FIDRES            0.389255 Hz
AQ                1.2845556 sec
RG                      362
DW                  19.600 usec
DE                   6.50 usec
TE                  297.0 K
D1               5.00000000 sec
D11              0.03000000 sec
TD0                      1

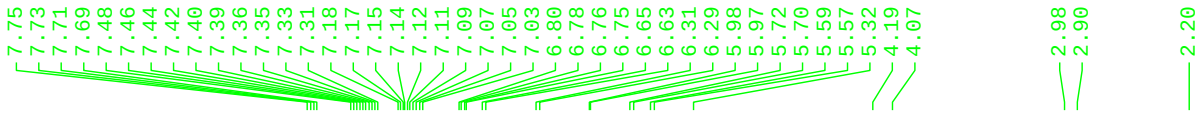
======== CHANNEL f1 ========
NUC1                   11B
P1                    7.60 usec
PL1                  -3.00 dB
PL1W          55.13059616 W
SFO1          128.3968556 MHz

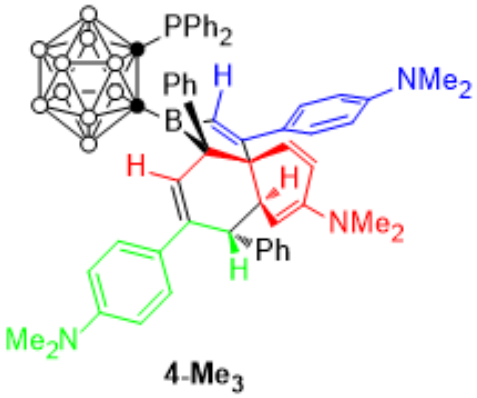
======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2                90.00 usec
PL2                  -1.00 dB
PL12                 15.16 dB
PL2W          13.56617069 W
PL12W          0.32844096 W
SFO2          400.1916008 MHz
SI                   32768
SF            128.3968897 MHz
WDW                    EM
SSB                       0
LB                    3.00 Hz
GB                        0
PC                    1.40
```



BRUKER

NAME	3-Ph
EXPNO	6
PROCNO	1
Date_	20121126
Time	20.12
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgdc30
TD	65536
SOLVENT	CD2Cl2
NS	16
DS	0
SWH	96153.844 Hz
FIDRES	1.467191 Hz
AQ	0.3408372 sec
RG	2050
DW	5.200 usec
DE	6.50 usec
TE	297.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	31P
P1	14.70 usec
PL1	4.00 dB
PL1W	10.30000019 W
SFO1	161.9917814 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-1.00 dB
PL12	15.16 dB
PL2W	13.56617069 W
PL12W	0.32844096 W
SFO2	400.1916008 MHz
SI	32768
SF	161.9997813 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

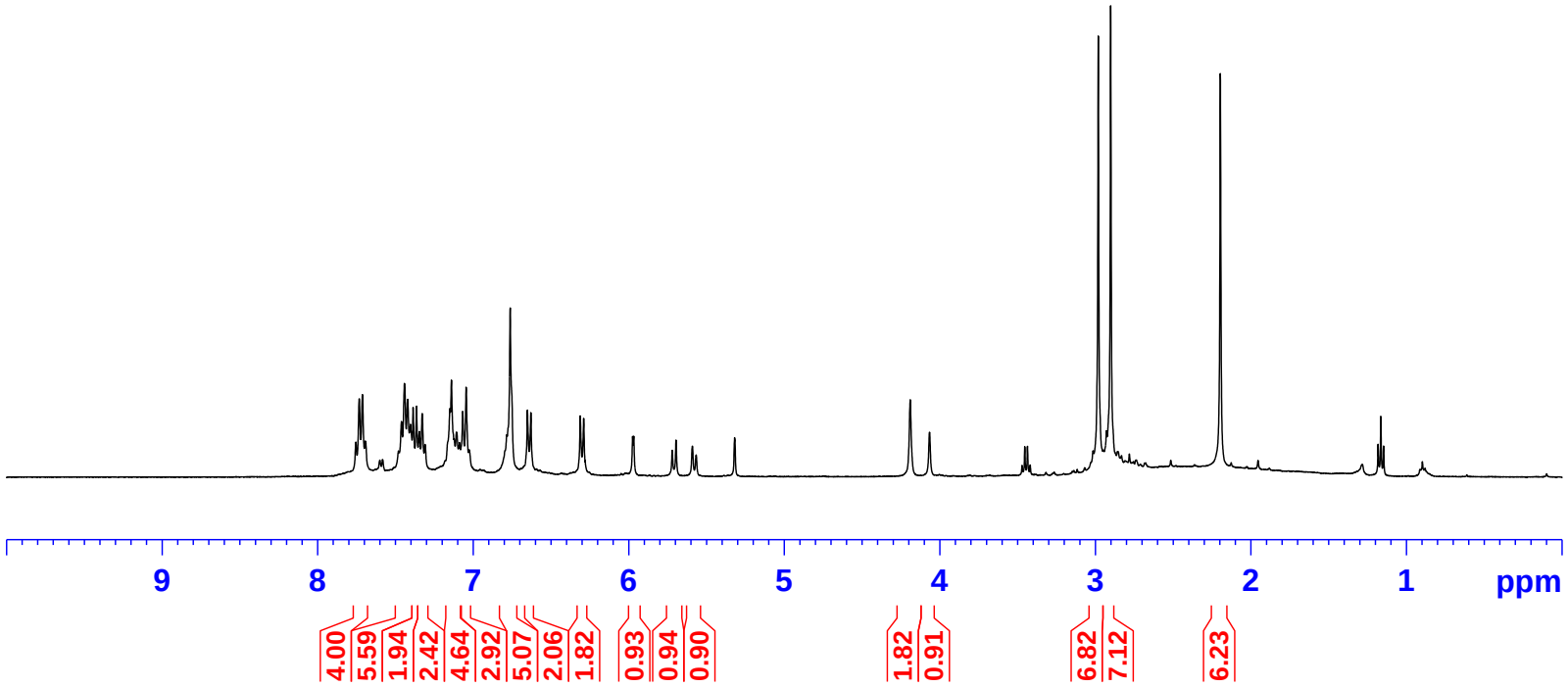


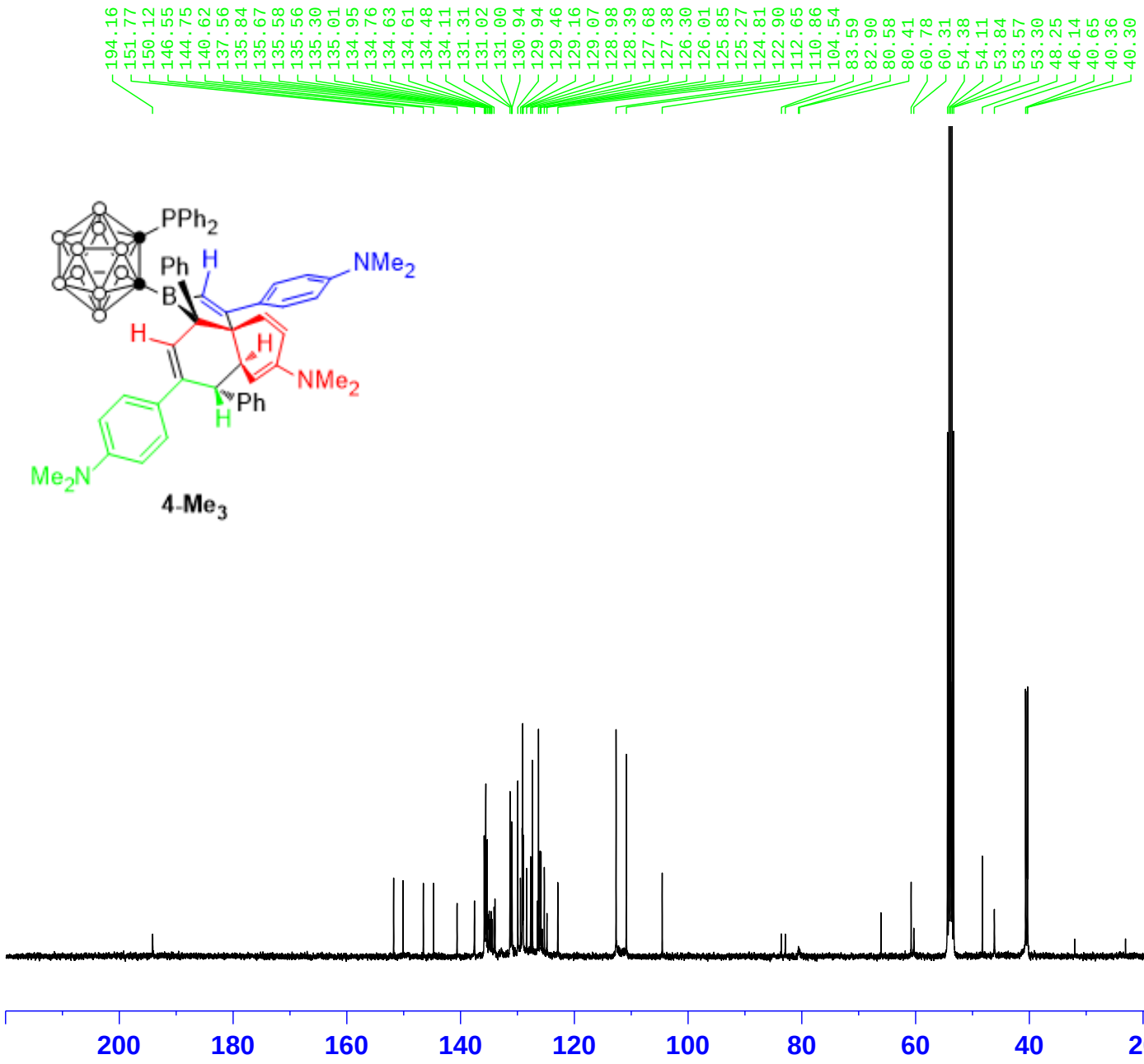


4-Me$_3$

```
NAME                 4-Me3
EXPNO                   1
PROCNO                  1
Date_             20120602
Time                20.32
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG              zg30
TD                  65536
SOLVENT           CD2Cl2
NS                      8
DS                      2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                     57
DW                 60.800 usec
DE                  6.50 usec
TE                 299.3 K
D1              1.00000000 sec
TD0                     1

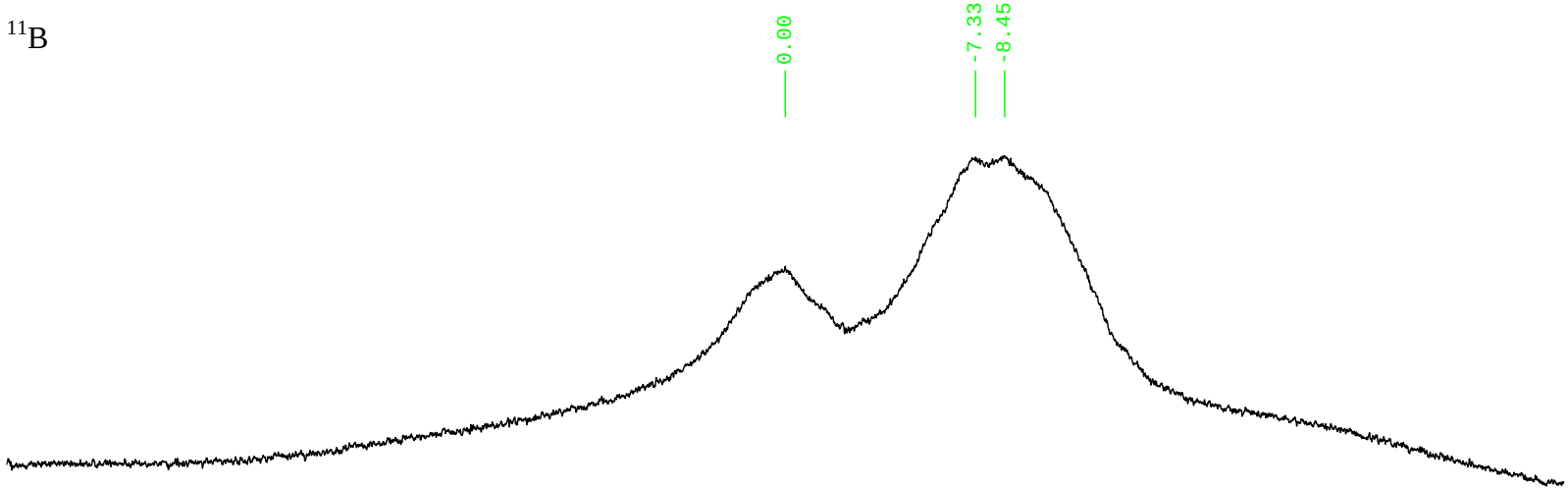
======== CHANNEL f1 ========
NUC1                   1H
P1                 14.00 usec
PL1                -1.00 dB
PL1W        13.56617069 W
SFO1        400.1924713 MHz
SI                  32768
SF          400.1900198 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                 1.00
```



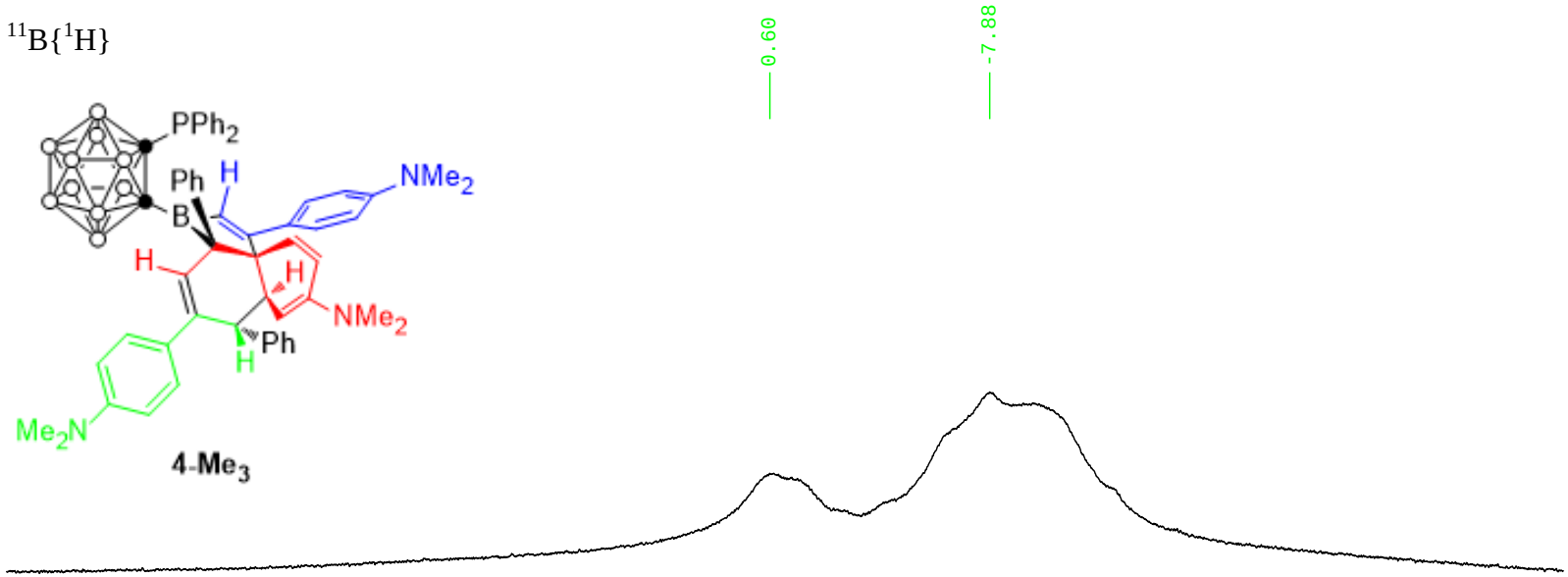


NAME	4-Me3
EXPNO	2
PROCNO	1
Date_	20120603
Time	9.18
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CD2Cl2
NS	8036
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	645
DW	20.800 usec
DE	6.50 usec
TE	299.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.90 usec
PL1	-2.00 dB
PL1W	55.33689499 W
SFO1	100.6379183 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-1.00 dB
PL12	15.16 dB
PL13	18.62 dB
PL2W	13.56617069 W
PL12W	0.32844096 W
PL13W	0.14806664 W
SFO2	400.1916008 MHz
SI	32768
SF	100.6278124 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

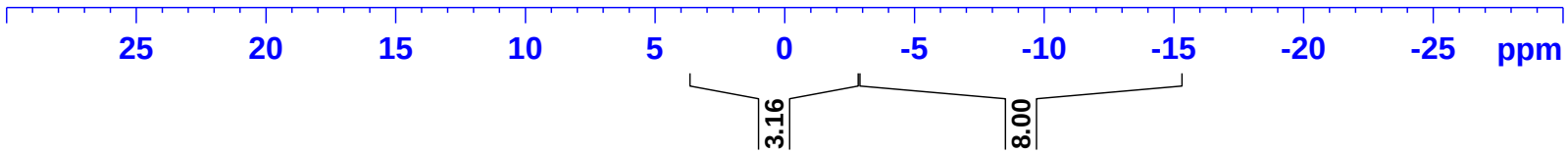
^{11}B



$^{11}B\{^{1}H\}$



4-Me$_3$

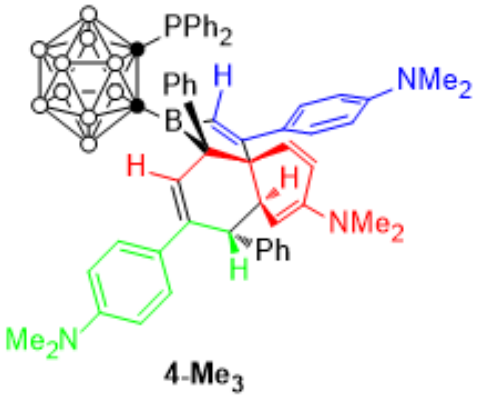



```
NAME                 4-Me3
EXPNO                   11
PROCNO                   1
Date_             20120602
Time                 20.37
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zgdc
TD                   65536
SOLVENT             CD2Cl2
NS                       32
DS                        0
SWH              25510.203 Hz
FIDRES            0.389255 Hz
AQ               1.2845556 sec
RG                     362
DW                19.600 usec
DE                 6.50 usec
TE                 299.4 K
D1             5.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   11B
P1                   7.60 usec
PL1                 -3.00 dB
PL1W          55.13059616 W
SFO1         128.3968556 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -1.00 dB
PL12                15.16 dB
PL2W          13.56617069 W
PL12W          0.32844096 W
SFO2         400.1916008 MHz
SI                   32768
SF           128.3968897 MHz
WDW                    EM
SSB                     0
LB                   3.00 Hz
GB                      0
PC                   1.40
```

^{31}P



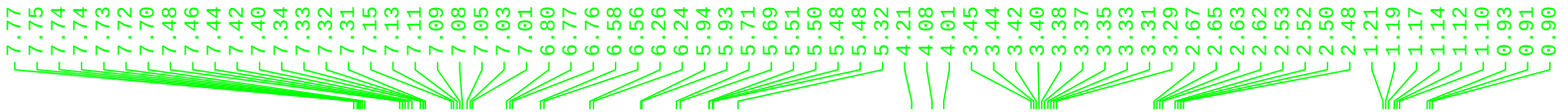
4-Me$_3$

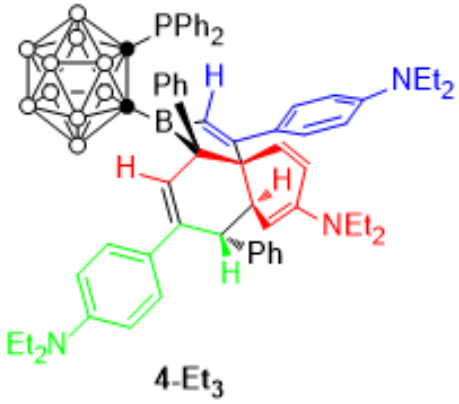
$^{31}P\{^{1}H\}$


```
NAME                 4-Me3
EXPNO                   13
PROCNO                   1
Date_             20120602
Time                 20.35
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG           zgdc30
TD                  65536
SOLVENT            CD2Cl2
NS                       8
DS                       0
SWH              64102.563 Hz
FIDRES           0.978127 Hz
AQ               0.5112308 sec
RG                    2050
DW                 7.800 usec
DE                 6.50 usec
TE                 299.5 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   31P
P1                  14.70 usec
PL1                  4.00 dB
PL1W         10.30000019 W
SFO1         161.9917814 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               90.00 usec
PL2                 -1.00 dB
PL12                15.16 dB
PL2W         13.56617069 W
PL12W         0.32844096 W
SFO2         400.1916008 MHz
SI                  32768
SF           161.9997813 MHz
WDW                    EM
SSB                     0
LB                  1.00 Hz
GB                      0
PC                  1.40
```

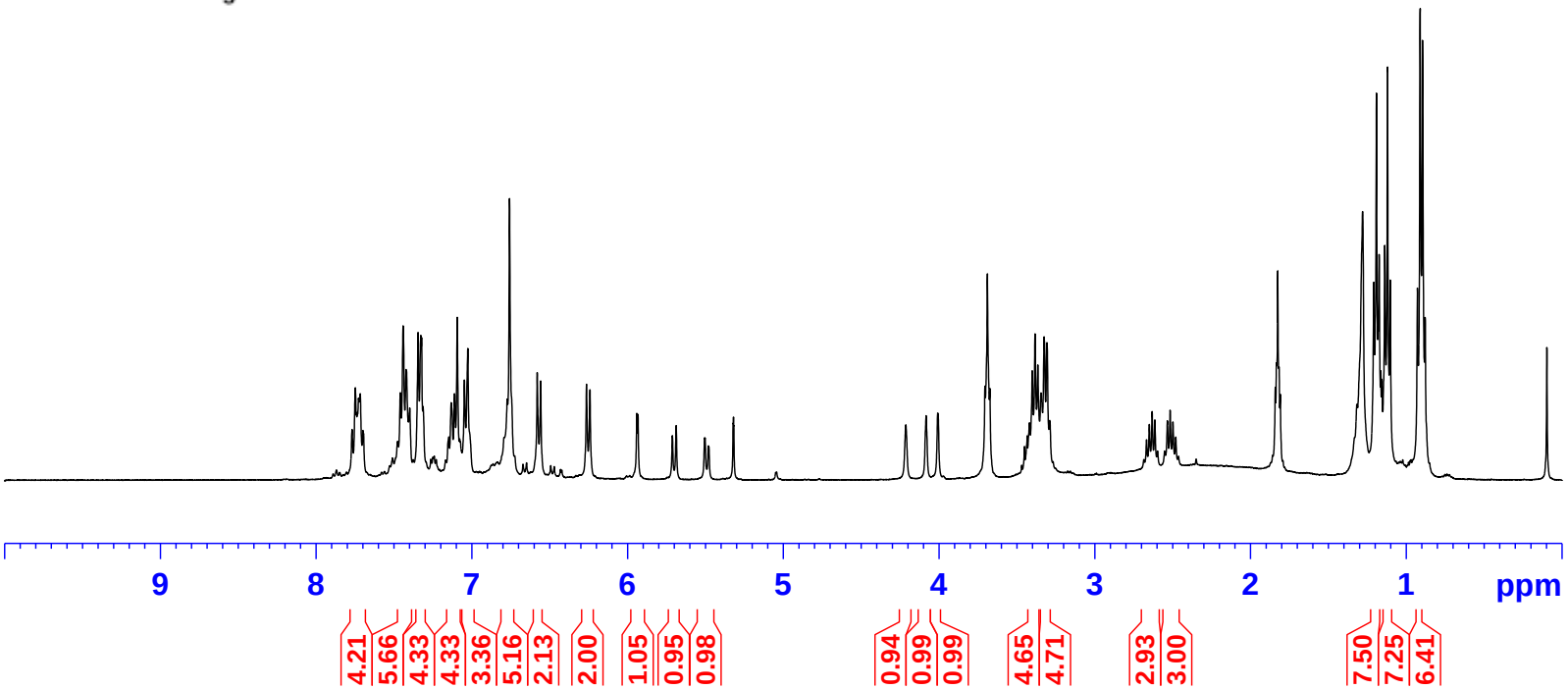


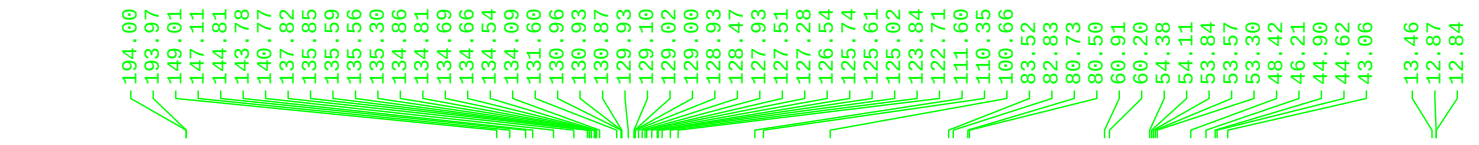


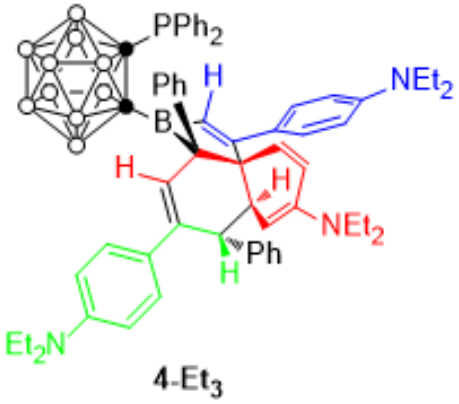
4-Et$_3$

```
NAME                  4-Et3
EXPNO                     1
PROCNO                    1
Date_              20130205
Time                 10.12
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT             CD2Cl2
NS                        8
DS                        2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                     40.3
DW                   60.800 usec
DE                    6.50 usec
TE                   295.3 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    14.00 usec
PL1                   -1.00 dB
PL1W          13.56617069 W
SFO1          400.1924713 MHz
SI                    32768
SF            400.1900197 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.00
```



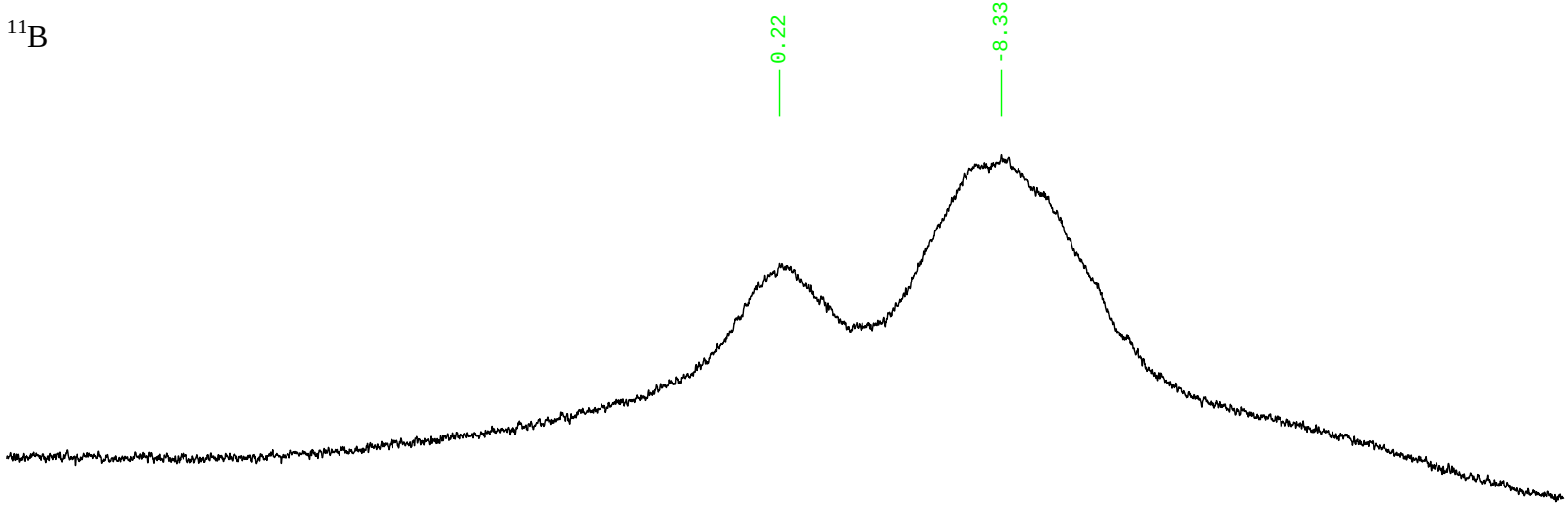




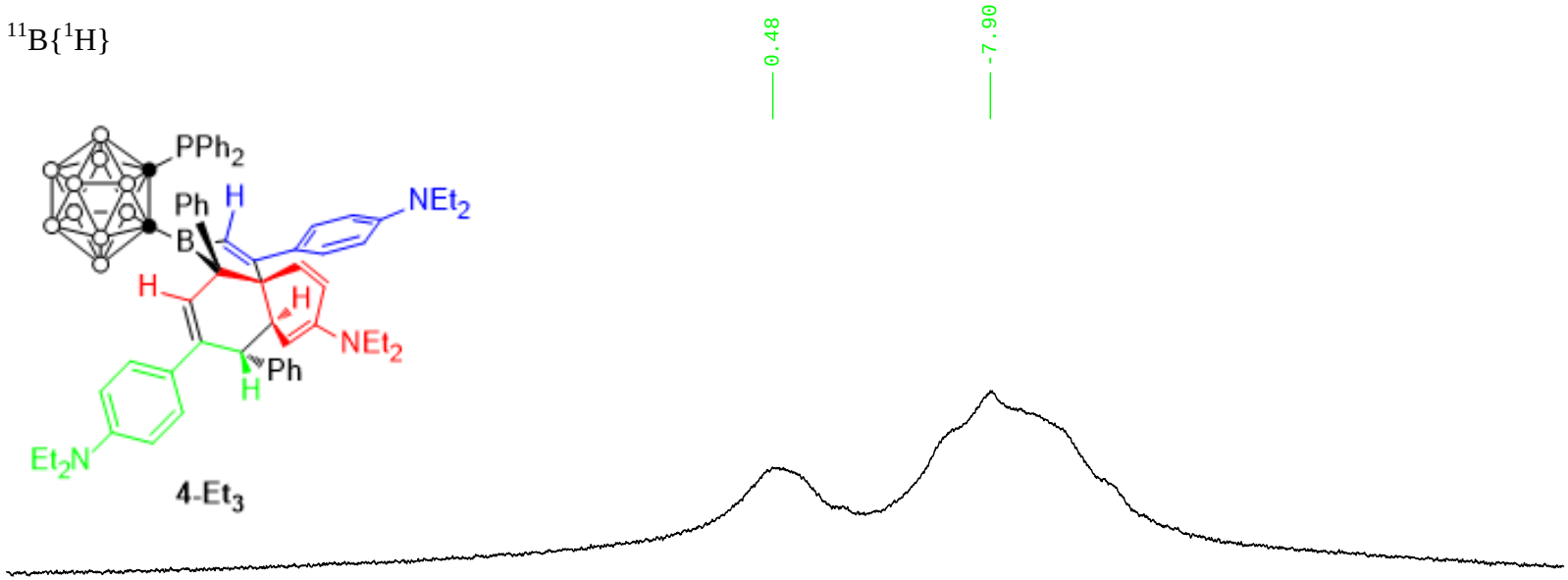
4-Et$_3$

NAME	4-Et3
EXPNO	2
PROCNO	1
Date_	20130207
Time	3.08
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CD2Cl2
NS	15216
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	228
DW	20.800 usec
DE	6.50 usec
TE	296.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.90 usec
PL1	-2.00 dB
PL1W	55.33689499 W
SFO1	100.6379183 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-1.00 dB
PL12	15.16 dB
PL13	18.62 dB
PL2W	13.56617069 W
PL12W	0.32844096 W
PL13W	0.14806664 W
SFO2	400.1916008 MHz
SI	32768
SF	100.6278145 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

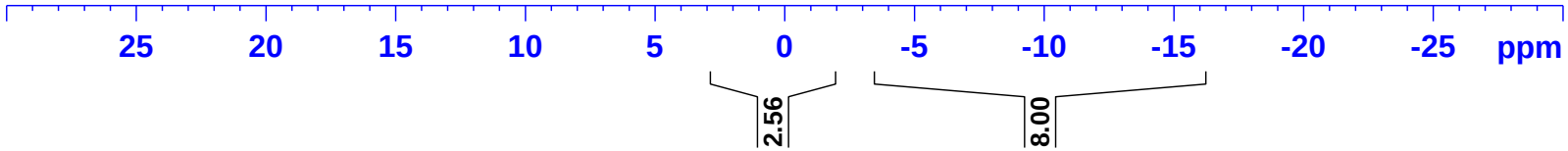
^{11}B



$^{11}B\{^{1}H\}$



4-Et$_3$

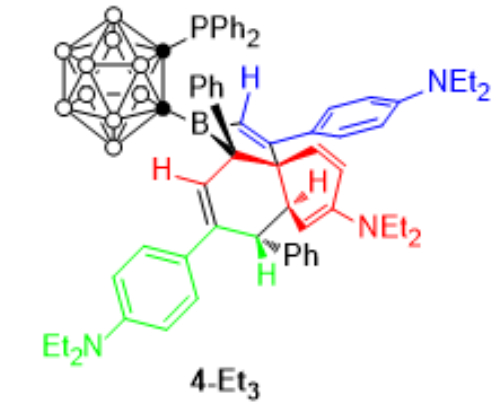



```
NAME                 4-Et3
EXPNO                    4
PROCNO                   1
Date_             20130205
Time                 10.18
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zgdc
TD                   65536
SOLVENT             CD2Cl2
NS                       16
DS                        0
SWH              25510.203 Hz
FIDRES            0.389255 Hz
AQ               1.2845556 sec
RG                      362
DW                19.600 usec
DE                 6.50 usec
TE                 295.7 K
D1             5.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   11B
P1                  7.60 usec
PL1                -3.00 dB
PL1W         55.13059616 W
SFO1        128.3968556 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              90.00 usec
PL2                -1.00 dB
PL12               15.16 dB
PL2W         13.56617069 W
PL12W         0.32844096 W
SFO2        400.1916008 MHz
SI                   32768
SF          128.3968897 MHz
WDW                    EM
SSB                     0
LB                 3.00 Hz
GB                      0
PC                 1.40
```

^{31}P



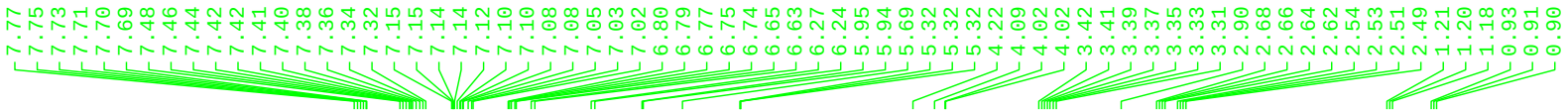
4-Et3

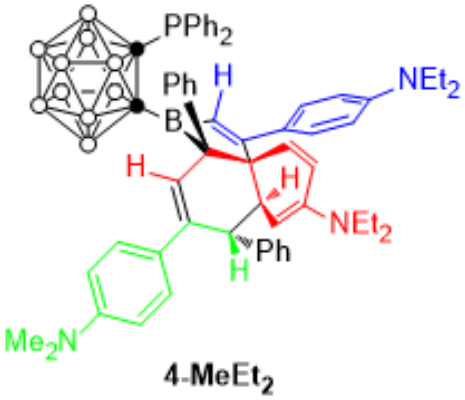
$^{31}P\{^{1}H\}$


```
NAME                  4-Et3
EXPNO                     6
PROCNO                    1
Date_              20130205
Time                 10.14
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG             zgdc30
TD                    65536
SOLVENT             CD2Cl2
NS                        16
DS                         0
SWH               96153.844 Hz
FIDRES             1.467191 Hz
AQ                 0.3408372 sec
RG                     2050
DW                    5.200 usec
DE                    6.50 usec
TE                   295.4 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     31P
P1                    14.70 usec
PL1                    4.00 dB
PL1W          10.30000019 W
SFO1          161.9917814 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                     1H
PCPD2                90.00 usec
PL2                  -1.00 dB
PL12                 15.16 dB
PL2W          13.56617069 W
PL12W          0.32844096 W
SFO2          400.1916008 MHz
SI                    32768
SF            161.9997813 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                    1.40
```

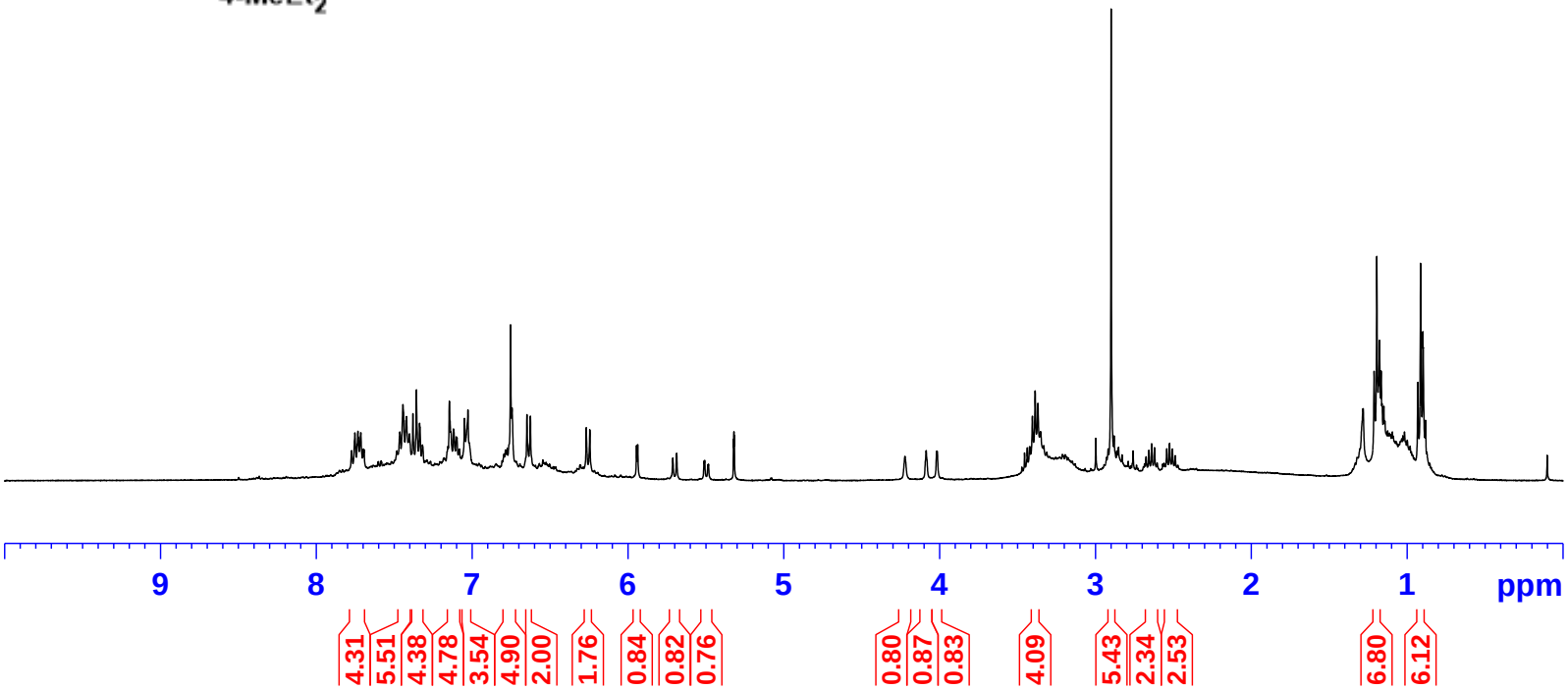


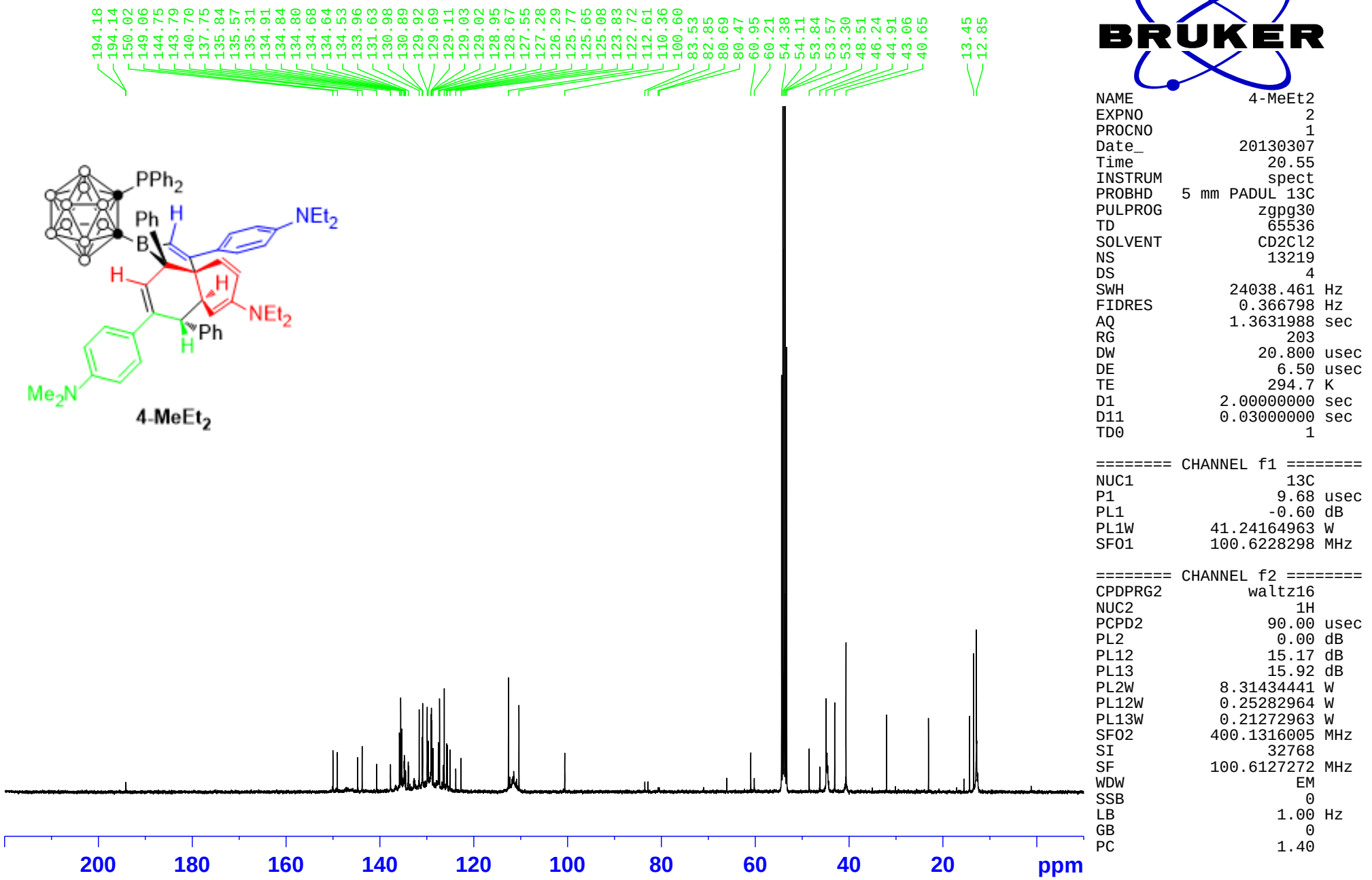


4-MeEt$_2$

```
NAME                 4-MeEt2
EXPNO                      1
PROCNO                     1
Date_               20130310
Time                   15.41
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                zg30
TD                    65536
SOLVENT            CD2Cl2
NS                         8
DS                         2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                      90.5
DW                    60.800 usec
DE                     6.50 usec
TE                    295.1 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     15.69 usec
PL1                     0.00 dB
PL1W            8.31434441 W
SFO1           400.1324710 MHz
SI                     32768
SF             400.1300154 MHz
WDW                       EM
SSB                        0
LB                     0.30 Hz
GB                         0
PC                      1.00
```



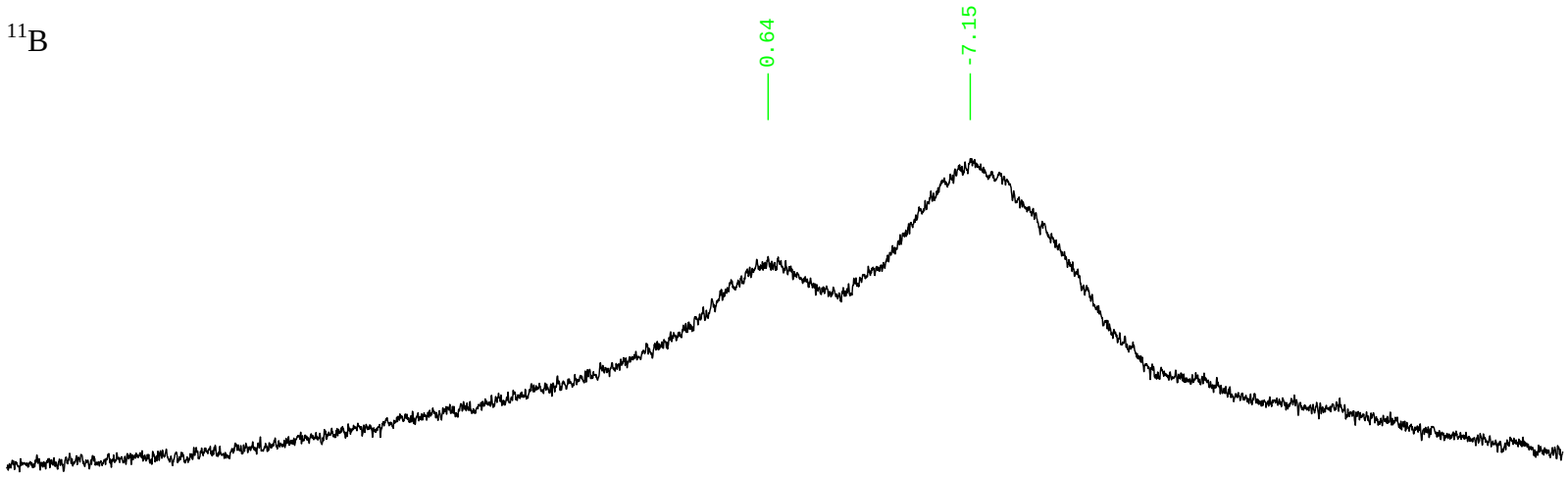



```
NAME                 4-MeEt2
EXPNO                      2
PROCNO                     1
Date_               20130307
Time                   20.55
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                zgpg30
TD                      65536
SOLVENT                CD2Cl2
NS                      13219
DS                          4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631988 sec
RG                        203
DW                     20.800 usec
DE                      6.50 usec
TE                     294.7 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

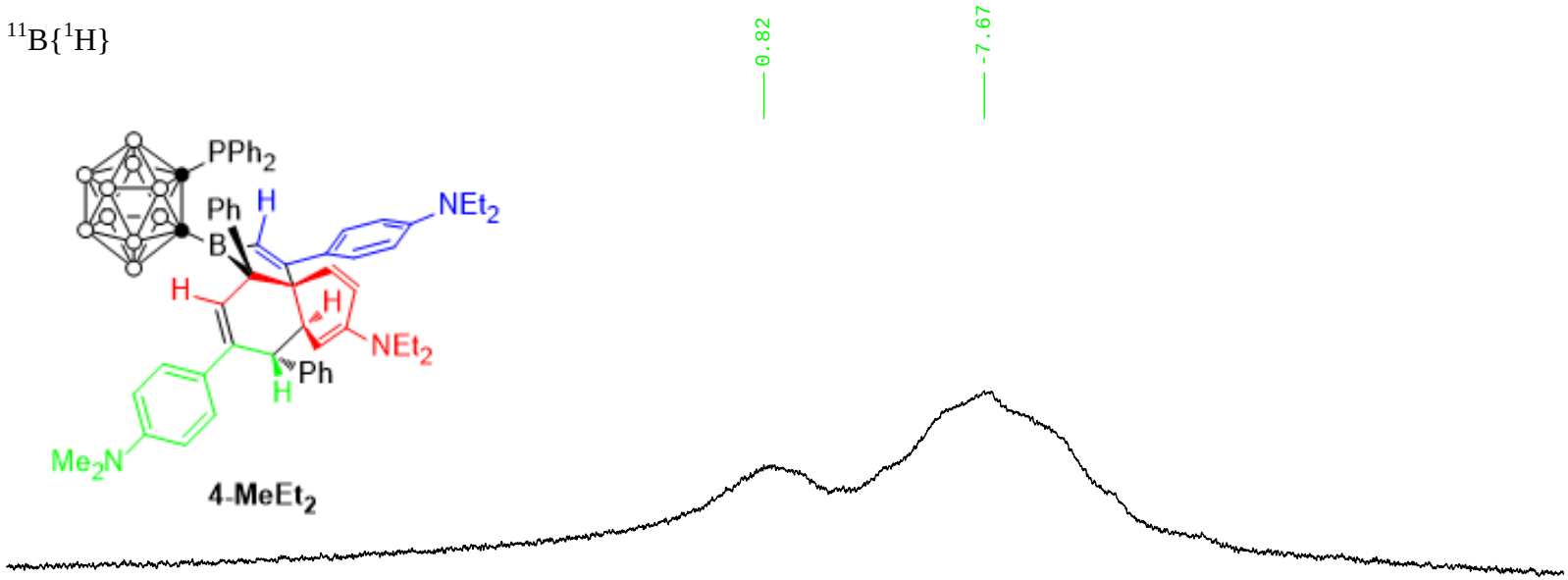
======== CHANNEL f1 ========
NUC1                      13C
P1                       9.68 usec
PL1                     -0.60 dB
PL1W              41.24164963 W
SFO1              100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   90.00 usec
PL2                      0.00 dB
PL12                    15.17 dB
PL13                    15.92 dB
PL2W               8.31434441 W
PL12W              0.25282964 W
PL13W              0.21272963 W
SFO2              400.1316005 MHz
SI                       32768
SF                100.6127272 MHz
WDW                         EM
SSB                          0
LB                      1.00 Hz
GB                           0
PC                      1.40
```

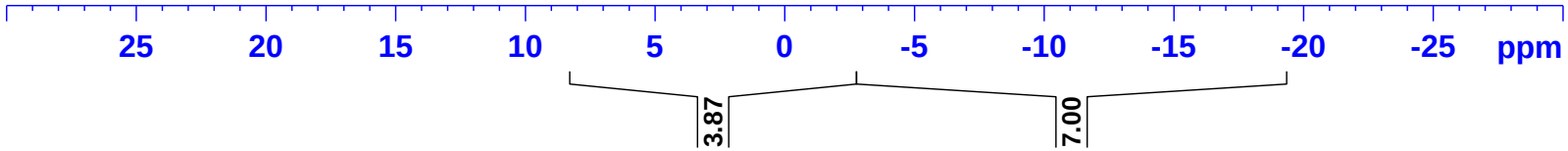
^{11}B



$^{11}B\{^{1}H\}$



4-MeEt$_2$

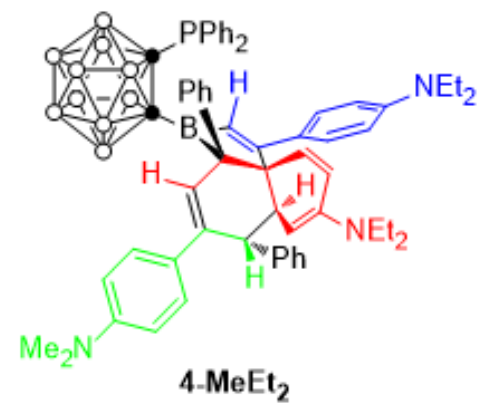



```
NAME                 4-MeEt2
EXPNO                      4
PROCNO                     1
Date_               20130308
Time                   17.03
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zgdc
TD                     65536
SOLVENT             CD2Cl2
NS                         8
DS                         0
SWH               25510.203 Hz
FIDRES             0.389255 Hz
AQ                1.2845556 sec
RG                       362
DW                  19.600 usec
DE                   6.50 usec
TE                   295.6 K
D1               5.00000000 sec
D11              0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     11B
P1                    7.60 usec
PL1                  -3.00 dB
PL1W          55.13059616 W
SFO1         128.3968556 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                90.00 usec
PL2                  -1.00 dB
PL12                 15.16 dB
PL2W          13.56617069 W
PL12W          0.32844096 W
SFO2         400.1916008 MHz
SI                   32768
SF           128.3968897 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.40
```

^{31}P



4-MeEt$_2$

17.53

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

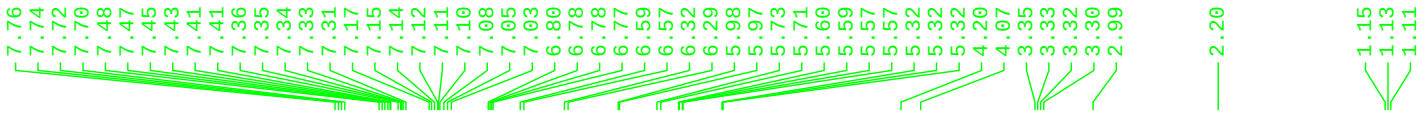
17.52

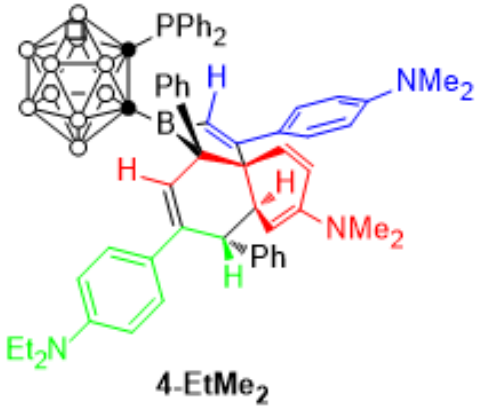
80 60 40 20 0 -20 -40 -60 -80 ppm

```
NAME                 4-MeEt2
EXPNO                      6
PROCNO                     1
Date_               20130308
Time                   17.00
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zgdc30
TD                      65536
SOLVENT                CD2Cl2
NS                         12
DS                          0
SWH                 96153.844 Hz
FIDRES               1.467191 Hz
AQ                   0.3408372 sec
RG                        2050
DW                       5.200 usec
DE                       6.50 usec
TE                      295.2 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      31P
P1                      14.70 usec
PL1                      4.00 dB
PL1W             10.30000019 W
SFO1             161.9917814 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   90.00 usec
PL2                     -1.00 dB
PL12                    15.16 dB
PL2W             13.56617069 W
PL12W             0.32844096 W
SFO2             400.1916008 MHz
SI                      32768
SF               161.9997813 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

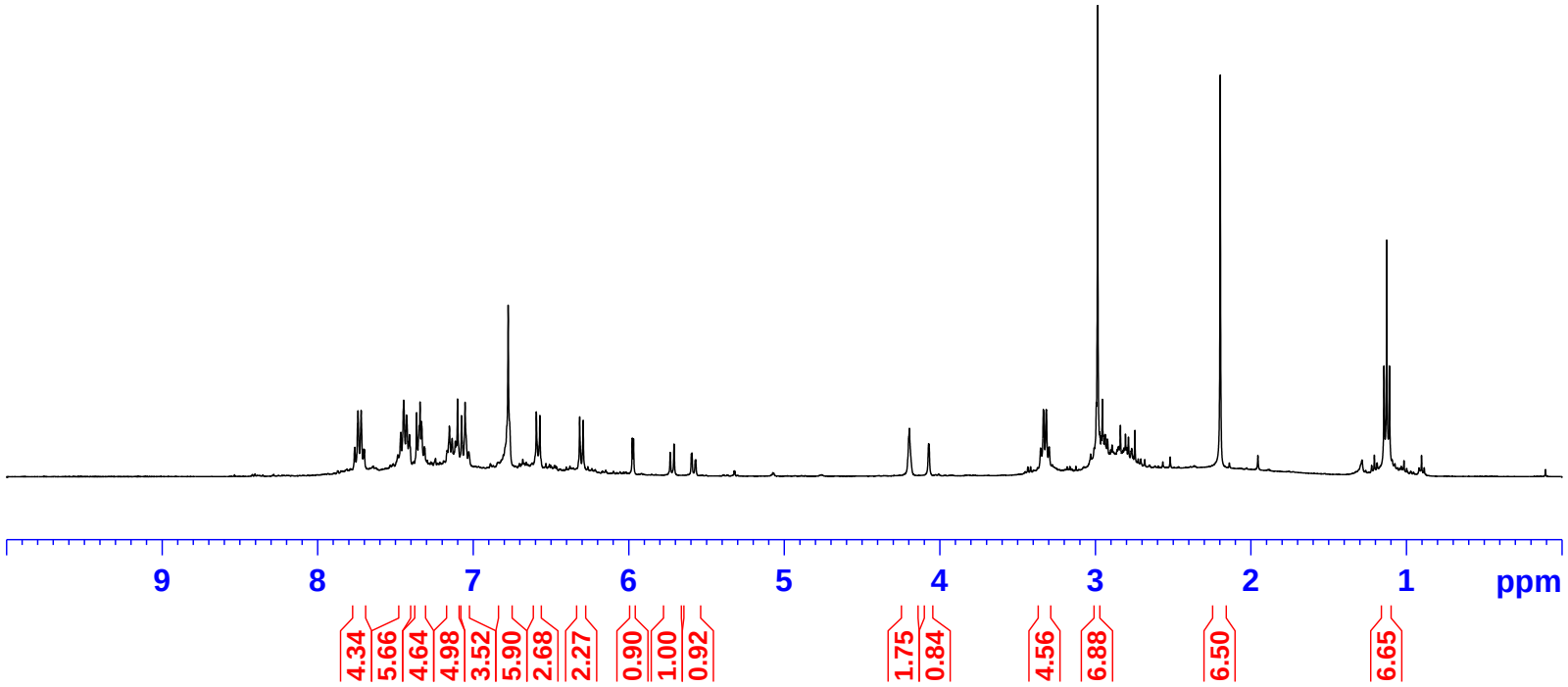


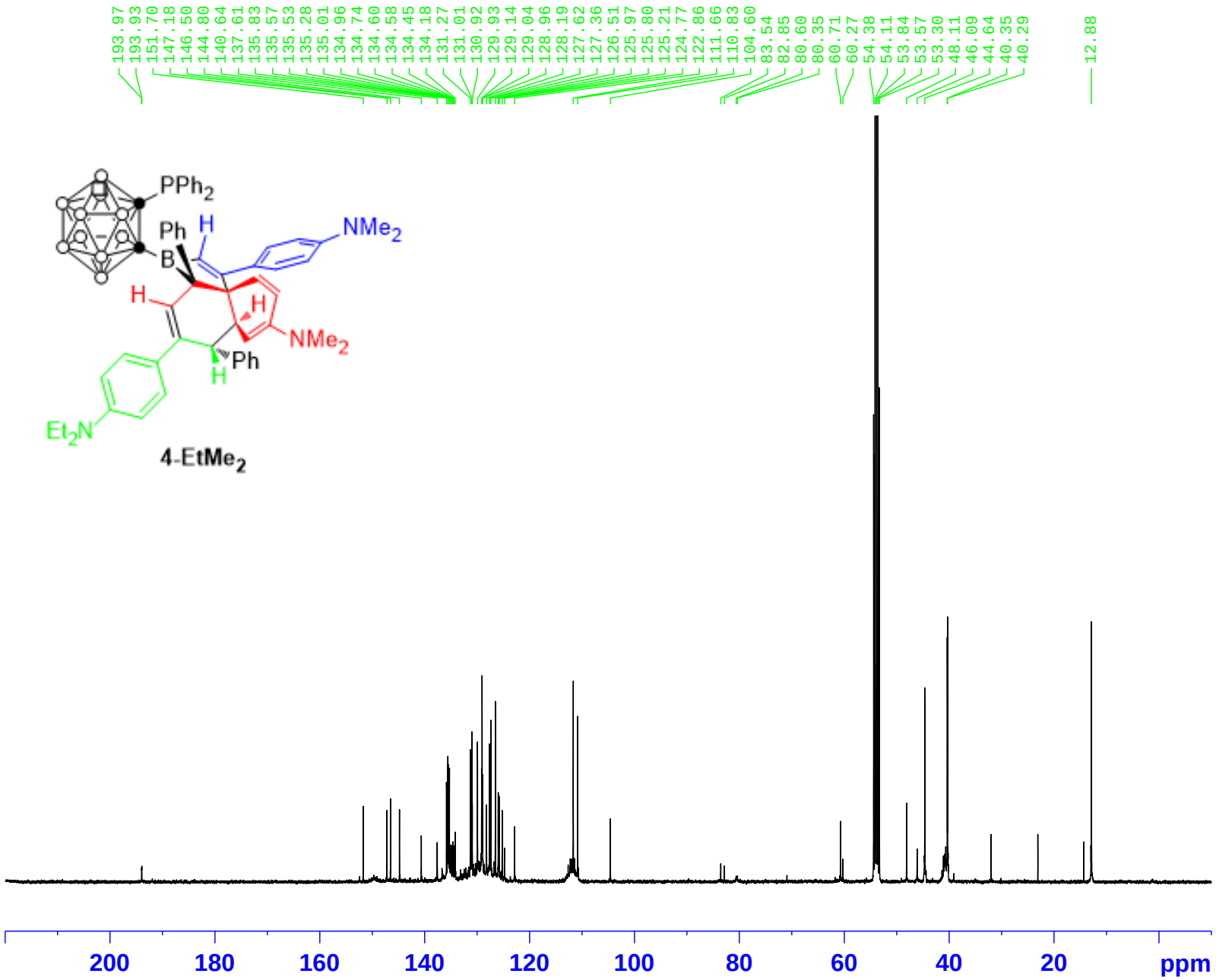


4-EtMe$_2$

```
NAME                 4-EtMe2
EXPNO                      1
PROCNO                     1
Date_               20130303
Time                   15.36
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                   zg
TD                     65536
SOLVENT             CD2Cl2
NS                         10
DS                          0
SWH              10000.000 Hz
FIDRES               0.152588 Hz
AQ                3.2768500 sec
RG                      45.2
DW                    50.000 usec
DE                     6.50 usec
TE                    295.2 K
D1              1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     15.69 usec
PL1                     0.00 dB
PL1W             8.31434441 W
SFO1           400.1318000 MHz
SI                     65536
SF             400.1300155 MHz
WDW                       EM
SSB                        0
LB                     0.30 Hz
GB                         0
PC                     1.00
```

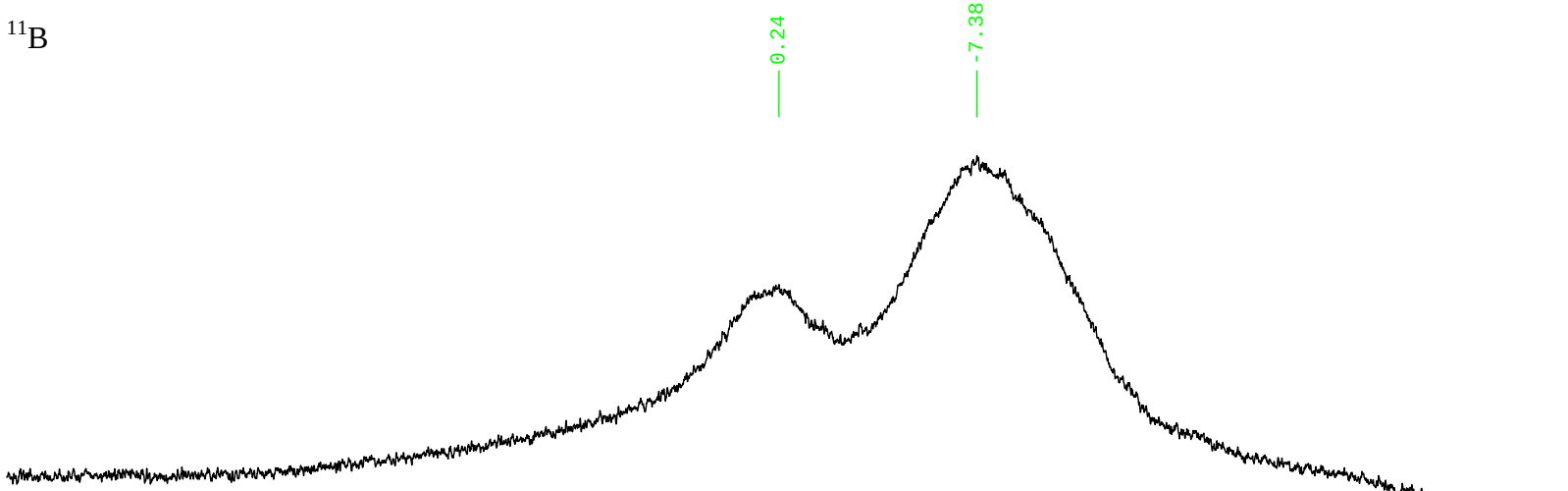




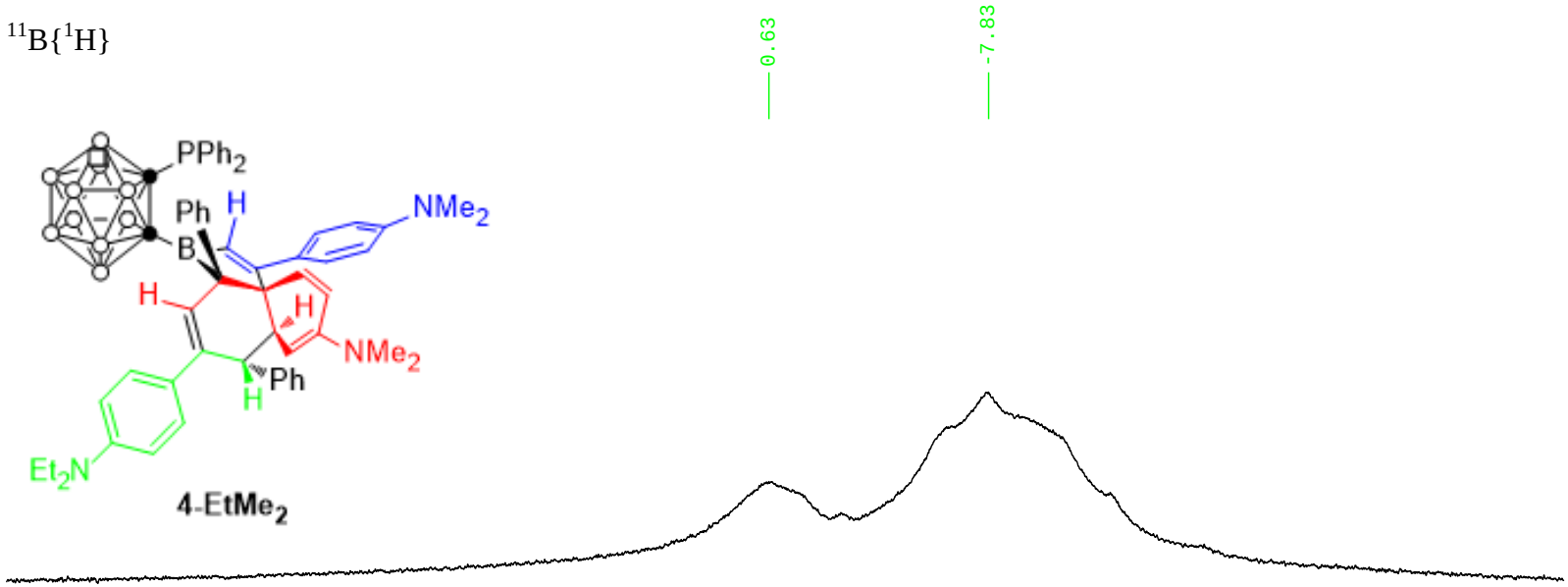
BRUKER

NAME	4-EtMe2
EXPNO	2
PROCNO	1
Date_	20130303
Time	15.42
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CD2Cl2
NS	18624
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	295.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.68 usec
PL1	-0.60 dB
PL1W	41.24164963 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	15.17 dB
PL13	15.92 dB
PL2W	8.31434441 W
PL12W	0.25282964 W
PL13W	0.21272963 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127278 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

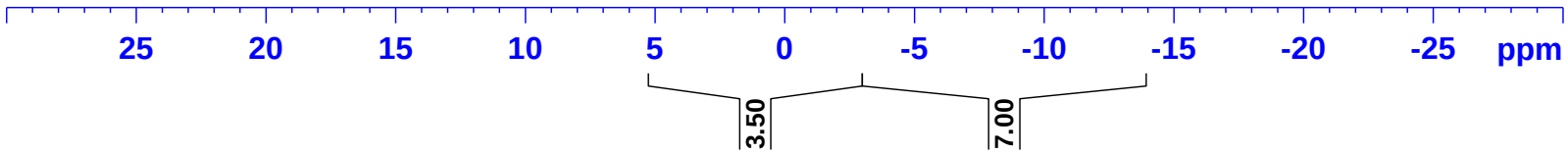
^{11}B



$^{11}B\{^{1}H\}$



4-EtMe$_2$

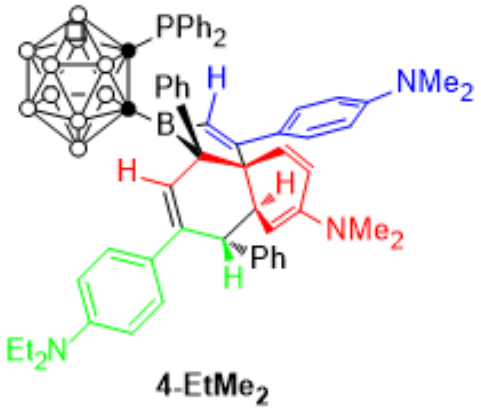



```
NAME                 4-EtMe2
EXPNO                      4
PROCNO                     1
Date_               20130304
Time                    9.37
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgdc
TD                     65536
SOLVENT             CD2Cl2
NS                         12
DS                          0
SWH                25510.203 Hz
FIDRES              0.389255 Hz
AQ                  1.2845556 sec
RG                        322
DW                   19.600 usec
DE                    6.50 usec
TE                    307.2 K
D1                 5.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      11B
P1                     7.60 usec
PL1                   -3.00 dB
PL1W            55.13059616 W
SFO1           128.3968556 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                      1H
PCPD2                 90.00 usec
PL2                   -1.00 dB
PL12                  15.16 dB
PL2W            13.56617069 W
PL12W            0.32844096 W
SFO2           400.1916008 MHz
SI                     32768
SF             128.3968897 MHz
WDW                       EM
SSB                        0
LB                    3.00 Hz
GB                         0
PC                    1.40
```

^{31}P



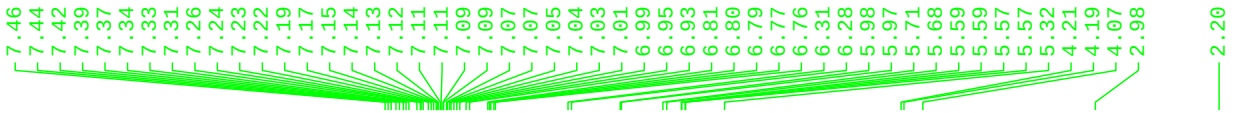
4-EtMe$_2$

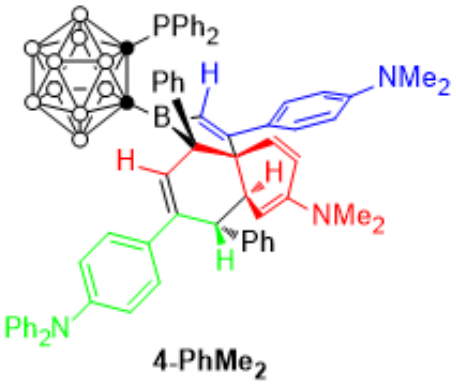
$^{31}P\{^{1}H\}$


```
NAME                 4-EtMe2
EXPNO                      6
PROCNO                     1
Date_               20130304
Time                    9.41
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zgdc30
TD                      65536
SOLVENT                CD2Cl2
NS                         8
DS                         0
SWH                 96153.844 Hz
FIDRES               1.467191 Hz
AQ                   0.3408372 sec
RG                        2050
DW                       5.200 usec
DE                       6.50 usec
TE                      305.6 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                        1

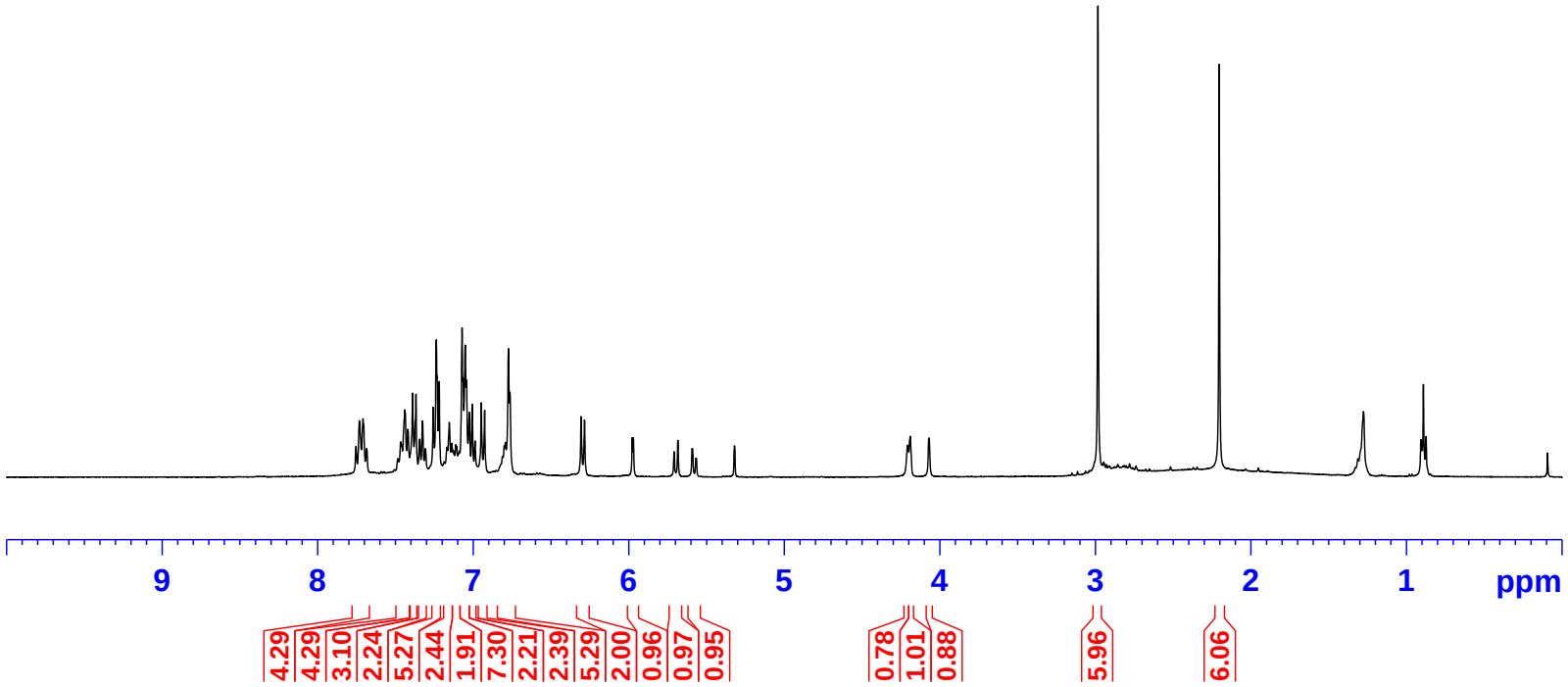
======== CHANNEL f1 ========
NUC1                      31P
P1                      14.70 usec
PL1                      4.00 dB
PL1W             10.30000019 W
SFO1             161.9917814 MHz

======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                      1H
PCPD2                   90.00 usec
PL2                     -1.00 dB
PL12                    15.16 dB
PL2W             13.56617069 W
PL12W             0.32844096 W
SFO2             400.1916008 MHz
SI                     32768
SF               161.9997813 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```



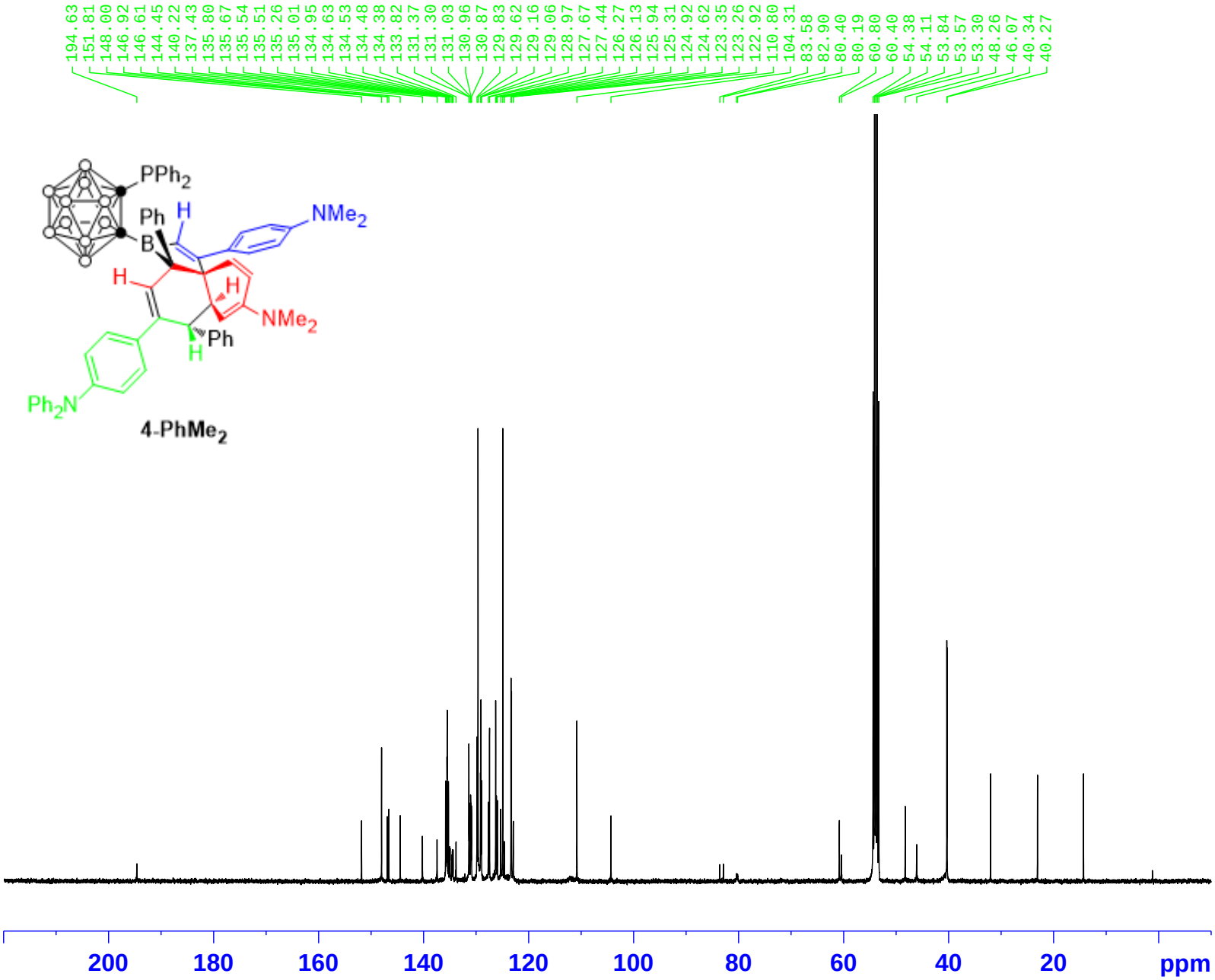


4-PhMe$_2$



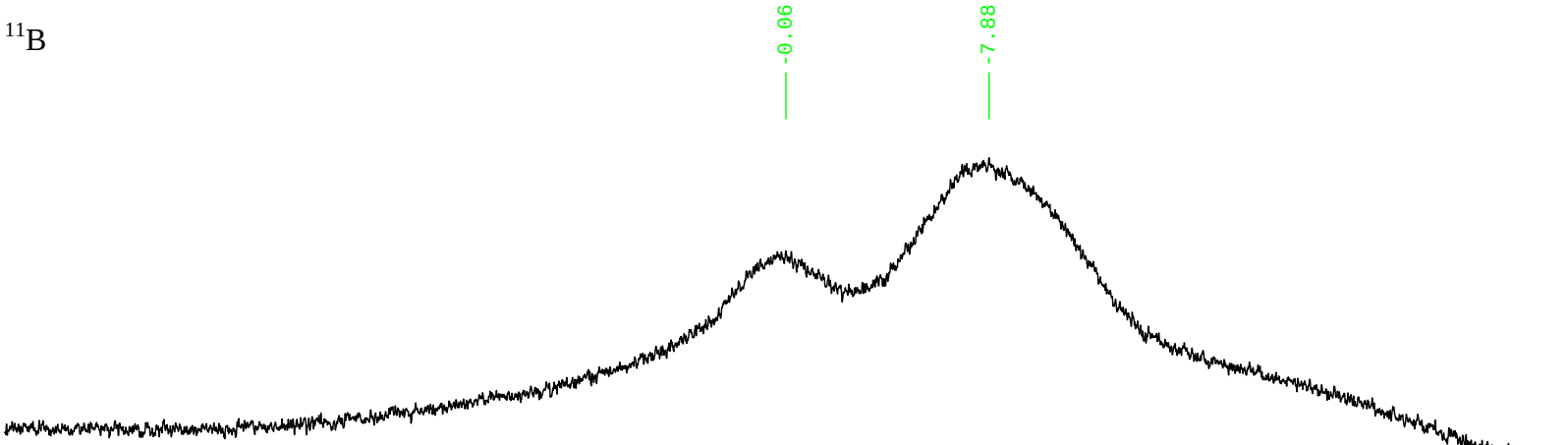

```
NAME                 4-PhMe2
EXPNO                      1
PROCNO                     1
Date_               20130220
Time                   13.46
INSTRUM                spect
PROBHD    5 mm PADUL 13C
PULPROG                zg30
TD                    65536
SOLVENT            CD2Cl2
NS                        16
DS                         2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                3.9846387 sec
RG                       144
DW                    60.800 usec
DE                     6.50 usec
TE                    294.8 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      1H
P1                     15.69 usec
PL1                     0.00 dB
PL1W             8.31434441 W
SFO1            400.1324710 MHz
SI                      32768
SF              400.1300155 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

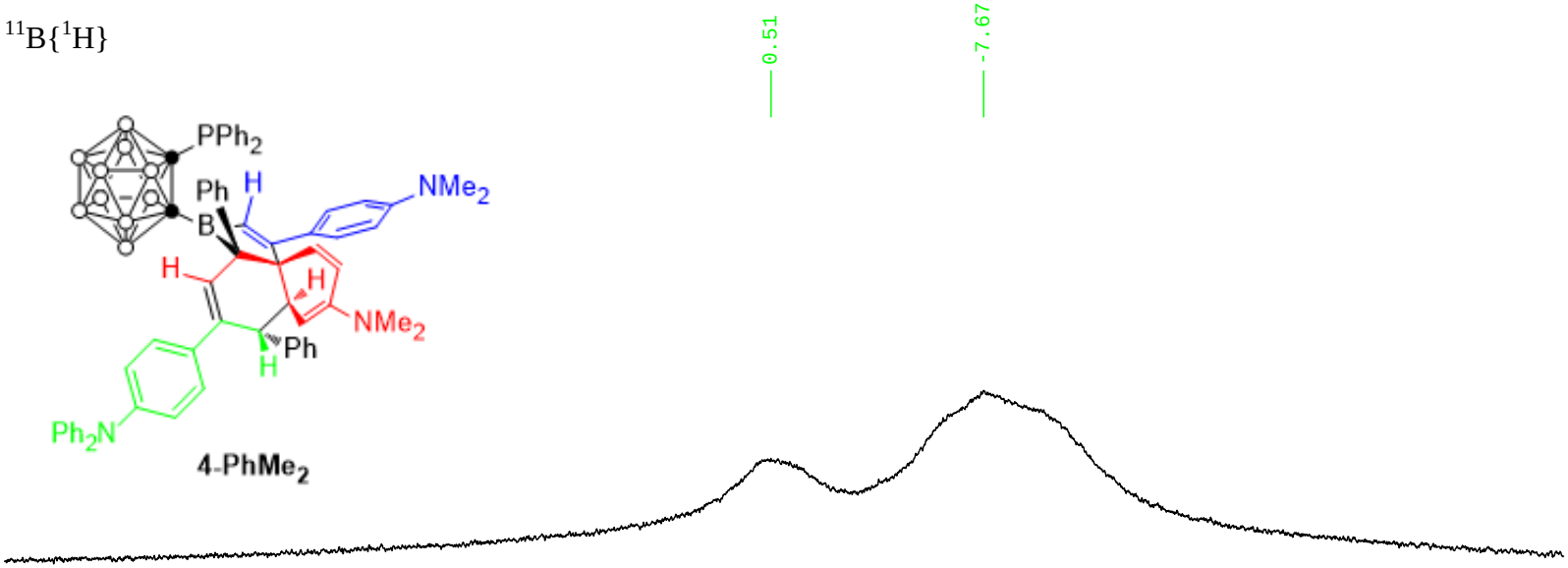


NAME	4-PhMe2
EXPNO	2
PROCNO	1
Date_	20130220
Time	19.40
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CD2Cl2
NS	15096
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	256
DW	20.800 usec
DE	6.50 usec
TE	299.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.90 usec
PL1	-2.00 dB
PL1W	55.33689499 W
SFO1	100.6379183 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-1.00 dB
PL12	15.16 dB
PL13	18.62 dB
PL2W	13.56617069 W
PL12W	0.32844096 W
PL13W	0.14806664 W
SFO2	400.1916008 MHz
SI	32768
SF	100.6278154 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

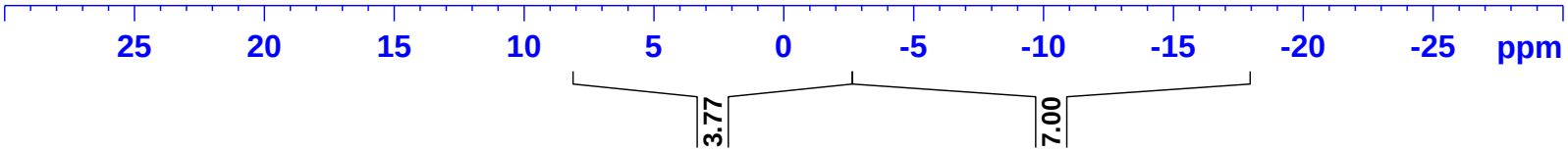
^{11}B



$^{11}B\{^{1}H\}$



4-PhMe$_2$

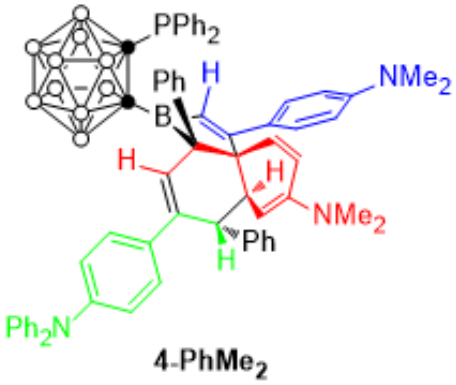



```
NAME                 4-PhMe2
EXPNO                      4
PROCNO                     1
Date_               20130220
Time                   10.19
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zgdc
TD                     65536
SOLVENT             CD2Cl2
NS                         16
DS                          0
SWH               25510.203 Hz
FIDRES             0.389255 Hz
AQ                1.2845556 sec
RG                       406
DW                  19.600 usec
DE                   6.50 usec
TE                   296.3 K
D1               5.00000000 sec
D11              0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     11B
P1                    7.60 usec
PL1                  -3.00 dB
PL1W           55.13059616 W
SFO1           128.3968556 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                90.00 usec
PL2                  -1.00 dB
PL12                 15.16 dB
PL2W           13.56617069 W
PL12W           0.32844096 W
SFO2           400.1916008 MHz
SI                       32768
SF             128.3968897 MHz
WDW                      EM
SSB                       0
LB                    3.00 Hz
GB                        0
PC                     1.40
```

^{31}P

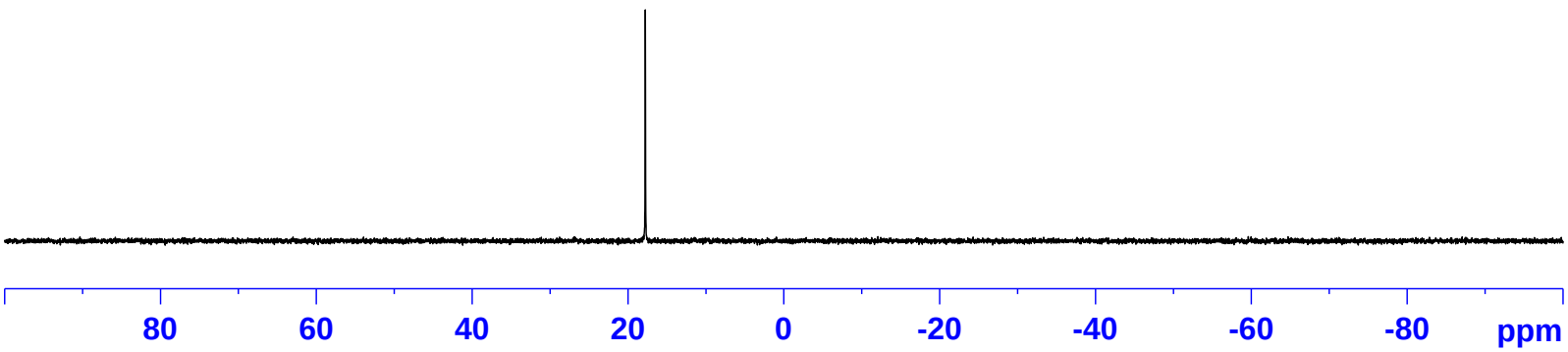


4-PhMe$_2$

17.77

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

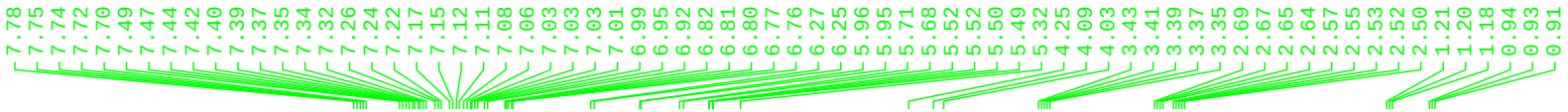
17.77

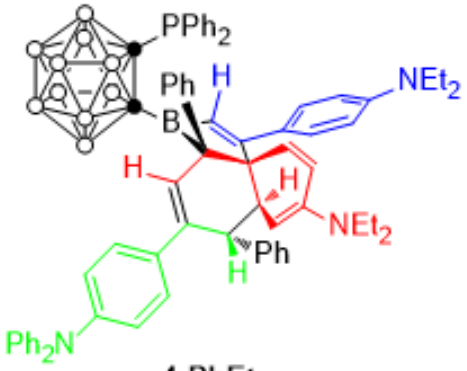



```
NAME                 4-PhMe2
EXPNO                      6
PROCNO                     1
Date_               20130220
Time                   10.16
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zgdc30
TD                      65536
SOLVENT                CD2Cl2
NS                         16
DS                          0
SWH                 96153.844 Hz
FIDRES               1.467191 Hz
AQ                   0.3408372 sec
RG                        2050
DW                       5.200 usec
DE                       6.50 usec
TE                      296.2 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      31P
P1                      14.70 usec
PL1                      4.00 dB
PL1W             10.30000019 W
SFO1             161.9917814 MHz

======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                      1H
PCPD2                   90.00 usec
PL2                     -1.00 dB
PL12                    15.16 dB
PL2W             13.56617069 W
PL12W             0.32844096 W
SFO2             400.1916008 MHz
SI                       32768
SF               161.9997813 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

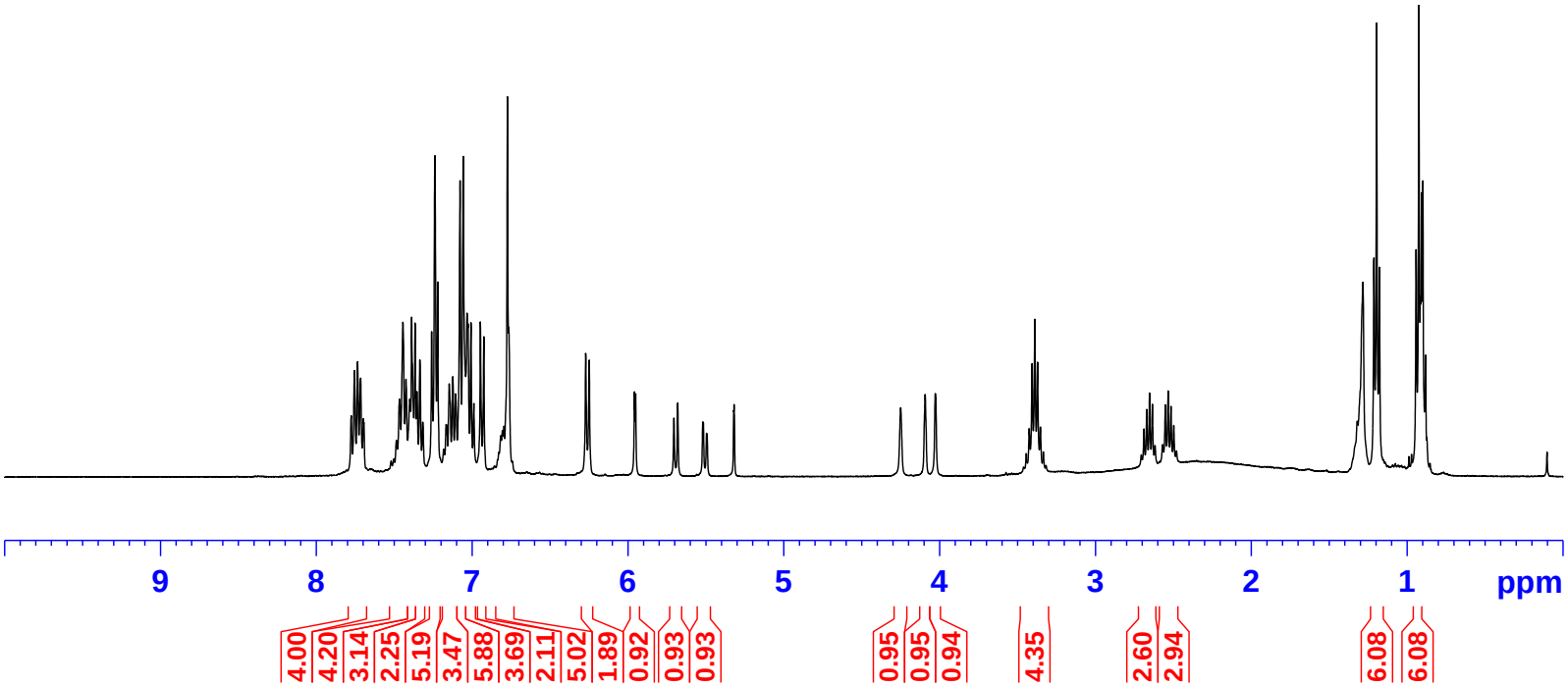


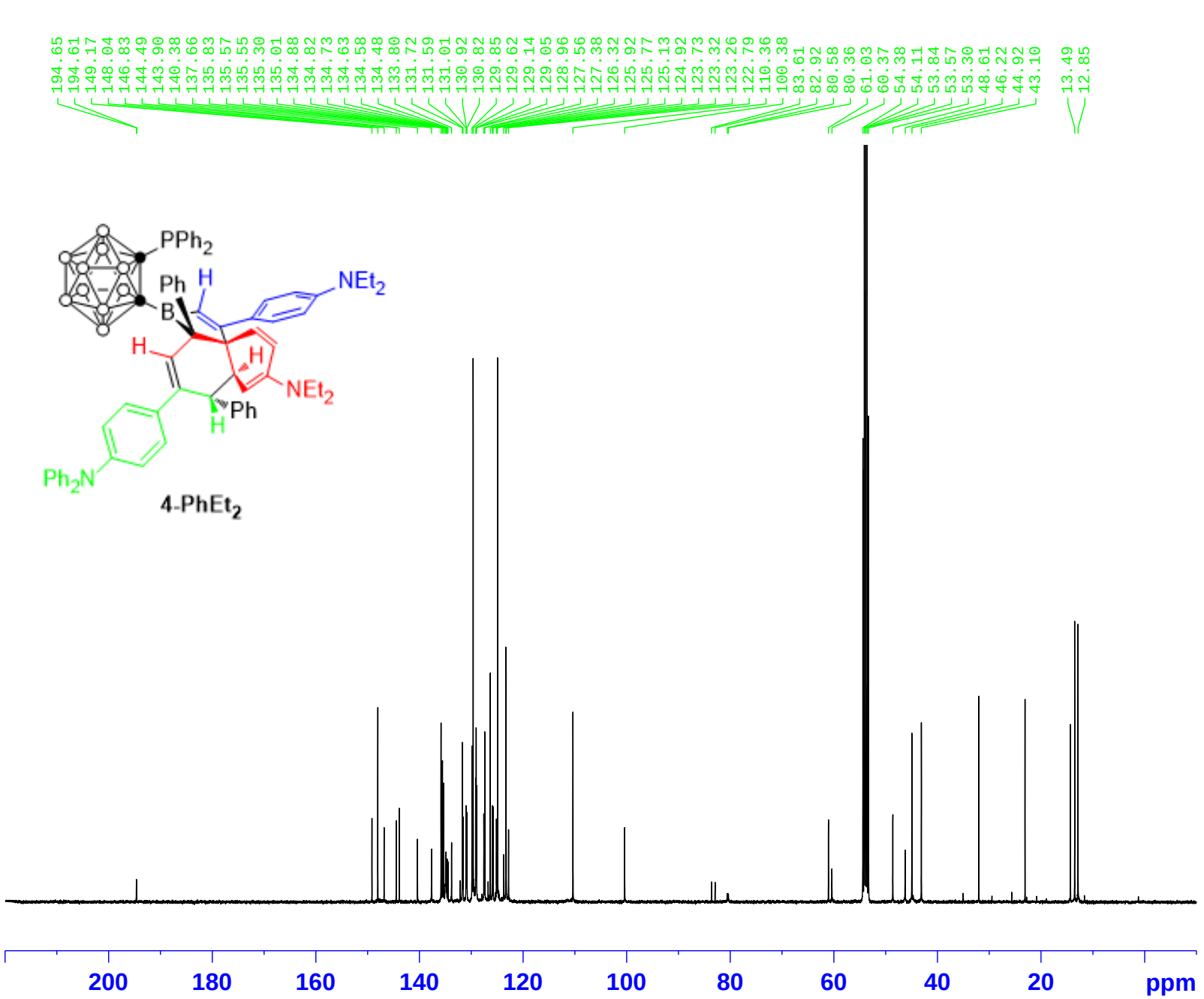


4-PhEt$_2$

```
NAME                  4-PhEt2
EXPNO                       1
PROCNO                      1
Date_                20130224
Time                    15.37
INSTRUM                 spect
PROBHD    5 mm PADUL 13C
PULPROG                 zg30
TD                     65536
SOLVENT             CD2Cl2
NS                          7
DS                          2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                      90.5
DW                    60.800 usec
DE                     6.50 usec
TE                    295.3 K
D1                 1.00000000 sec
TD0                         1

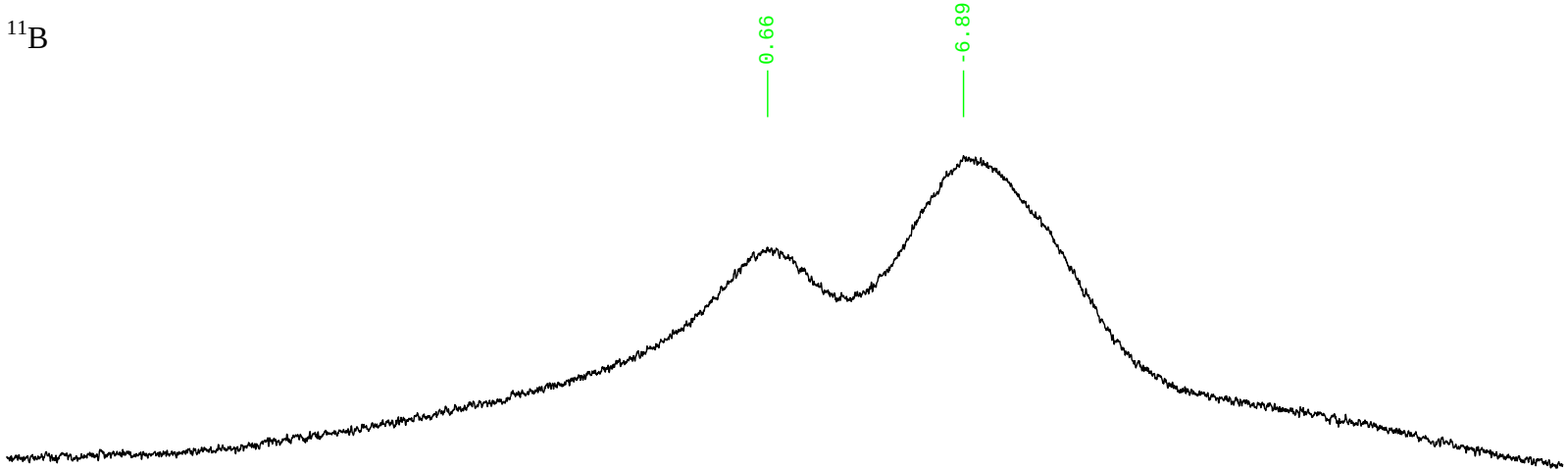
======== CHANNEL f1 ========
NUC1                       1H
P1                      15.69 usec
PL1                      0.00 dB
PL1W               8.31434441 W
SFO1              400.1324710 MHz
SI                        32768
SF                400.1300155 MHz
WDW                        EM
SSB                         0
LB                      0.30 Hz
GB                          0
PC                      1.00
```



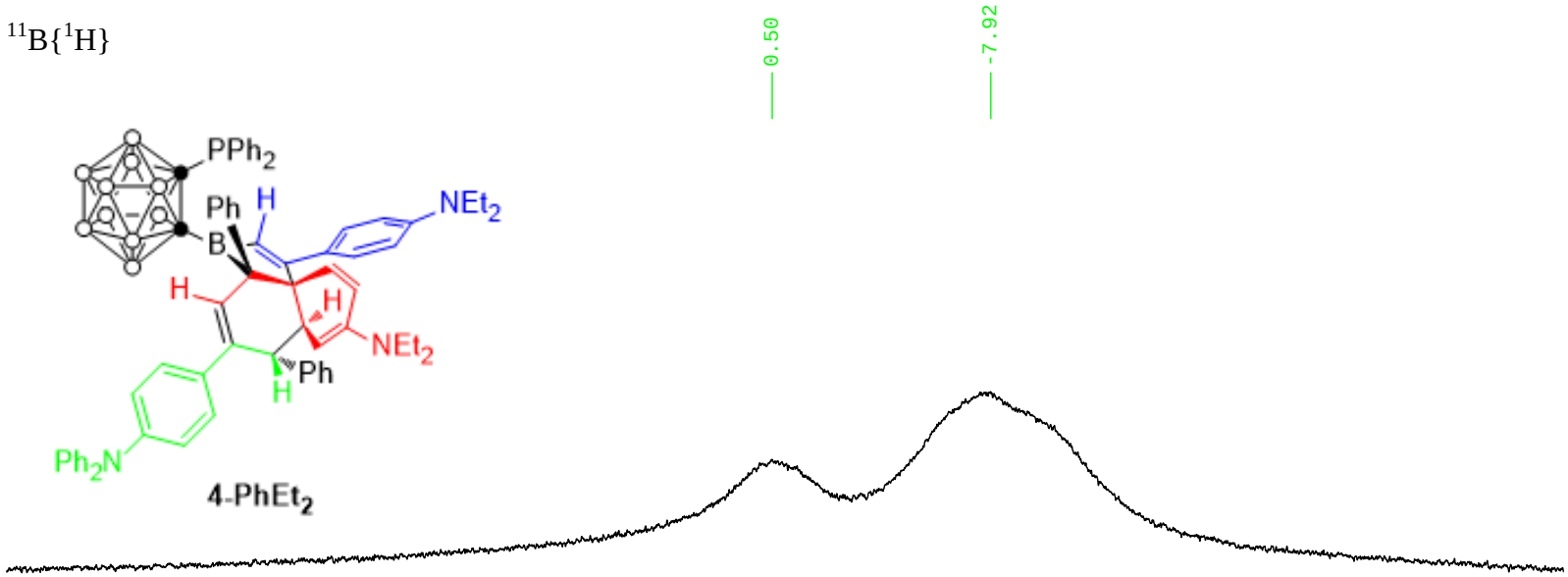


NAME	4-PhEt2
EXPNO	2
PROCNO	1
Date_	20130224
Time	15.40
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zgpg30
TD	65536
SOLVENT	CD2Cl2
NS	18132
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	203
DW	20.800 usec
DE	6.50 usec
TE	295.5 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	9.68 usec
PL1	-0.60 dB
PL1W	41.24164963 W
SFO1	100.6228298 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	0.00 dB
PL12	15.17 dB
PL13	15.92 dB
PL2W	8.31434441 W
PL12W	0.25282964 W
PL13W	0.21272963 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127271 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

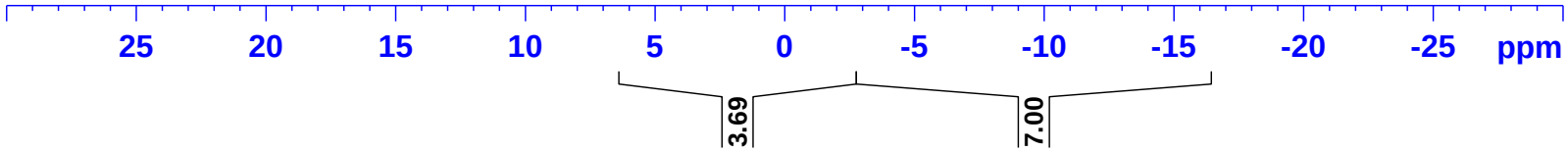
^{11}B



$^{11}B\{^{1}H\}$



4-PhEt$_2$

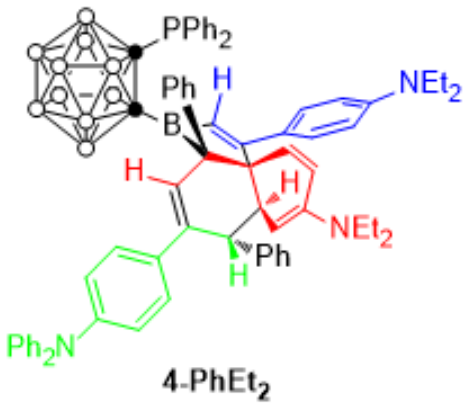



```
NAME                 4-PhEt2
EXPNO                      4
PROCNO                     1
Date_               20130223
Time                   16.46
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                 zgdc
TD                     65536
SOLVENT             CD2Cl2
NS                         8
DS                         0
SWH               25510.203 Hz
FIDRES             0.389255 Hz
AQ                1.2845556 sec
RG                       362
DW                  19.600 usec
DE                   6.50 usec
TE                   295.6 K
D1               5.00000000 sec
D11              0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     11B
P1                    7.60 usec
PL1                  -3.00 dB
PL1W          55.13059616 W
SFO1          128.3968556 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                90.00 usec
PL2                  -1.00 dB
PL12                 15.16 dB
PL2W          13.56617069 W
PL12W          0.32844096 W
SFO2          400.1916008 MHz
SI                   32768
SF            128.3968897 MHz
WDW                     EM
SSB                      0
LB                    3.00 Hz
GB                       0
PC                    1.40
```

^{31}P



4-PhEt$_2$

17.61

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

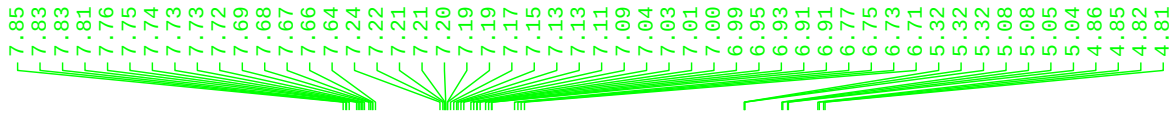
17.61

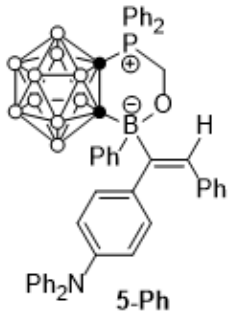
80 60 40 20 0 -20 -40 -60 -80 ppm

```
NAME                 4-PhEt2
EXPNO                      6
PROCNO                     1
Date_               20130223
Time                   16.43
INSTRUM                spect
PROBHD    5 mm PABBO BB-
PULPROG                zgdc30
TD                      65536
SOLVENT                CD2Cl2
NS                         10
DS                          0
SWH                 96153.844 Hz
FIDRES               1.467191 Hz
AQ                   0.3408372 sec
RG                        2050
DW                       5.200 usec
DE                       6.50 usec
TE                      295.5 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                         1

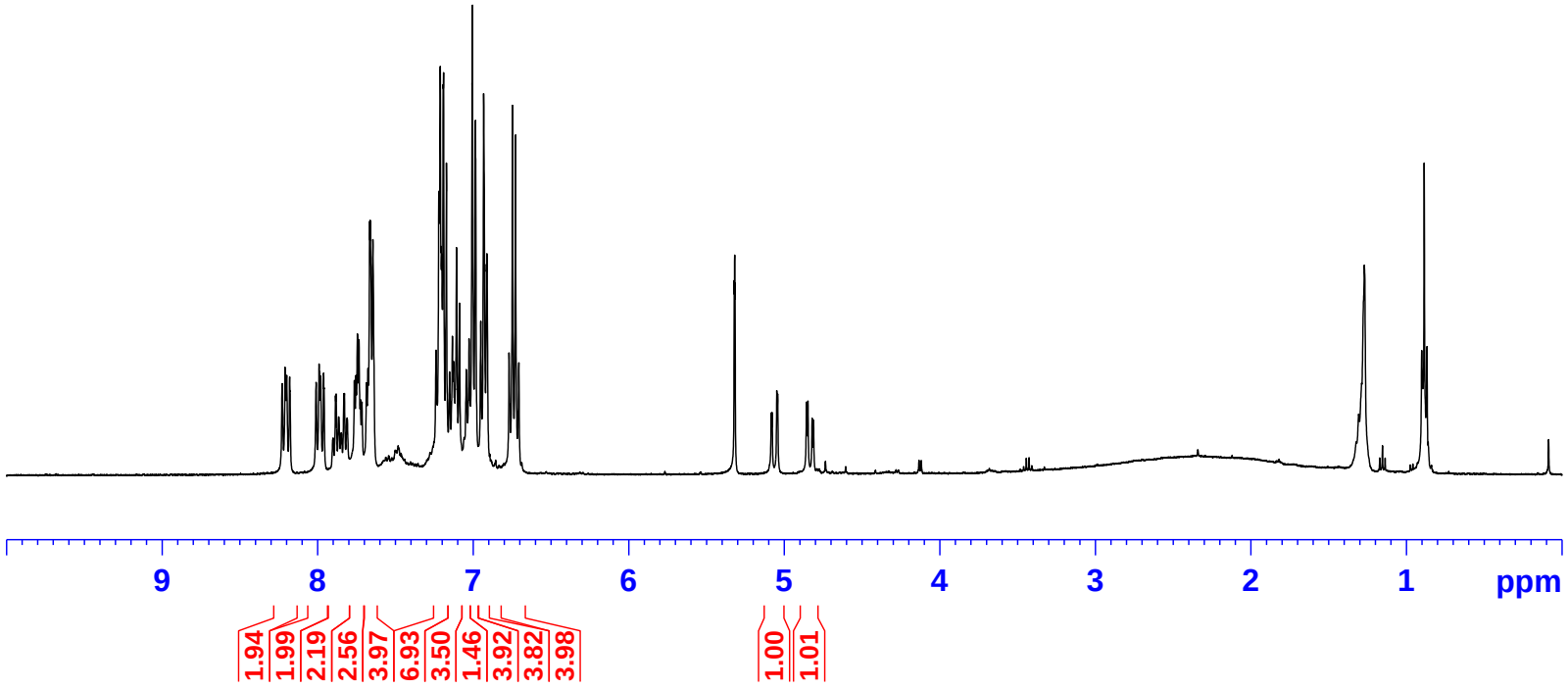
======== CHANNEL f1 ========
NUC1                      31P
P1                      14.70 usec
PL1                      4.00 dB
PL1W             10.30000019 W
SFO1             161.9917814 MHz

======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                      1H
PCPD2                   90.00 usec
PL2                     -1.00 dB
PL12                    15.16 dB
PL2W             13.56617069 W
PL12W             0.32844096 W
SFO2             400.1916008 MHz
SI                       32768
SF               161.9997813 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB                          0
PC                       1.40
```

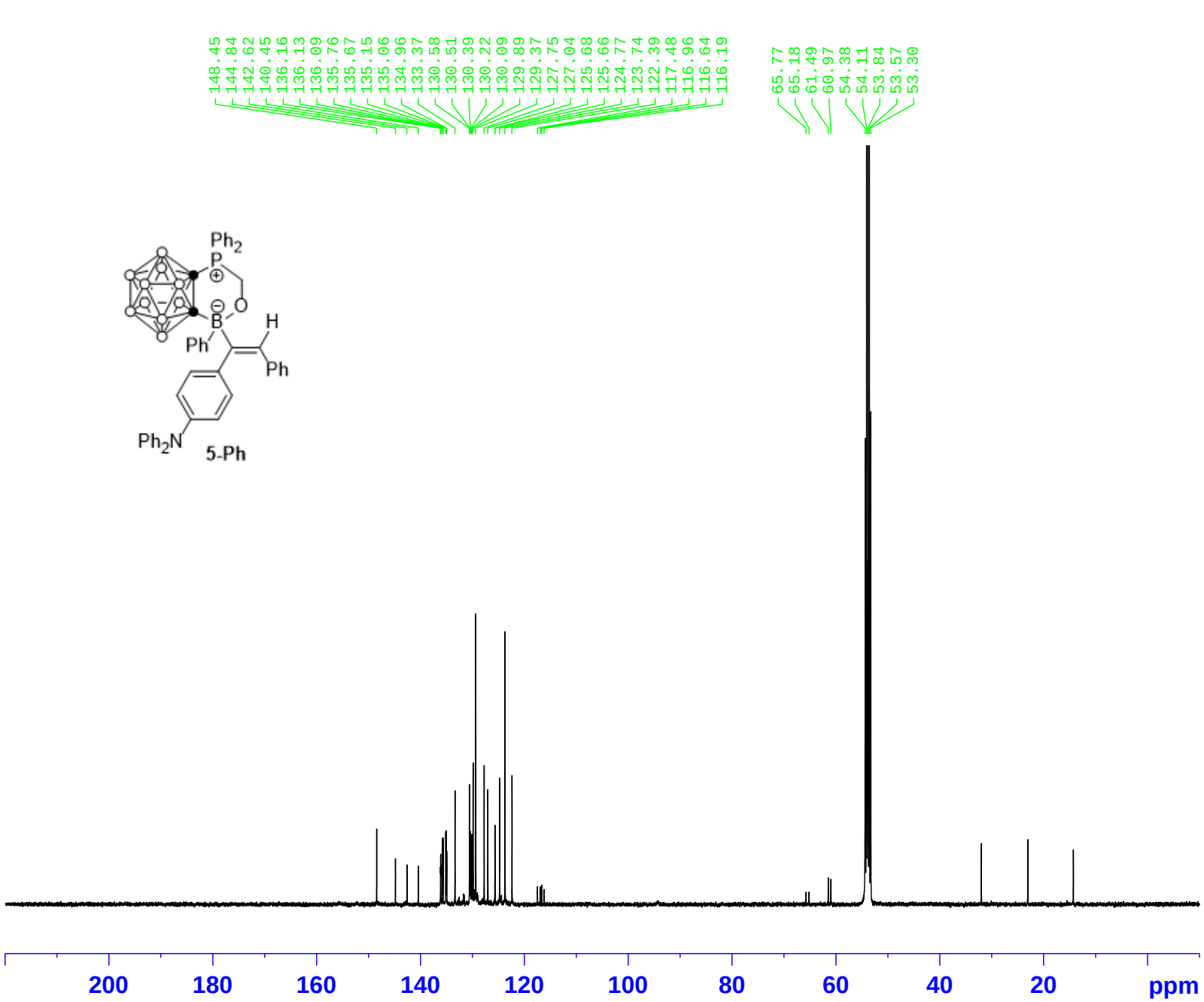




5-Ph



NAME	5-Ph
EXPNO	1
PROCNO	1
Date_	20130222
Time	18.26
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CD2Cl2
NS	16
DS	0
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	203
DW	60.800 usec
DE	6.50 usec
TE	295.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	15.69 usec
PL1	0.00 dB
PL1W	8.31434441 W
SFO1	400.1324710 MHz
SI	32768
SF	400.1300155 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



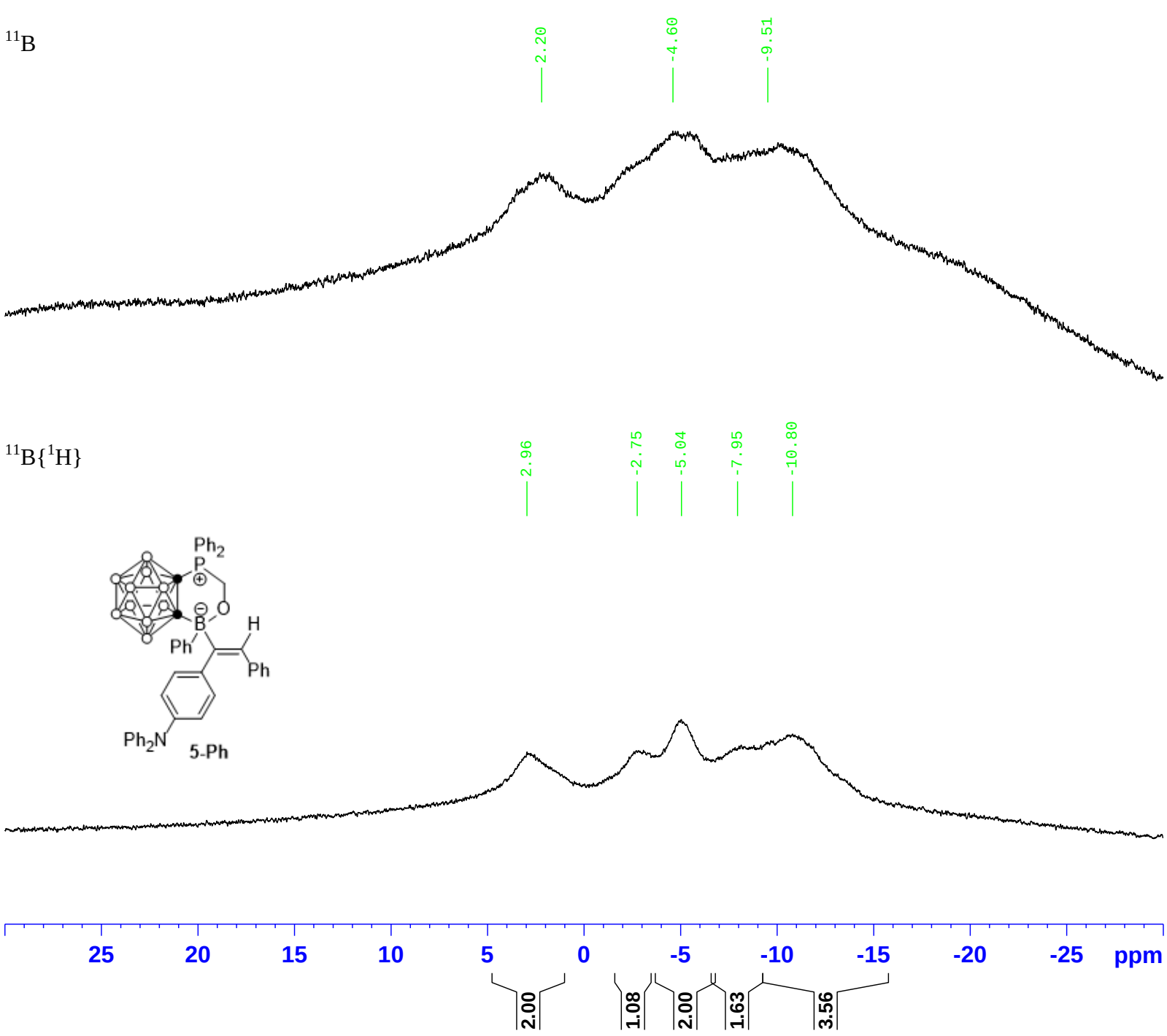

```
NAME                  5-Ph
EXPNO                    2
PROCNO                   1
Date_             20130223
Time                 21.57
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG              zgpg30
TD                    65536
SOLVENT              CD2Cl2
NS                    21568
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                      203
DW                   20.800 usec
DE                    6.50 usec
TE                   295.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    9.90 usec
PL1                  -2.00 dB
PL1W          55.33689499 W
SFO1          100.6379183 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2                90.00 usec
PL2                  -1.00 dB
PL12                 15.16 dB
PL13                 18.62 dB
PL2W          13.56617069 W
PL12W          0.32844096 W
PL13W          0.14806664 W
SFO2          400.1916008 MHz
SI                   32768
SF            100.6278156 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

^{11}B

$^{11}B\{^{1}H\}$

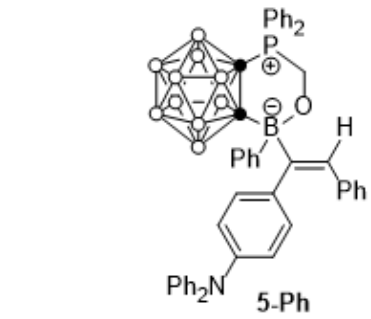



```
NAME                  5-Ph
EXPNO                    4
PROCNO                   1
Date_             20130221
Time                 10.30
INSTRUM              spect
PROBHD    5 mm PABBO BB-
PULPROG               zgdc
TD                   65536
SOLVENT           CD2Cl2
NS                       28
DS                        0
SWH              25510.203 Hz
FIDRES            0.389255 Hz
AQ                1.2845556 sec
RG                      406
DW                   19.600 usec
DE                    6.50 usec
TE                   296.5 K
D1             5.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   11B
P1                    7.60 usec
PL1                  -3.00 dB
PL1W          55.13059616 W
SFO1         128.3968556 MHz

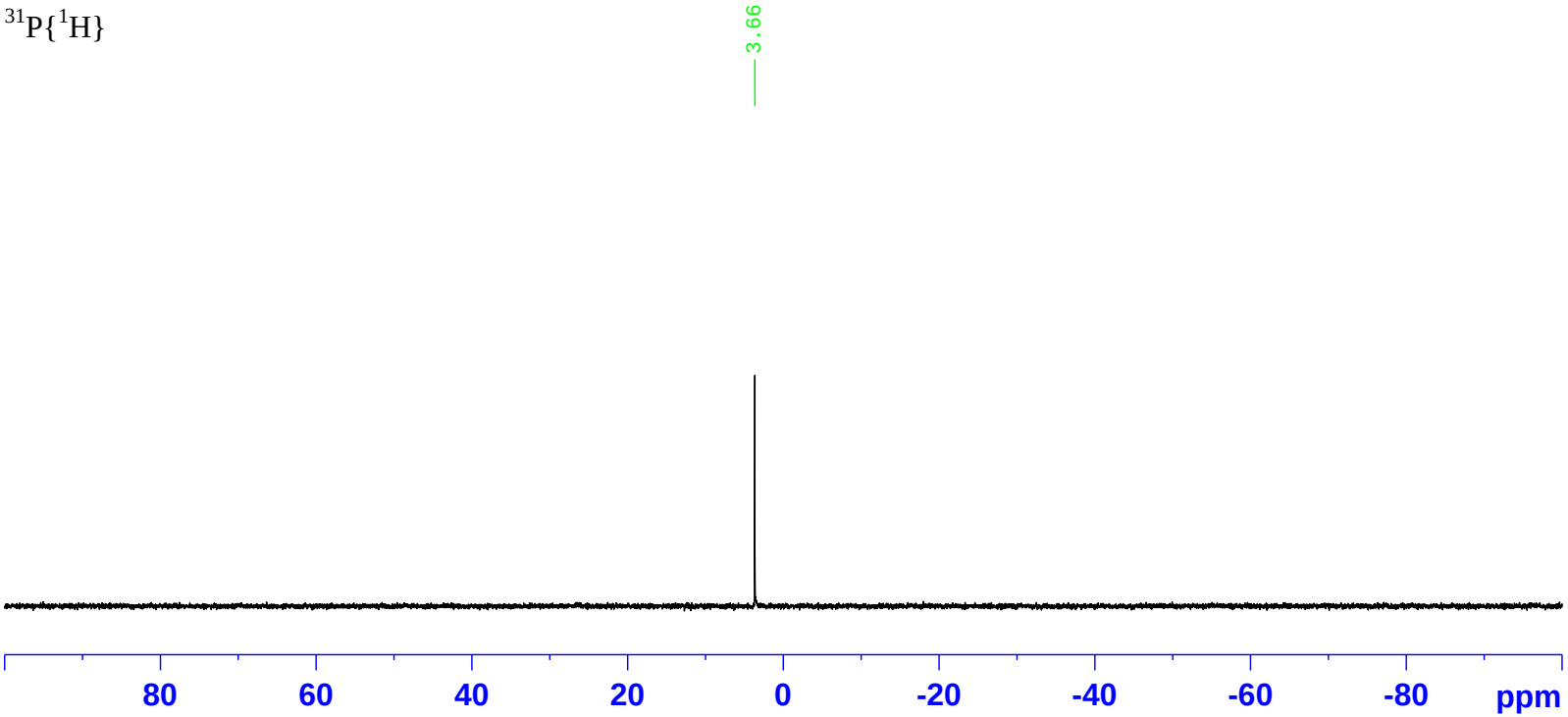
======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2                90.00 usec
PL2                  -1.00 dB
PL12                 15.16 dB
PL2W          13.56617069 W
PL12W          0.32844096 W
SFO2         400.1916008 MHz
SI                   32768
SF           128.3968897 MHz
WDW                    EM
SSB                       0
LB                    3.00 Hz
GB                        0
PC                    1.40
```

^{31}P



5-Ph

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




```
NAME                 5-Ph
EXPNO                   6
PROCNO                  1
Date_            20130221
Time                10.26
INSTRUM             spect
PROBHD    5 mm PABBO BB-
PULPROG           zgdc30
TD                 65536
SOLVENT            CD2Cl2
NS                     10
DS                      0
SWH             96153.844 Hz
FIDRES           1.467191 Hz
AQ               0.3408372 sec
RG                  2050
DW                  5.200 usec
DE                  6.50 usec
TE                 296.3 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  31P
P1                  14.70 usec
PL1                  4.00 dB
PL1W         10.30000019 W
SFO1         161.9917814 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2               90.00 usec
PL2                 -1.00 dB
PL12                15.16 dB
PL2W         13.56617069 W
PL12W         0.32844096 W
SFO2         400.1916008 MHz
SI                 32768
SF           161.9997813 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```