Electronic Supplementary Material (ESI) for ChemComm.

Formation of phosphanoxy-substituted phosphaalkenes via metathesis-type cleavage of the kinetically stabilised P=P bond

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1. General Information

All manipulations with organolithium reagents were carried out under an argon atmosphere by means of the standard Schlenk techniques, and the employed solvents were dried by appropriate methods. ^{1}H, $^{13}C\{^{1}H\}$, and ^{19}F NMR spectra were recorded on a Bruker AV300M spectrometer in $CDCl_3$ at 298 K with internal Me_4Si (^{1}H, ^{13}C) and benzotrifluoride (BTF, ^{19}F) standards. $^{31}P\{^{1}H\}$ NMR spectra were recorded on a Bruker AV300M spectrometer in $CDCl_3$ at 298 K with an external 85% H_3PO_4 (^{31}P) standard. UV-Vis spectra were recorded with a Jasco V570 and a Hitachi U-4100 spectrometers. MS spectra were taken on a JEOL T100LC spectrometer. X-ray diffraction data were collected on a Rigaku RAXIS-Rapid diffractometer, and structures were solved by a direct method (SHELXL-97).[1] The X-ray structure solution and refinement were carried out using the Yadokari-XG software.[2] DFT calculations for a single and isolated species were carried out with Gaussian 09 program package.[3] Compound **1** was synthesized according to the previous report.[4]

2. Experimental Details

3: To a solution of **1** (276 mg, 0.50 mmol) in THF (13 mL) was added *n*-butyllithium (0.75 mmol, 1.60 M solution in hexane, 1 M = 1 mol dm^{-3}) at –40 °C. After stirring at –40 °C for 5 min, benzoyl chloride (93 μL 0.80 mmol) was added to reaction mixture at –40 °C. The reaction mixture was allowed to warm to room temperature and stirred for 1 h. The volatile materials were removed *in vacuo*, and the resulting residue was dissolved in hexane and filtrated off. The solvent was removed *in vacuo*, and residual solid was recrystallized from EtOAc-MeCN to afford **3** as a yellow solid (243 mg, 68% yield).

Mp 137–139 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.79 (t, 3H, PC_3H_6*Me*), 1.30–1.60 (m, 5H, PBu), 1.37 (s, 9H, *p*-*t*Bu), 1.39 (s, 9H, *p*-*t*Bu), 1.42 (s, 9H, *o*-*t*Bu), 1.54 (s, 9H, *o*-*t*Bu), 1.65 (s, 18H, *o*-*t*Bu), 2.35–2.42 (m, 1H, PCH*H*Pr), 6.65 (d, 2H, ^{3}J(H,H) = 7.8 Hz, Ph), 6.92 (pt, 2H, {^{3}J(H,H)+^{3}J(H,H)}/2 = 7.7 Hz, Ph), 7.03 (t, 1H, ^{3}J(H,H) = 7.5 Hz, Ph), 7.39 (d, ^{4}J(P,H) = 0.90 Hz, 2H, *m*-Mes*), 7.48 (d, ^{4}J(P,H) = 1.8 Hz, 2H, *m*-Mes*); ^{31}P{^{1}H} NMR (121 MHz, $CDCl_3$) δ 127.5 (d, ^{2}J(P,P) = 135.5 Hz, P_{sp3}), 166.5 (d, ^{2}J(P,P) = 135.5 Hz, P_{sp2}); ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.0 (s, PC_3H_6*Me*), 23.8 (d, ^{3}J(P,C) = 13.6 Hz, PC_2H_4*C*H_2Me), 28.2 (d, ^{2}J(P,C) = 21.9 Hz, PCH_2*C*H_2Et), 31.5 (s, *p*-C*Me*$_3$), 31.7 (s, *p*-C*Me*$_3$), 32.5 (d, ^{4}J(P,C) = 7.5 Hz, *o*-C*Me*$_3$), 32.8 (d, ^{4}J(P,C) = 6.8 Hz, *o*-C*Me*$_3$), 34.5 (d, ^{4}J(P,C) = 9.1 Hz, *o*-C*Me*$_3$), 35.1 (s, *p*-*C*Me$_3$), 35.2 (s, *p*-*C*Me$_3$), 37.3 (dd, ^{1}J(P,C) = 25.3 Hz, ^{4}J(P,C) = 3.4 Hz, P*C*H_2Pr), 38.2 (d, ^{3}J(P,C) = 7.5 Hz, *o*-*C*Me$_3$), 39.1 (d, ^{3}J(P,C) = 3.8 Hz, *o*-*C*Me$_3$), 122.3 (d, ^{3}J(P,C) = 15.1 Hz, *m*-Mes*), 122.9 (d, ^{3}J(P,C) = 5.3 Hz, *m*-Mes*), 126.8 (d, *J*(P,C) = 6.0 Hz, Ph), 127.2 (d, *J*(P,C) = 2.3 Hz, Ph), 127.3 (d, *J*(P,C) = 3.8 Hz, Ph), 133.4 (dd, ^{1}J(P,C) = 59.6 Hz, ^{4}J(P,C) = 11.3 Hz, *ipso*-Mes*), 139.6 (d, ^{2}J(P,C) = 16.6 Hz, *ipso*-Ph), 140.4 (d, ^{1}J(P,C) = 61.9 Hz, *ipso*-Mes*), 150.0 (d, ^{4}J(P,C) = 1.5 Hz, *p*-Mes*), 150.7 (s, *p*-Mes*), 155.1 (d, ^{2}J(P,C) = 2.3 Hz, *o*-Mes*), 155.4 (d, ^{2}J(P,C) = 14.3 Hz, *o*-Mes*), 194.7 (dd, ^{1}J(P,C) = 40.4 Hz, ^{2}J(P,C) = 15.5 Hz, P=C); APCI-MS Calcd. for $C_{47}H_{73}OP_2$ $[M+H]^+$: 715.5137, Found: 715.5102.

5: To a solution of **1** (276 mg, 0.50 mmol) in THF (13 mL) was added *n*-butyllithium (0.75 mmol) at –40 °C. After stirring at –40 °C for 5 min, 4-(trifluoromethyl)benzoyl chloride (119 μL 0.80 mmol) was added to reaction mixture at –40 °C. The reaction mixture was allowed to warm to room temperature and stirred for 1 h. The volatile materials were removed *in vacuo*, and the resulting residue was dissolved in hexane and filtrated off. The solvent was removed *in vacuo*, and residual solid was recrystallized from EtOH-MeCN to afford **5** as a

yellow solid (165 mg, 42% yield).

Mp 124–125 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.77 (t, 3H, ^{3}J(H,H) = 7.1 Hz, PC_3H_6*Me*), 1.30–1.60 (m, 5H, PBu), 1.34 (s, 9H, *p*-*t*Bu), 1.36 (s, 9H, *p*-*t*Bu), 1.38 (s, 9H, *o*-*t*Bu), 1.50 (s, 9H, *o*-*t*Bu), 1.61 (s, 18H, *o*-*t*Bu), 2.36–2.42 (m, 1H, PCH*H*Pr), 6.70 (d, 2H, ^{3}J(H,H) = 8.3 Hz, Ph), 7.15 (d, 2H, ^{3}J(H,H) = 8.3 Hz, Ph), 7.36 (s, 2H, *m*-Mes*), 7.46 (d, 2H, ^{4}J(P,H) = 2.1 Hz, *m*-Mes*); ^{31}P{^{1}H} NMR (121 MHz, $CDCl_3$) δ 129.3 (d, ^{3}J(P,P) = 135.5 Hz, P_{sp3}), 176.0 (d, ^{3}J(P,P) = 135.5 Hz, P_{sp2}); ^{19}F NMR (282 MHz, $CDCl_3$) δ –62.7 (s); ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.0 (s, PC_3H_6*Me*), 23.8 (d, ^{3}J(P,C) = 12.8 Hz, PC_2H_4*C*H_2Me), 28.2 (d, ^{2}J(P,C) = 21.1 Hz, PCH_2*C*H_2Et), 31.4 (s, *p*-C*Me*$_3$), 31.6 (s, *p*-C*Me*$_3$), 32.5 (d, ^{4}J(P,C) = 7.5 Hz, *o*-C*Me*$_3$), 32.8 (d, ^{4}J(P,C) = 6.8 Hz, *o*-C*Me*$_3$), 34.5 (d, ^{4}J(P,C) = 7.5 Hz, *o*-C*Me*$_3$), 35.1 (s, *p*-*C*Me$_3$), 35.2 (s, *p*-*C*Me$_3$), 37.2 (dd, ^{1}J(P,C) = 24.9 Hz, ^{4}J(P,C) = 3.0 Hz, P*C*H_2Pr), 38.2 (d, ^{3}J(P,C) = 7.5 Hz, *o*-*C*Me$_3$), 39.1 (d, ^{3}J(P,C) = 3.8 Hz, *o*-*C*Me$_3$), 122.4 (d, ^{3}J(P,C) = 20.4 Hz, *m*-Mes*), 123.0 (d, ^{3}J(P,C) = 6.0 Hz, *m*-Mes*), 124.0–124.2 (m, Ph), 124.3 (q, ^{1}J(F,C) = 271.7 Hz, CF_3), 126.8 (d, ^{3}J(P,C) = 6.8 Hz, Ph), 128.6 (qd, ^{2}J(F,C) = 32.5 Hz, ^{5}J(P,C) = 4.5 Hz, Ph), 132.5 (dd, ^{1}J(P,C) = 59.6 Hz, ^{4}J(P,C) = 12.1 Hz, *ipso*-Mes*), 139.7 (d, ^{1}J(P,C) = 61.1 Hz, *ipso*-Mes*), 142.4 (d, ^{2}J(P,C) = 15.1 Hz, Ph), 150.2 (d, ^{4}J(P,C) = 1.5 Hz, *p*-Mes*), 151.2 (s, *p*-Mes*), 155.2 (d, ^{2}J(P,C) = 2.3 Hz, *o*-Mes*), 155.4 (d, ^{2}J(P,C) = 14.3 Hz, *o*-Mes*), 192.8 (dd, ^{1}J(P,C) = 40.4 Hz, ^{2}J(P,C) = 16.2 Hz, P=C); APCI-MS Calcd. for $C_{48}H_{72}F_3OP_2$ $[M+H]^+$: 783.5011, Found: 783.5045.

6: To a solution of **1** (276 mg, 0.50 mmol) in THF (13 mL) was added *n*-butyllithium (0.75 mmol) at –40 °C. After stirring at –40 °C for 5 min, a solution of 4-methoxybenzoyl chloride (136 mg 0.80 mmol) in THF (1 mL) was added to reaction mixture at –40 °C. The reaction mixture was allowed to warm to room temperature and stirred for 1 h. The volatile materials were removed *in vacuo*, and the resulting residue was dissolved in hexane and filtrated off. The solvent was removed *in vacuo*, and residual solid was recrystallized from EtOH-MeCN to afford **6** as a pale yellow solid (277 mg, 74% yield).

Mp 148–150 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.76 (t, 3H, ^{3}J(H,H) = 7.1 Hz, PC_3H_6*Me*), 1.30–1.60 (m, 5H, PBu), 1.33 (s, 9H, *t*Bu), 1.36 (s, 9H, *t*Bu), 1.40 (s, 9H, *t*Bu), 1.50 (s, 9H, *t*Bu), 1.61 (s, 18H, *o*-*t*Bu), 2.31–2.36 (m, 1H, PC*H*$_2$Pr), 3.70 (s, 3H, OMe), 6.42 (d, 2H, ^{3}J(H,H) = 9.3 Hz, Ar), 6.55 (dd, 2H, ^{3}J(H,H) = 9.3 Hz, ^{4}J(H,P) = 2.1 Hz, Ar), 7.36 (d, 2H, ^{4}J(H,P) = 1.2 Hz, *m*-Mes*), 7.44 (d, 2H, ^{4}J(H,P) = 1.8 Hz, *m*-Mes*); ^{31}P{^{1}H} NMR (121 MHz, $CDCl_3$) δ 126.8 (d, ^{3}J(P,P) = 135.5 Hz, P_{sp3}), 157.5 (d, ^{3}J(P,P) = 135.5 Hz, P_{sp2}); ^{13}C

NMR (75 MHz, $CDCl_3$) δ 14.1 (s, PC_3H_6*Me*), 23.8 (d, ^3J(P,C) = 12.8 Hz, PC_2H_4*C*H_2Me), 28.3 (d, ^2J(P,C) = 21.9 Hz, PCH_2*C*H_2Et), 31.5 (s, *p*-C*Me*$_3$), 31.7 (s, *p*-C*Me*$_3$), 32.5 (d, ^4J(P,C) = 7.5 Hz, *o*-C*Me*$_3$), 32.7 (d, ^4J(P,C) = 6.8 Hz, *o*-C*Me*$_3$), 34.5 (d, ^4J(P,C) = 7.5 Hz, *o*-C*Me*$_3$), 35.1 (s, *p*-*C*Me$_3$), 35.2 (s, *p*-*C*Me$_3$), 37.4 (dd, ^1J(P,C) = 25.7 Hz, ^4J(P,C) = 3.0 Hz, P*C*H_2Pr), 38.3 (d, ^3J(P,C) = 7.5 Hz, *o*-*C*Me$_3$), 39.1 (d, ^3J(P,C) = 3.8 Hz, *o*-*C*Me$_3$), 55.1 (s, OMe), 112.5 (d, ^3J(P,C) = 2.3 Hz, Ph), 122.3 (d, ^3J(P,C) = 25.7 Hz, *m*-Mes*), 122.9 (d, ^3J(P,C) = 6.0 Hz, *m*-Mes*), 128.4 (d, ^4J(P,C) = 6.8 Hz, Ph), 133.0 (d, ^2J(P,C) = 16.6 Hz, Ph), 133.9 (dd, ^1J(P,C) = 60.4 Hz, ^4J(P,C) = 11.3 Hz, *ipso*-Mes*), 140.6 (d, ^1J(P,C) = 61.9 Hz, *ipso*-Mes*), 149.9 (d, ^4J(P,C) = 1.5 Hz, *p*-Mes*), 150.5 (s, *p*-Mes*), 155.2 (s, *o*-Mes*), 155.4 (d, ^2J(P,C) = 15.8 Hz, *o*-Mes*), 158.7 (d, ^5J(P,C) = 4.5 Hz, Ph), 194.4 (dd, ^1J(P,C) = 40.8 Hz, ^2J(P,C) = 15.8 Hz, P=C); APCI-MS Calcd. for $C_{48}H_{75}O_2P_2$ $[M+H]^+$: 745.5242, Found: 745.5236.

7: To a solution of **1** (276 mg, 0.50 mmol) in THF (13 mL) was added *n*-butyllithium (0.75 mmol) at –40 °C. After stirring at –40 °C for 5 min, pivaloyl chloride (68 μL 0.80 mmol) was added to reaction mixture at –40 °C. The reaction mixture was allowed to warm to room temperature and stirred for 1 h. The volatile materials were removed *in vacuo*, and the resulting residue was dissolved in hexane and filtrated off. The solvent was removed *in vacuo*, and residual solid was recrystallized from *i*PrOH-MeCN to afford **7** as a colorless solid (124 mg, 36% yield).

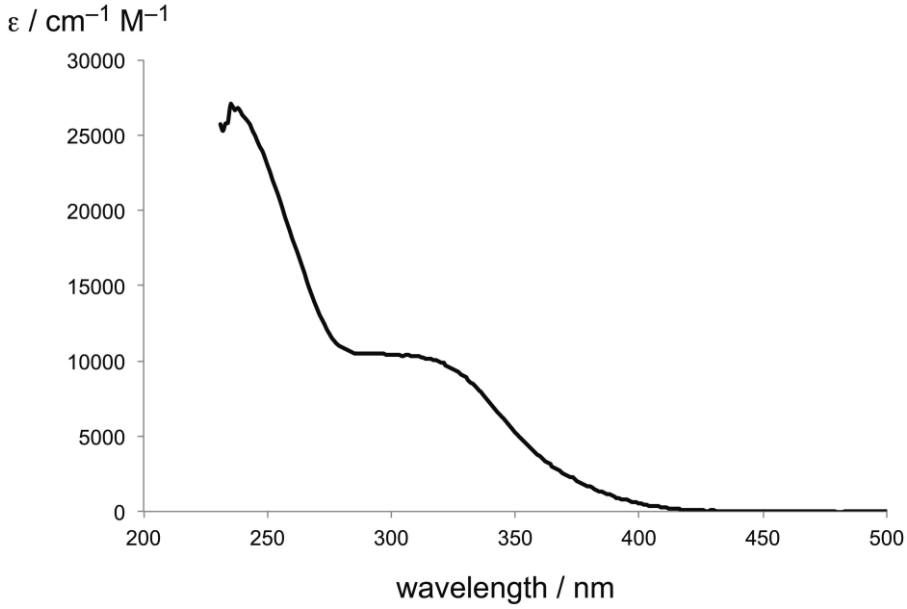
Mp 138–139 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 0.73 (t, 3H, ^3J(H,H) = 7.2 Hz, PC_3H_6*Me*), 0.76 (s, 9H, P*t*Bu), 0.80–1.40 (m, 4H, PCH_2C_2*H*$_4$Me), 1.30 (s, 9H, *p*-*t*Bu), 1.31 (s, 9H, *p*-*t*Bu), 1.49–1.61 (m, 1H, PC*H*$_2$Pr), 1.54 (s, 9H, *o*-*t*Bu), 1.60 (s, 18H, *o*-*t*Bu), 1.61 (s, 9H, *o*-*t*Bu), 2.18–2.27 (m, 1H, PC*H*$_2$Pr), 7.25–7.29 (m, 2H, *m*-Mes*), 7.39 (d, 2H, ^4J(P,H) = 1.8 Hz, *m*-Mes*); ^{31}P{^{1}H} NMR (121 MHz, $CDCl_3$) δ 124.3 (d, ^3J(P,P) = 145.8 Hz, P_{sp3}), 153.1 (d, ^3J(P,P) = 145.8 Hz, P_{sp2}); ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.0 (s, PC_3H_6*Me*), 24.2 (d, ^3J(P,C) = 13.6 Hz, PC_2H_4*C*H_2Me), 27.6 (d, ^2J(P,C) = 24.2 Hz, PCH_2*C*H_2Et), 27.9 (d, ^3J(P,C) = 3.8 Hz, P=CC*Me*$_3$), 31.4 (s, *p*-C*Me*$_3$), 31.6 (s, *p*-C*Me*$_3$), 33.3 (d, ^4J(P,C) = 8.3 Hz, *o*-C*Me*$_3$), 33.5 (d, ^4J(P,C) = 7.5 Hz, *o*-C*Me*$_3$), 34.6 (d, ^4J(P,C) = 7.5 Hz, *o*-C*Me*$_3$), 35.0 (s, *p*-*C*Me$_3$), 35.1 (s, *p*-*C*Me$_3$), 37.6 (dd, ^1J(P,C) = 25.7 Hz, ^4J(P,C) = 4.5 Hz, P*C*H_2Pr), 38.4 (d, ^3J(P,C) = 3.0 Hz, *o*-*C*Me$_3$), 39.1 (d, ^3J(P,C) = 4.5 Hz, *o*-*C*Me$_3$), 44.8 (d, ^2J(P,C) = 16.6 Hz, P=C*C*Me$_3$), 121.0 (s, *m*-Mes*), 122.5 (d, ^3J(P,C) = 6.0 Hz, *m*-Mes*), 133.6 (dd, ^1J(P,C) = 64.2 Hz, ^4J(P,C) = 12.1 Hz, *ipso*-Mes*), 142.0 (dd, ^1J(P,C) = 63.0 Hz, ^4J(P,C) = 2.6 Hz, *ipso*-Mes*), 150.0 (d, ^4J(P,C) = 2.3 Hz, *p*-Mes*), 150.2 (s, *p*-Mes*), 155.3 (dd, ^2J(P,C) = 7.9 Hz, ^5J(P,C) = 2.6 Hz, *o*-Mes*),

156.1 (d, ^{2}J(P,C) = 14.3 Hz, *o*-Mes*), 205.8 (dd, ^{1}J(P,C) = 50.6 Hz, ^{2}J(P,C) = 9.8 Hz, P=C); APCI-MS Calcd. for $C_{45}H_{77}OP_2$ $[M+H]^+$: 695.5450, Found: 695.5471.

8: To a solution of **1** (276 mg, 0.50 mmol) in THF (13 mL) was added phenyllithium (0.75 mmol, 1.08 M solution in cyclohexane-diethylether) at room temperature. After stirring at room temperature for 5 min, benzoyl chloride (93 μL 0.80 mmol) was added to reaction mixture at room temperature and stirred for 2 h. The volatile materials were removed *in vacuo*, and the resulting residue was dissolved in hexane and filtrated off. The solvent was removed *in vacuo*, and residual solid was recrystallized from *i*PrOH-MeCN to afford **8** as a yellow solid (202 mg, 55% yield).

Mp 152–153 °C; ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.37 (s, 9H, *t*Bu), 1.39 (s, 9H, *t*Bu), 1.40 (s, 9H, *t*Bu), 1.47 (s, 18H, *t*Bu), 1.54 (s, 9H, *t*Bu), 6.62–6.65 (m, 2H, Ph), 6.78–6.84 (m, 2H, Ph), 6.90 (pt, 2H, {^{3}J(H,H)+^{3}J(H,H)}/2 = 7.8 Hz, Ph), 7.03 (t, 1H ^{3}J(H,H) = 7.5 Hz, Ph), 7.14–7.17 (m, 3H, Ph), 7.39 (d, 2H, ^{4}J(P,H) = 2.4 Hz, *m*-Mes*), 7.54 (d, 2H, ^{4}J(P,H) = 2.4 Hz, *m*-Mes*); ^{31}P{^{1}H} NMR (121 MHz, $CDCl_3$) δ 108.0 (d, ^{3}J(P,P) = 131.8 Hz, P_{sp3}), 164.6 (d, ^{3}J(P,P) = 131.8 Hz, P_{sp2}); ^{13}C NMR (75 MHz, $CDCl_3$) δ 31.4 (s, *p*-C*Me*$_3$), 31.7 (s, *p*-C*Me*$_3$), 32.6 (d, ^{4}J(P,C) = 7.5 Hz, *o*-C*Me*$_3$), 32.8 (d, ^{4}J(P,C) = 6.8 Hz, *o*-C*Me*$_3$), 34.4 (d, ^{4}J(P,C) = 8.3 Hz, *o*-C*Me*$_3$), 35.2 (s, *p*-*C*Me$_3$), 35.2 (s, *p*-*C*Me$_3$), 38.3 (d, ^{3}J(P,C) = 3.0 Hz, *o*-*C*Me$_3$), 39.4 (d, ^{3}J(P,C) = 4.5 Hz, *o*-*C*Me$_3$), 122.4 (d, ^{3}J(P,C) = 4.5 Hz, *m*-Mes*), 123.3 (d, ^{3}J(P,C) = 6.8 Hz, *m*-Mes*), 127.0 (d, *J*(P,C) = 6.0 Hz, Ph), 127.3 (d, *J*(P,C) = 3.0 Hz, Ph), 127.4–127.6 (m, Phx2), 130.3 (d, ^{2}J(P,C) = 17.4 Hz, Ph), 132.8 (dd, ^{1}J(P,C) = 60.4 Hz, ^{4}J(P,C) = 10.6 Hz, *ipso*-Ar), 135.5 (dd, ^{1}J(P,C) = 59.2 Hz, ^{4}J(P,C) = 1.9 Hz, *ipso*-Ar), 139.5 (d, ^{2}J(P,C) = 15.8 Hz, *ipso*-Ph), 146.1 (dd, ^{1}J(P,C) = 27.2 Hz, ^{4}J(P,C) = 2.3 Hz, *ipso*-Ar), 150.9 (s, *p*-Mes*), 151.6 (d, ^{4}J(P,C) = 1.5 Hz, *p*-Mes*), 155.2 (dd, ^{2}J(P,C) = 3.8 Hz, ^{5}J(P,C) = 2.3 Hz, *o*-Mes*), 157.8 (d, ^{2}J(P,C) = 15.8 Hz, *o*-Mes*), 193.9 (dd, ^{1}J(P,C) = 41.5 Hz, ^{2}J(P,C) = 15.8 Hz, P=C); APCI-MS Calcd. for $C_{49}H_{69}OP_2$ $[M+H]^+$: 735.4824, Found: 735.4818.

3. UV-Vis Spectrum



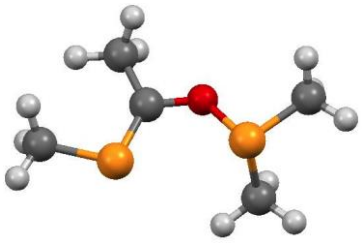
UV-Vis spectrum of **3** in CH_2Cl_2.

4. X-ray Crystallography

3: $C_{47}H_{70}OP_2$, pale yellow prisms (CH_2Cl_2-MeOH), Mw = 714.99, crystal dimensions = 0.26 x 0.21 x 0.16 mm^3, monoclinic, space group $P2_1/n$ (#14), a = 15.1477(6), b = 18.6492(7), c = 16.6712(7) Å, β = 111.4075(13)°, V = 4384.6(3) $Å^3$, Z = 4, λ = 0.71075 Å, T = 123 K, ρ_{calcd} = 1.083 g cm^{-3}, $\mu_{MoK\alpha}$ = 0.131 mm^{-1}, F_{000} = 1568, 41578 total reflections ($2\theta_{max}$ = 54.96°), index ranges = $-18 \le h \le 19$, $-24 \le k \le 24$, $-21 \le l \le 21$, 10032 unique reflections (R_{int} = 0.0549), $R1$ = 0.0394 ($I \ge 2\sigma(I)$), 0.0544 (all data), $wR2$ = 0.1164 ($I \ge 2\sigma(I)$), 0.1311 (all data), S = 0.937 (739 parameters). CCDC-1000474 contains 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.

5. DFT Calculation Data

3A

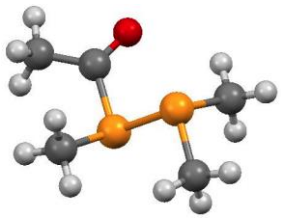


opt freq b3lyp/6-31g(d)
E(RB3LYP) = -955.676137762 A.U.
Dipole moment (field-independent basis, Debye):
X= -0.7293 Y= 1.3573 Z= 1.0316 Tot= 1.8543
Zero-point correction= 0.162929 (Hartree/Particle)
Sum of electronic and zero-point Energies= -955.513208
Sum of electronic and thermal Energies= -955.500980
Sum of electronic and thermal Enthalpies= -955.500036
Sum of electronic and thermal Free Energies= -955.551682

Standard orientation:

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms) X	Y	Z
1	15	0	-1.616500	-0.339521	-0.513229
2	15	0	1.558642	-0.899960	-0.234738
3	6	0	-1.939269	-1.386818	0.987606
4	1	0	-2.821125	-2.009850	0.794578
5	1	0	-1.087913	-2.049321	1.165960
6	1	0	-2.119109	-0.780128	1.881844
7	6	0	0.852315	0.619082	0.119177
8	8	0	-0.498845	0.795970	0.141938
9	6	0	1.519047	1.923229	0.460352
10	1	0	2.604136	1.837603	0.522193
11	1	0	1.265725	2.678345	-0.295360
12	1	0	1.137927	2.292069	1.421099
13	6	0	3.402808	-0.531047	-0.169013
14	1	0	3.736141	-0.225801	0.828358
15	1	0	3.920533	-1.461816	-0.421718
16	1	0	3.709016	0.227704	-0.896168
17	6	0	-3.065889	0.809562	-0.371924
18	1	0	-2.977970	1.606151	-1.116847
19	1	0	-3.983419	0.247532	-0.581763
20	1	0	-3.139389	1.257921	0.624630

4A

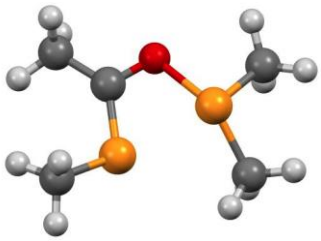


opt freq b3lyp/6-31g(d)
E(RB3LYP) = -955.694370212 A.U.
Dipole moment (field-independent basis, Debye):
X= -0.0491 Y= 2.5921 Z= 0.2970 Tot= 2.6095
Zero-point correction= 0.162866 (Hartree/Particle)
Sum of electronic and zero-point Energies= -955.531505
Sum of electronic and thermal Energies= -955.519043
Sum of electronic and thermal Enthalpies= -955.518099
Sum of electronic and thermal Free Energies= -955.569959

Standard orientation:

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms) X	Y	Z
1	15	0	1.362963	-0.210695	-0.616590
2	15	0	-0.329502	0.525242	0.673962
3	6	0	2.689266	0.957420	0.011339
4	1	0	3.653769	0.614919	-0.380954
5	1	0	2.520034	1.968070	-0.374243
6	1	0	2.752513	0.997470	1.104414
7	6	0	-1.681308	-0.608123	0.006257
8	8	0	-1.441855	-1.782906	-0.210734
9	6	0	-3.084805	-0.043502	-0.143379
10	1	0	-3.127504	0.634187	-1.005144
11	1	0	-3.786434	-0.866760	-0.300472
12	1	0	-3.377508	0.533672	0.741403
13	6	0	-0.727588	2.151163	-0.171934
14	1	0	-1.664126	2.550764	0.229444
15	1	0	0.061980	2.870081	0.068035
16	1	0	-0.808531	2.071183	-1.260624
17	6	0	1.817738	-1.735830	0.361848
18	1	0	1.012999	-2.466891	0.255382
19	1	0	2.735504	-2.159427	-0.062045
20	1	0	1.980417	-1.528988	1.425316

TS 4A to 3A

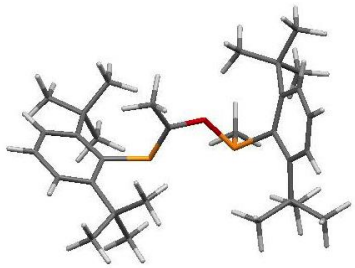


opt=qst2 freq b3lyp/6-31g(d)
E(RB3LYP) = -955.628723220 A.U.
Dipole moment (field-independent basis, Debye):
X= -1.2483 Y= -0.0813 Z= -0.0572 Tot= 1.2523
Zero-point correction= 0.161903 (Hartree/Particle)
Sum of electronic and zero-point Energies= -955.466820
Sum of electronic and thermal Energies= -955.455147
Sum of electronic and thermal Enthalpies= -955.454203
Sum of electronic and thermal Free Energies= -955.504248

Standard orientation:

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms) X	Y	Z
1	15	0	1.394697	-0.112128	0.575770
2	15	0	-0.977596	-0.836273	-0.411057
3	6	0	2.141938	-1.571707	-0.356625
4	1	0	3.202102	-1.646064	-0.077250
5	1	0	1.622356	-2.491263	-0.070462
6	1	0	2.087121	-1.467967	-1.445291
7	6	0	-1.087689	0.890799	-0.105267
8	8	0	0.065373	1.426742	0.031048
9	6	0	-2.283765	1.802765	-0.113855
10	1	0	-3.216097	1.269176	-0.312080
11	1	0	-2.361625	2.313355	0.853922
12	1	0	-2.144432	2.577066	-0.877698
13	6	0	-2.473860	-1.633284	0.394747
14	1	0	-3.384041	-1.085899	0.122504
15	1	0	-2.574362	-2.656069	0.020275
16	1	0	-2.393772	-1.665293	1.485198
17	6	0	2.644581	1.112735	-0.106765
18	1	0	2.447286	2.112189	0.285916
19	1	0	3.638811	0.786730	0.224240
20	1	0	2.649925	1.158268	-1.201759

3B



opt freq b3lyp/6-31g(d)
E(RB3LYP) = -1968.07934506 A.U.
Dipole moment (field-independent basis, Debye):
X= 1.3368 Y= -0.5033 Z= -0.7042 Tot= 1.5926
Zero-point correction= 0.723092 (Hartree/Particle)
Sum of electronic and zero-point Energies= -1967.356253
Sum of electronic and thermal Energies= -1967.316480
Sum of electronic and thermal Enthalpies= -1967.315535
Sum of electronic and thermal Free Energies= -1967.426959

Standard orientation:

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms) X	Y	Z
1	15	0	1.576604	-0.055324	0.932446
2	15	0	-1.647730	0.009077	0.796437
3	6	0	1.469400	-1.392101	2.226550
4	1	0	2.216722	-1.177419	2.998766
5	1	0	0.479092	-1.287800	2.682372
6	1	0	1.591621	-2.421290	1.890976
7	6	0	-0.680135	-0.505778	-0.517549
8	8	0	0.658462	-0.728311	-0.358319
9	6	0	-1.064052	-0.806640	-1.938253
10	1	0	-2.134885	-0.695703	-2.105905
11	1	0	-0.520189	-0.135126	-2.615433
12	1	0	-0.765562	-1.831837	-2.191997
13	6	0	3.226585	-0.058708	0.029109
14	6	0	3.738982	-1.099943	-0.825659
15	6	0	3.782655	1.262079	-0.064200
16	6	0	4.534716	-0.710678	-1.911139
17	6	0	4.606022	1.561652	-1.161102
18	6	0	4.917733	0.608354	-2.114400
19	1	0	4.884253	-1.451110	-2.617225
20	1	0	5.002851	2.560432	-1.284599
21	1	0	5.511717	0.876148	-2.984644
22	6	0	-3.341024	0.193563	0.005144
23	6	0	-3.719971	1.440869	-0.593560
24	6	0	-4.245357	-0.920013	-0.017589
25	6	0	-4.893250	1.466708	-1.363798
26	6	0	-5.399805	-0.809308	-0.808263
27	6	0	-5.701905	0.349921	-1.505768
28	1	0	-5.193295	2.375255	-1.867547
29	1	0	-6.086982	-1.640508	-0.886422
30	1	0	-6.592810	0.396603	-2.127223
31	6	0	3.595757	2.405744	0.991245
32	6	0	3.628992	-2.635873	-0.560387
33	6	0	-4.079988	-2.240228	0.806026
34	6	0	-2.958930	2.797385	-0.422572

35	6	0	−3.801373	3.993711	−0.933620
36	1	0	−3.270900	4.922002	−0.695454
37	1	0	−3.943357	3.977412	−2.019778
38	1	0	−4.785668	4.043260	−0.455203
39	6	0	−1.637116	2.838120	−1.228229
40	1	0	−1.202442	3.844211	−1.174437
41	1	0	−0.894928	2.134819	−0.850689
42	1	0	−1.821961	2.610098	−2.284794
43	6	0	−2.693534	3.095200	1.074009
44	1	0	−3.634750	3.115578	1.635760
45	1	0	−2.034047	2.363369	1.545037
46	1	0	−2.216777	4.077775	1.175802
47	6	0	−3.819526	−1.927090	2.300486
48	1	0	−3.773062	−2.862659	2.871396
49	1	0	−2.880952	−1.394577	2.467949
50	1	0	−4.632571	−1.319018	2.713972
51	6	0	−5.374807	−3.092308	0.788778
52	1	0	−5.601211	−3.497419	−0.203473
53	1	0	−5.238134	−3.947922	1.458840
54	1	0	−6.246359	−2.530726	1.142422
55	6	0	−2.967242	−3.156241	0.239761
56	1	0	−3.148400	−3.373397	−0.819677
57	1	0	−1.974441	−2.715894	0.333239
58	1	0	−2.963932	−4.111114	0.780489
59	6	0	2.336442	3.260974	0.706590
60	1	0	1.414831	2.687666	0.818647
61	1	0	2.297576	4.109906	1.401319
62	1	0	2.370253	3.663867	−0.312393
63	6	0	4.808142	3.375944	0.957102
64	1	0	5.762781	2.843444	1.032080
65	1	0	4.830901	3.999347	0.057916
66	1	0	4.736866	4.060693	1.809197
67	6	0	3.568767	1.857388	2.439825
68	1	0	2.665334	1.286268	2.665571
69	1	0	4.441000	1.223099	2.635863
70	1	0	3.592212	2.692333	3.150004
71	6	0	4.535814	−3.457044	−1.513474
72	1	0	5.582907	−3.139711	−1.472979
73	1	0	4.499782	−4.507948	−1.205595
74	1	0	4.197720	−3.415727	−2.554868
75	6	0	2.216759	−3.236706	−0.747291
76	1	0	1.465400	−2.822672	−0.082818
77	1	0	1.866152	−3.066171	−1.771241
78	1	0	2.261778	−4.320347	−0.582451
79	6	0	4.187428	−2.911274	0.860250
80	1	0	4.069655	−3.971702	1.116651
81	1	0	5.257253	−2.674271	0.895769
82	1	0	3.701079	−2.317383	1.631983

4B

opt freq b3lyp/6-31g(d)
E(RB3LYP) = -1968.06278369 A.U.
Dipole moment (field-independent basis, Debye):
X= -2.3505 Y= 0.7180 Z= -0.8323 Tot= 2.5948
Zero-point correction= 0.722731 (Hartree/Particle)
Sum of electronic and zero-point Energies= -1967.340053
Sum of electronic and thermal Energies= -1967.300093
Sum of electronic and thermal Enthalpies= -1967.299149
Sum of electronic and thermal Free Energies= -1967.408393

Standard orientation:

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms) X	Y	Z
1	15	0	0.815673	-0.520639	-0.765549
2	15	0	-0.704527	0.289654	0.731008
3	6	0	0.074212	0.091404	-2.375193
4	1	0	0.761560	-0.193595	-3.179459
5	1	0	-0.852101	-0.472796	-2.518047
6	1	0	-0.150171	1.153832	-2.470823
7	6	0	-0.168373	-0.577450	2.306460
8	8	0	0.997804	-0.898045	2.455567
9	6	0	-1.143493	-0.627927	3.476450
10	1	0	-2.178896	-0.782593	3.162134
11	1	0	-0.831967	-1.425122	4.157212
12	1	0	-1.091757	0.324188	4.018977
13	6	0	2.393831	0.398359	-0.263635
14	6	0	2.572853	1.810161	-0.034205
15	6	0	3.386502	-0.508403	0.243461
16	6	0	3.465684	2.196073	0.973483
17	6	0	4.263545	-0.041077	1.234513
18	6	0	4.244818	1.274821	1.661470
19	1	0	3.575197	3.242648	1.224582
20	1	0	4.971316	-0.719924	1.691532
21	1	0	4.892841	1.600778	2.471092
22	6	0	-2.365970	-0.280198	0.046433
23	6	0	-3.275722	0.809149	-0.207712
24	6	0	-2.603746	-1.543133	-0.604507
25	6	0	-4.146686	0.702835	-1.303353
26	6	0	-3.500531	-1.561013	-1.686272
27	6	0	-4.213613	-0.440921	-2.081247
28	1	0	-4.789819	1.537363	-1.558463
29	1	0	-3.649193	-2.483533	-2.235986
30	1	0	-4.862481	-0.479263	-2.952423
31	6	0	3.651609	-1.961388	-0.282283
32	6	0	2.009423	2.949978	-0.940291
33	6	0	-2.093265	-2.973219	-0.223538

34	6	0	−3.521867	2.102170	0.647816
35	6	0	−5.026622	2.087060	1.051936
36	1	0	−5.252367	2.974876	1.654376
37	1	0	−5.705662	2.090472	0.196028
38	1	0	−5.256775	1.201873	1.656113
39	6	0	−3.238186	3.372540	−0.187534
40	1	0	−3.470824	4.268168	0.401560
41	1	0	−2.184653	3.424199	−0.476159
42	1	0	−3.839920	3.413998	−1.101084
43	6	0	−2.762529	2.237479	1.983320
44	1	0	−2.860118	1.336729	2.595839
45	1	0	−1.703734	2.457983	1.854077
46	1	0	−3.208771	3.063647	2.550534
47	6	0	−3.357605	−3.833896	0.071373
48	1	0	−3.050729	−4.842707	0.372026
49	1	0	−3.940540	−3.399352	0.891641
50	1	0	−4.020680	−3.935190	−0.791664
51	6	0	−1.308826	−3.621147	−1.390439
52	1	0	−0.379554	−3.077322	−1.585638
53	1	0	−1.045649	−4.653578	−1.129954
54	1	0	−1.887389	−3.654790	−2.319046
55	6	0	−1.227989	−3.083376	1.038036
56	1	0	−0.253205	−2.614574	0.925319
57	1	0	−1.736719	−2.665241	1.910720
58	1	0	−1.050098	−4.144450	1.249752
59	6	0	2.810508	−3.036462	0.444109
60	1	0	1.751353	−2.951996	0.204837
61	1	0	3.152668	−4.036630	0.146784
62	1	0	2.918816	−2.944619	1.529832
63	6	0	5.140472	−2.346066	−0.057360
64	1	0	5.825884	−1.578187	−0.432812
65	1	0	5.376422	−2.535069	0.994600
66	1	0	5.349264	−3.276550	−0.596991
67	6	0	3.447481	−2.051137	−1.814650
68	1	0	2.409706	−1.890624	−2.114530
69	1	0	4.074788	−1.318397	−2.335776
70	1	0	3.732307	−3.050449	−2.165531
71	6	0	2.711524	4.305816	−0.669628
72	1	0	3.800999	4.232692	−0.750271
73	1	0	2.371696	5.029940	−1.418980
74	1	0	2.461798	4.721133	0.312857
75	6	0	0.505372	3.235435	−0.760162
76	1	0	−0.129357	2.375588	−0.956721
77	1	0	0.299092	3.555116	0.267131
78	1	0	0.200258	4.045974	−1.434674
79	6	0	2.352886	2.604061	−2.411486
80	1	0	1.929373	3.356410	−3.088805
81	1	0	3.440090	2.599120	−2.551600
82	1	0	1.983897	1.625800	−2.712748

#p casscf(2,2)/6-31g(d)

```
    ( 1)        EIGENVALUE      -1958.0331008043
(       1) 0.9999974 (       3)-0.0022851 (       2)-0.0000023 (
Final one electron symbolic density matrix:
                          1                   2
       1    0.199999D+01
       2 -0.651500D-05    0.104431D-04
MCSCF converged.
```

SP Calculation for 3 (X-ray)

b3lyp/6-31g(d)
E(RB3LYP) = -2591.47150581 A.U.
Dipole moment (field-independent basis, Debye):
X= 0.1814 Y= 0.3223 Z= 0.9734 Tot= 1.0413

Standard orientation:

Center Number	Atomic Number	Atomic Type	Coordinates (Angstroms) X	Y	Z
1	15	0	-1.661000	1.090734	-0.207279
2	15	0	1.051850	-0.117762	-0.947633
3	8	0	-0.501856	0.821644	0.981064
4	6	0	-3.027166	-0.165714	-0.070852
5	6	0	-3.878525	-0.132792	-1.227392
6	6	0	-3.469713	-0.892270	1.086154
7	6	0	-4.773817	-1.362036	1.104446
8	6	0	4.819632	-1.734368	-0.761357
9	6	0	2.677913	-2.920891	-1.378802
10	6	0	3.733539	0.821579	-1.026018
11	6	0	-2.546744	2.461526	0.646920
12	6	0	5.076935	0.620009	-0.682389
13	6	0	-5.182723	-0.625338	-1.103544
14	6	0	3.466293	-1.619861	-1.064081
15	6	0	0.794533	0.429359	0.642840
16	6	0	-2.292200	-0.063626	3.204690
17	6	0	-2.143988	-0.234480	-3.108713
18	6	0	-5.671549	-1.203122	0.050976
19	6	0	-3.244329	-2.325678	3.206087
20	6	0	1.656916	0.565909	1.846715
21	6	0	1.366451	1.550719	2.800372
22	6	0	5.624257	-0.632028	-0.477365
23	6	0	2.125715	1.670404	3.960347
24	6	0	7.060447	-0.849484	0.014791
25	6	0	-3.476493	0.384490	-2.647167
26	6	0	6.992623	-1.513299	1.396125
27	6	0	2.726050	-0.300449	2.091504
28	6	0	3.176845	0.795785	4.197160
29	6	0	-3.418235	1.920591	-2.731264
30	6	0	3.315512	2.277261	-1.394310
31	6	0	-1.764167	3.773887	0.563033
32	6	0	-7.100064	-1.744294	0.173561
33	6	0	-2.570879	-1.274082	2.298549
34	6	0	2.886741	-0.311874	-1.048924
35	6	0	3.467296	-0.183750	3.266659
36	6	0	2.869760	3.064298	-0.153325
37	6	0	2.207987	2.328333	-2.468340
38	6	0	1.814895	-2.746339	-2.650710
39	6	0	-7.948872	-1.420756	-1.062062
40	6	0	-7.045092	-3.267111	0.337361
41	6	0	1.813424	-3.341554	-0.176706

42	6	0	7.825771	0.467060	0.172649
43	6	0	4.516613	3.025263	-2.002161
44	6	0	-1.278094	-1.940379	1.794383
45	6	0	-2.644056	5.006847	0.774928
46	6	0	7.832729	-1.747273	-0.949842
47	6	0	3.620779	-4.096761	-1.676391
48	6	0	-4.505448	-0.052263	-3.714253
49	6	0	-7.787859	-1.125025	1.395700
50	6	0	-3.584226	5.266238	-0.383862
51	1	0	5.637822	1.346389	-0.594984
52	1	0	5.197240	-2.629791	-0.719142
53	1	0	0.627625	2.155049	2.643821
54	1	0	-1.788681	0.635743	2.799838
55	1	0	-5.060177	-1.850460	1.836749
56	1	0	-2.666030	2.317807	-2.222730
57	1	0	-2.538858	-2.636517	3.908049
58	1	0	-3.379827	2.524710	0.159542
59	1	0	-4.026301	-1.953447	3.690767
60	1	0	3.703236	0.894798	5.041601
61	1	0	-5.786770	-0.550825	-1.869669
62	1	0	2.930054	-0.985016	1.490714
63	1	0	4.108220	-4.389963	-0.858611
64	1	0	8.771791	-1.862891	-0.642373
65	1	0	-3.467899	-3.096850	2.695762
66	1	0	-3.334160	2.197419	-3.688510
67	1	0	3.105714	-4.827500	-1.977067
68	1	0	7.897417	-1.365410	-1.841721
69	1	0	-4.223162	2.373497	-2.367850
70	1	0	4.228277	-3.898797	-2.400704
71	1	0	-1.784191	-0.328496	3.939674
72	1	0	7.353472	1.062959	0.795115
73	1	0	4.204582	-0.812616	3.402197
74	1	0	-3.155300	0.329723	3.501570
75	1	0	7.436038	-2.623541	-1.030719
76	1	0	-2.770928	2.253270	1.544491
77	1	0	1.931642	2.353552	4.614413
78	1	0	8.781832	0.271303	0.445009
79	1	0	7.962098	0.943265	-0.694298
80	1	0	-1.419899	0.073125	-2.541688
81	1	0	-0.875583	-1.474115	1.066078
82	1	0	6.569361	-2.415214	1.377218
83	1	0	7.909429	-1.666048	1.743394
84	1	0	-1.451070	-2.860537	1.469362
85	1	0	2.767708	4.015542	-0.369605
86	1	0	-5.336504	0.446952	-3.614912
87	1	0	-4.161605	0.208467	-4.581209
88	1	0	6.497521	-0.911648	2.070127
89	1	0	2.462337	-3.486813	0.649186
90	1	0	3.545360	3.002439	0.596401
91	1	0	1.362412	2.101785	-2.112092
92	1	0	2.054615	2.721451	0.196013
93	1	0	-1.944299	0.048677	-4.029324
94	1	0	-8.699048	-1.521097	1.531498
95	1	0	1.157624	-2.708925	0.032790

96	1	0	−7.251813	−1.335832	2.240342
97	1	0	2.401721	1.691160	−3.213899
98	1	0	5.242324	3.204638	−1.349863
99	1	0	2.147563	3.244008	−2.823498
100	1	0	1.126134	−2.101442	−2.602698
101	1	0	1.382184	−4.222072	−0.431974
102	1	0	−6.569263	−3.646790	−0.436287
103	1	0	2.432224	−2.519573	−3.386675
104	1	0	−2.216847	−1.195752	−3.100576
105	1	0	−7.995349	−0.426481	−1.226442
106	1	0	4.234110	3.931679	−2.353707
107	1	0	−6.545071	−3.506599	1.186961
108	1	0	−7.554452	−1.843116	−1.951278
109	1	0	4.949998	2.527944	−2.690299
110	1	0	−7.972524	−3.638644	0.432749
111	1	0	−0.660938	−1.997476	2.498333
112	1	0	1.349624	−3.640920	−2.858227
113	1	0	−4.650227	−1.034228	−3.694104
114	1	0	−7.839188	−0.172776	1.324675
115	1	0	−8.829030	−1.774207	−0.967742
116	1	0	−1.060657	3.746838	1.200161
117	1	0	−3.184922	4.861632	1.619933
118	1	0	−1.323066	3.842792	−0.325282
119	1	0	−4.193258	4.545353	−0.554344
120	1	0	−2.073407	5.764579	0.884303
121	1	0	−3.063754	5.415212	−1.230859
122	1	0	−4.155640	6.074961	−0.195433

TD-SCF Calculation for 3

td=(nstates=10) wb97xd/6-31g(2d,p)

Excited State 1: Singlet-A 3.6360 eV 340.99 nm f=0.0533 <S**2>=0.000

194 -> 197 0.15657

196 -> 197 0.66249

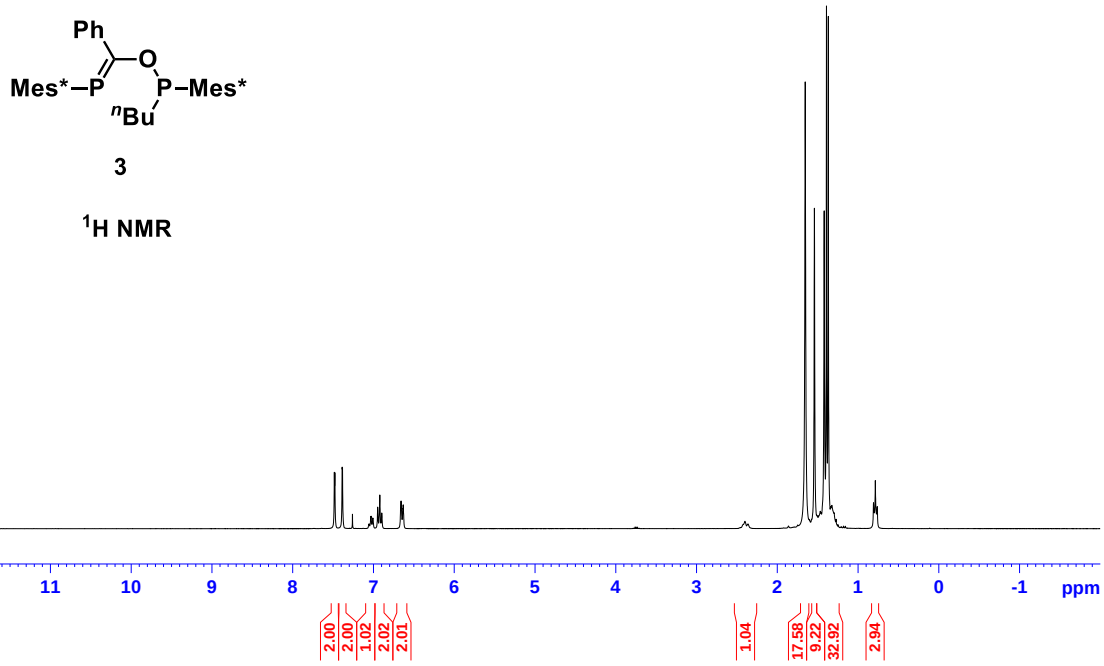
Excited State 2: Singlet-A 4.1617 eV 297.92 nm f=0.1715 <S**2>=0.000

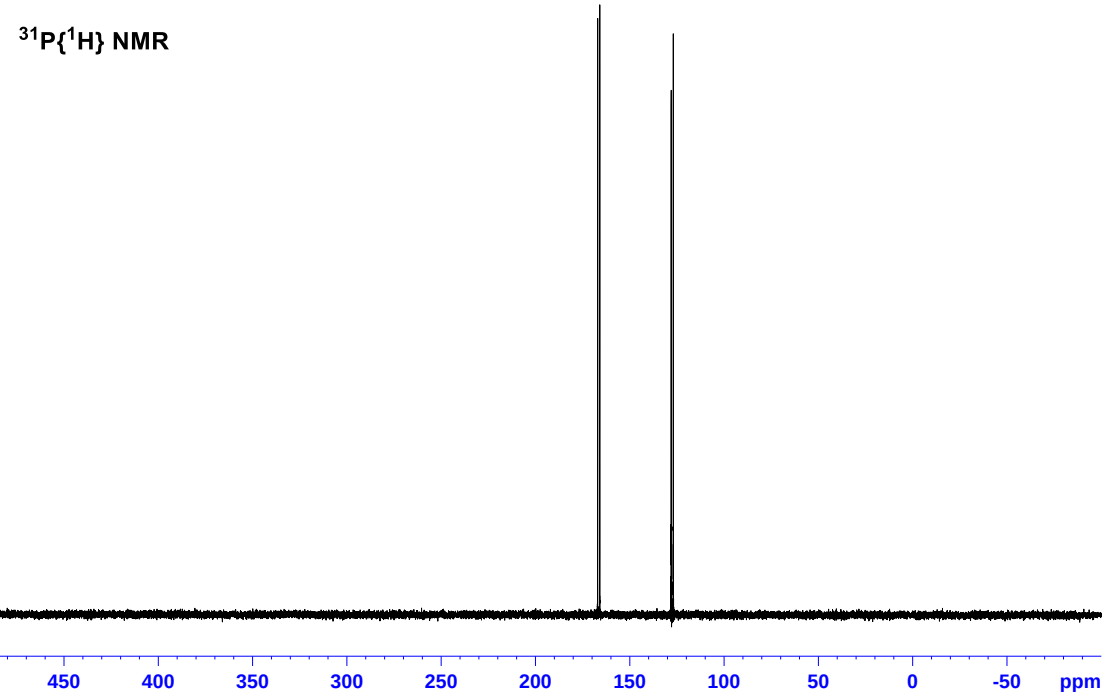
194 -> 197 -0.19521

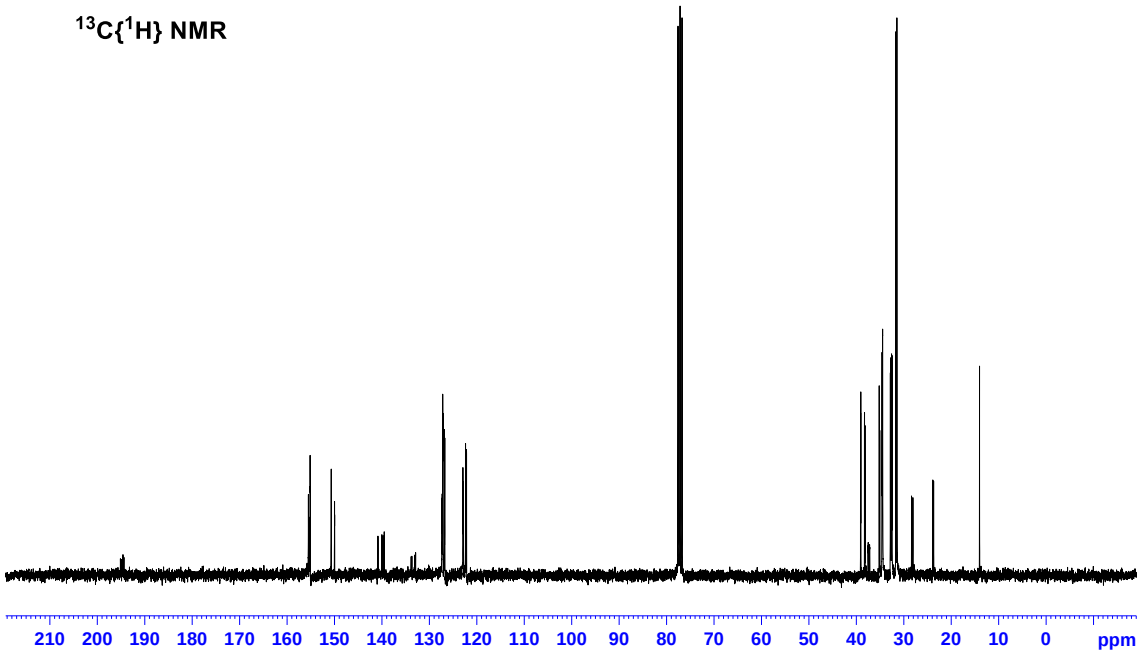
195 -> 197 0.63666

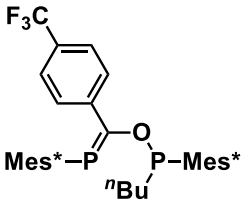
196 -> 197 0.12650

6. NMR Charts


Ph
O
Mes*–P
P–Mes*
nBu
3
1H NMR
11
10
9
8
7
6
5
4
3
2
1
0
-1
ppm
2.00
2.00
1.02
2.02
2.01
1.04
17.58
9.22
32.92
2.94


31P{1H} NMR
450
400
350
300
250
200
150
100
50
0
-50
ppm

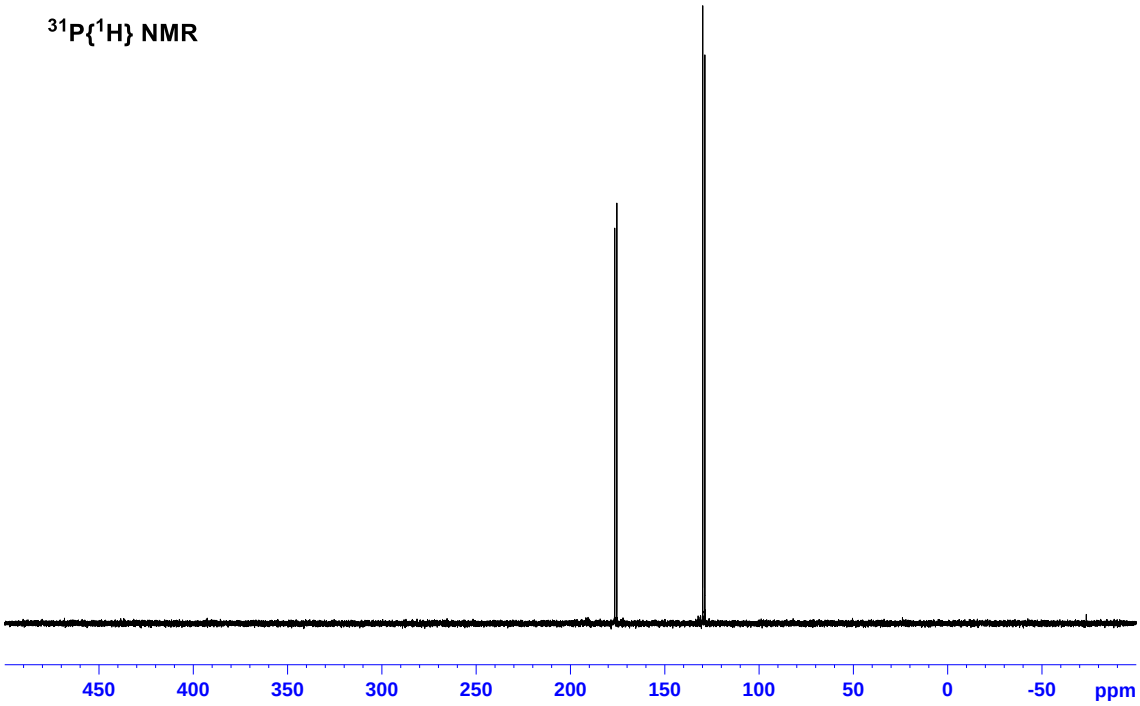

$^{13}C\{^{1}H\}$ NMR
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

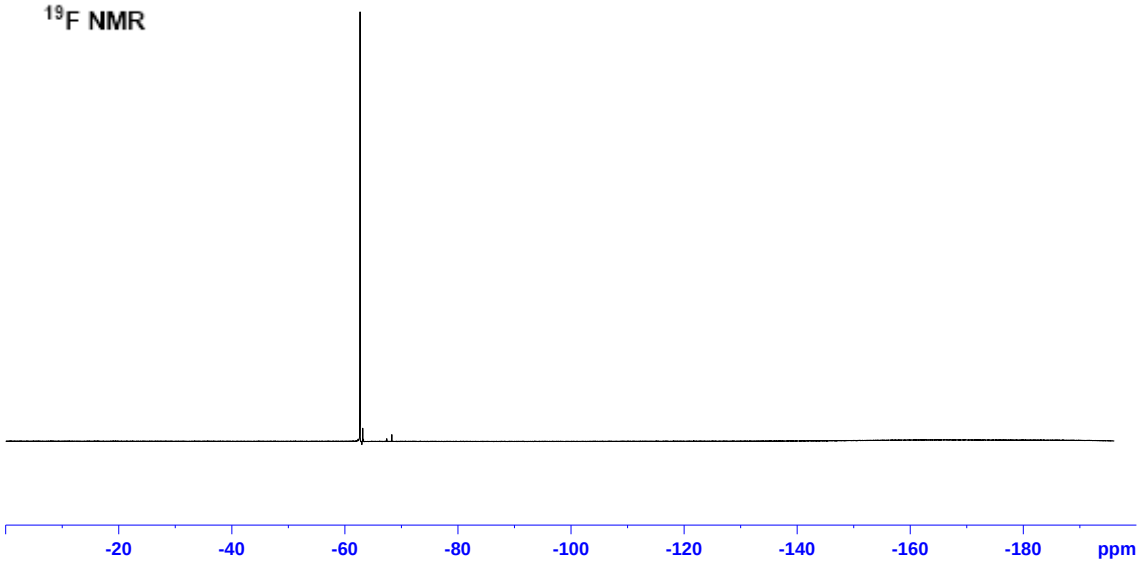


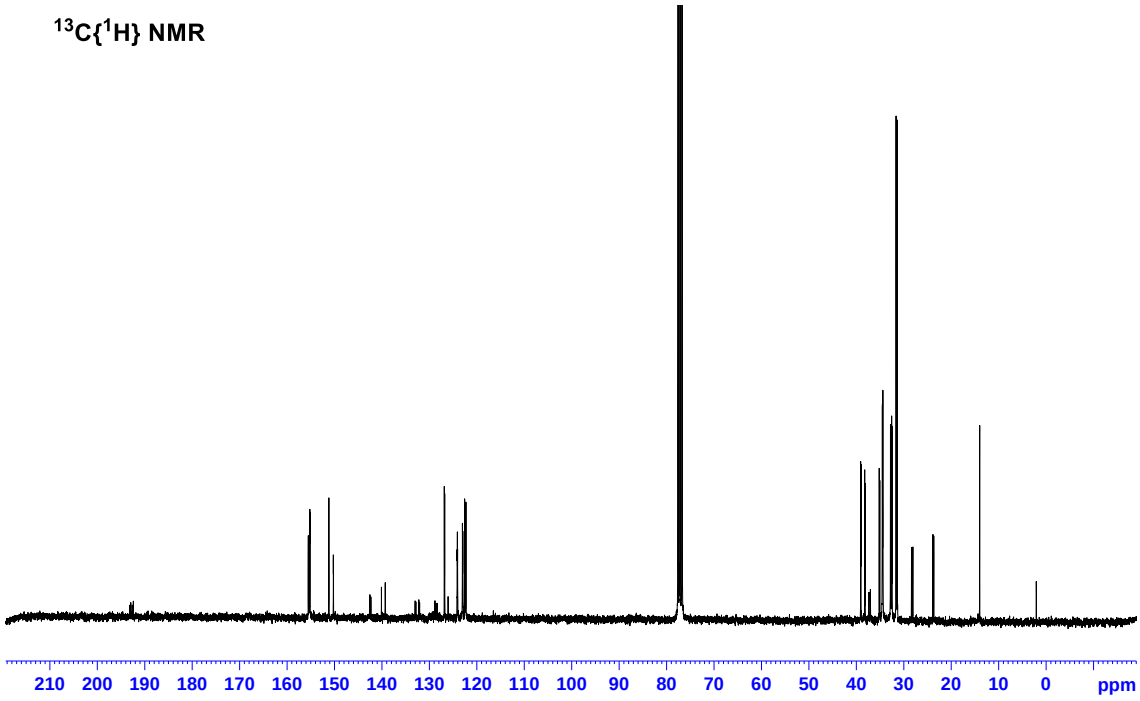
5

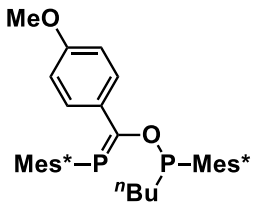
^{1}H NMR

^{31}P{^{1}H} NMR




^{19}F NMR
-20
-40
-60
-80
-100
-120
-140
-160
-180
ppm

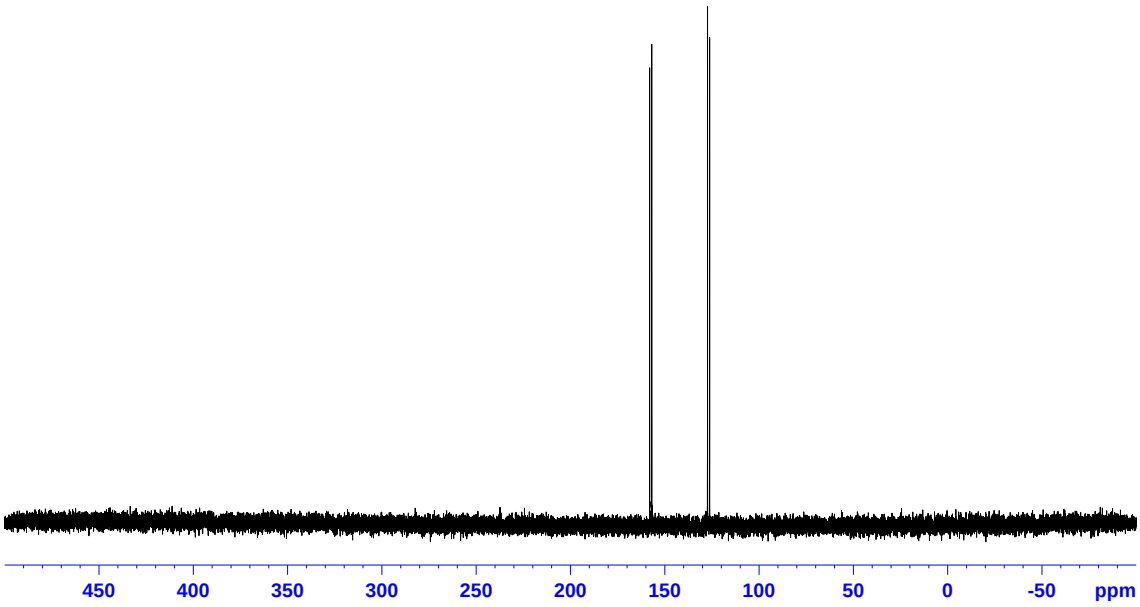

^{13}C{^{1}H} NMR
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm



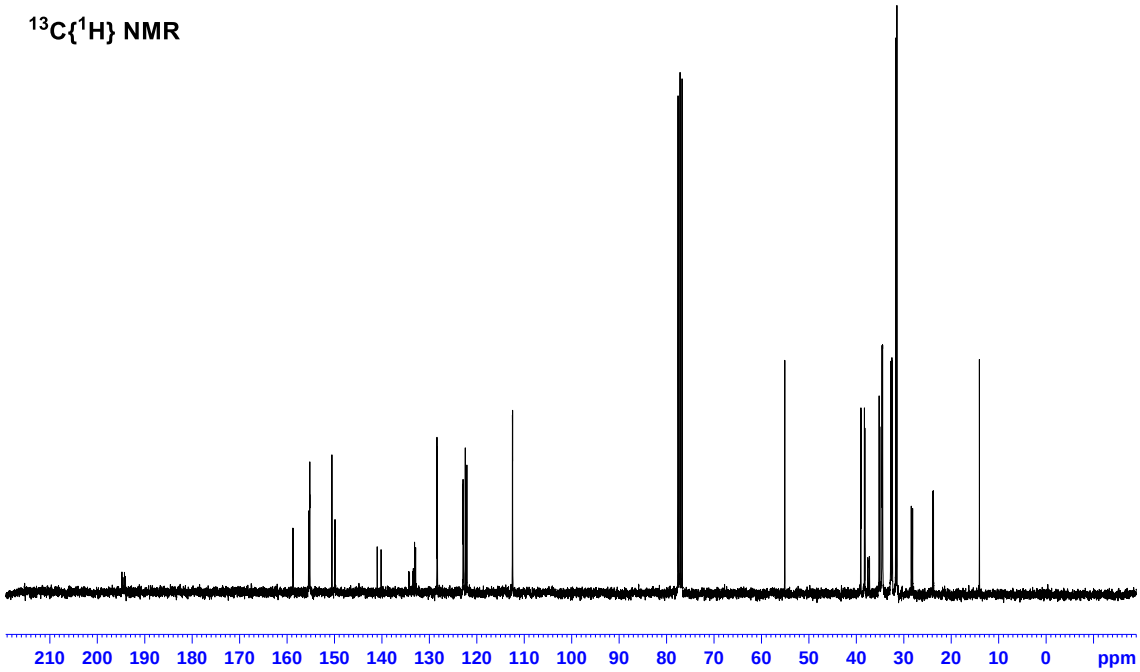
6

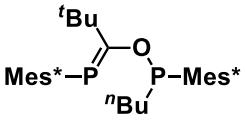
^{1}H NMR

^{31}P{^{1}H} NMR



$^{13}C\{^{1}H\}$ NMR

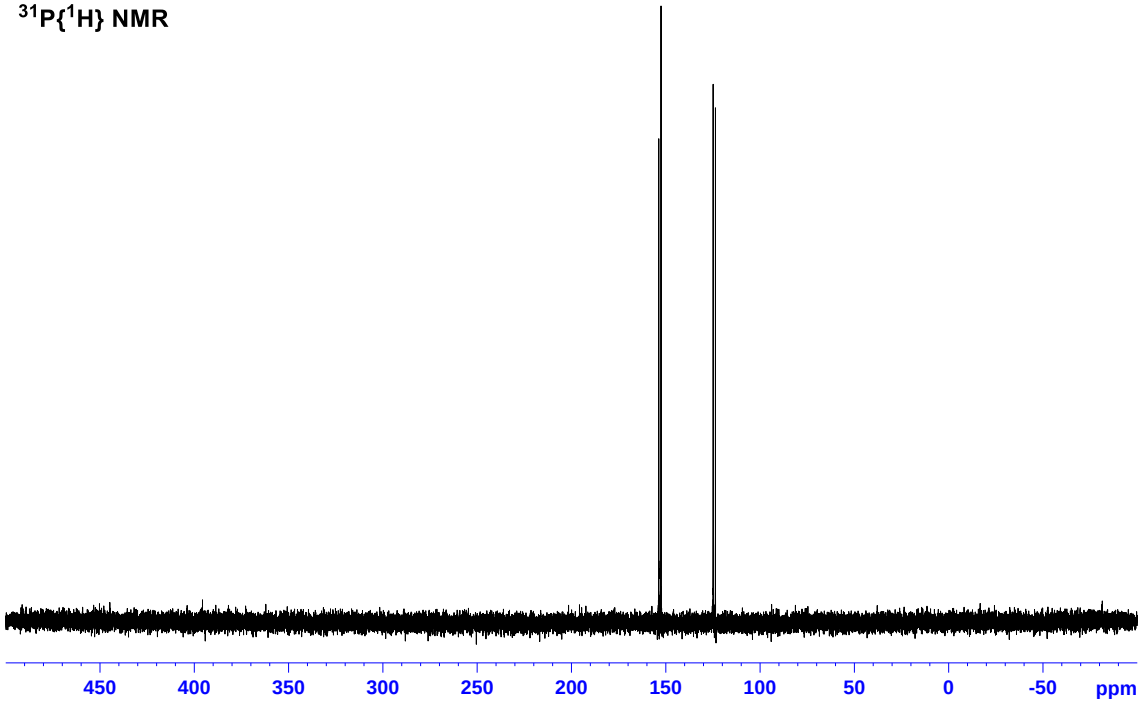




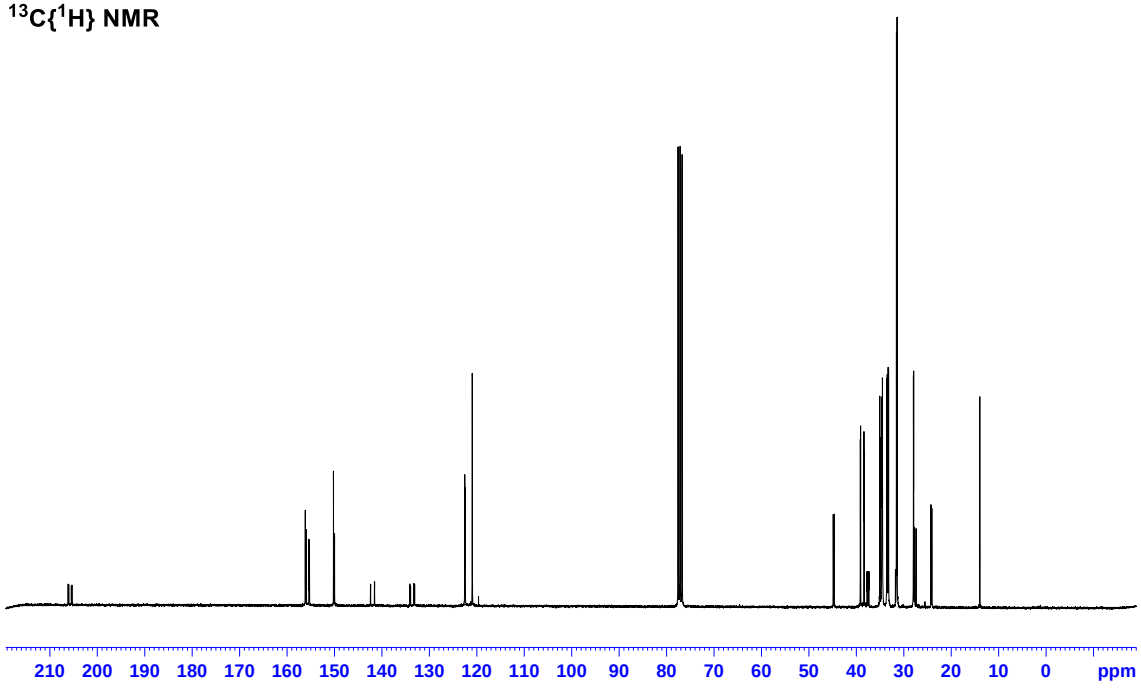
7

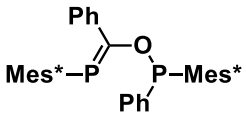
^{1}H NMR

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



$^{13}C\{^{1}H\}$ NMR

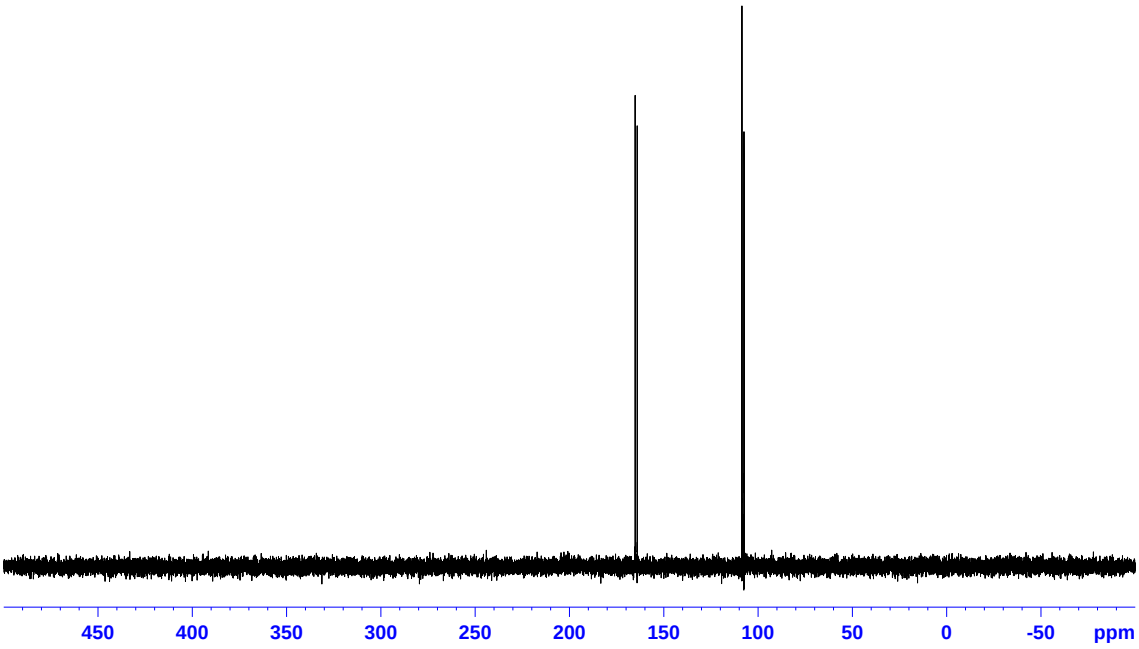




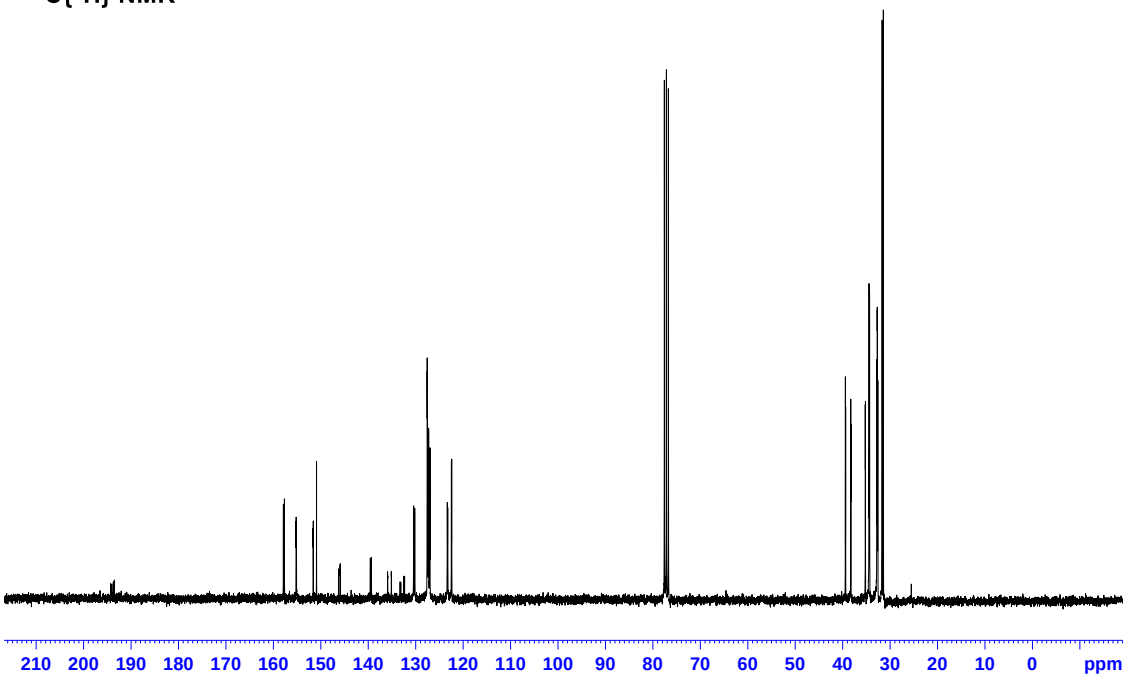
8

^{1}H NMR

^{31}P{^{1}H} NMR



$^{13}C\{^{1}H\}$ NMR


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

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