Supporting Information

Copper-Mediated Oxidative Decarboxylative Coupling of Arylpropiolic Acids with Dialkyl H-Phosphonates in Water

Xiang Li, Fan Yang,* Yangjie Wu, and Yusheng Wu*

The College of Chemistry and Molecular Engineering, Henan Key Laboratory of Chemical Biology and Organic Chemistry, Key Laboratory of Applied Chemistry of Henan Universities, Zhengzhou University, Zhengzhou 450052, People's Republic of China

E-mail: yangf@zzu.edu.cn; yusheng.wu@tetranovglobal.com

Contents

General Methods.

^{1}H, ^{13}C, and ^{31}P NMR spectra were recorded on a Bruker DPX-400 spectrometer with $CDCl_3$ as the solvent and TMS as an internal standard. Melting points were measured using a WC-1 microscopic apparatus and were uncorrected. GC analysis was performed on Agilent 4890D gas chromatograph. Mass spectra were measured on an LC-MSD-Trap-XCT instrument. High-resolution mass spectra were measured on a MALDI-FTMS. Ethyl acetate and hexane (analytical grade) were used for column chromatography without purification. The other chemicals were bought from commercial sources and used as-received unless otherwise noted.

Typical Procedure for the Copper-Mediated Oxidative Decarboxylative Coupling of Arylpropiolic Acids with Dialkyl H-Phosphonates in Water: Arylpropiolic acid (0.2 mmol), H-phosphonate (0.4 mmol), $Cu(OAc)_2 \cdot H_2O$ (2.0 equiv), 1,10-phenanthroline (2.5 equiv), K_3PO_4 (2.0 equiv), and $^{i}PrOH$ (3.0 equiv) were added to a 10 mL round-bottomed flask and then H_2O (2.5 mL) was added. The reaction mixture was stirred at 60 ^{o}C for 24 h. After the reaction was complete, the mixture was added into H_2O (25 mL) and extracted with CH_2Cl_2 three times. The combined organic layer was dried with anhydrous Na_2SO_4 and evaporated in vacuum. The crude product was purified by flash chromatography on silica gel using hexane/ethyl acetate as the eluent to give the pure product.

^{1}H NMR, ^{13}C NMR, and ^{31}P NMR data of all products

Diisopropyl (phenylethynyl)phosphonate (**3a**):[1] colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.56−7.54 (m, 2H), 7.47−7.43 (m, 1H), 7.39−7.35 (m, 2H), 4.86−4.78 (m, 2H), 1.41 (d, J = 6.2 Hz, 6H), 1.40 (d, J = 6.2 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 132.5 (d, $J_{C\text{-}P}$ = 2.4 Hz), 130.5, 128.6, 119.8 (d, $J_{C\text{-}P}$ = 5.6 Hz), 98.2 (d, $J_{C\text{-}P}$ = 52.7 Hz), 79.8 (d, $J_{C\text{-}P}$ = 296.9 Hz), 72.4 (d, $J_{C\text{-}P}$ = 5.6 Hz), 23.9 (d, $J_{C\text{-}P}$ = 4.6 Hz), 23.6 (d, $J_{C\text{-}P}$ = 4.8 Hz); ^{31}P NMR ($CDCl_3$, 163 MHz): δ -8.08; HRMS (ESI^+) calcd for $C_{14}H_{19}O_3P$ $[M+H]^+$: 267.1145, found: 267.1147.

Dimethyl (phenylethynyl)phosphonate (**3b**):[2] colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.50 (d, J = 7.2 Hz, 2H), 7.42−7.36 (m, 1H), 7.35−7.27 (m, 2H), 3.80 (s, 3H), 3.77 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 132.7 (d, $J_{C\text{-}P}$ = 2.4 Hz), 130.9, 128.6, 119.3 (d, $J_{C\text{-}P}$ = 5.6 Hz), 100.1 (d, $J_{C\text{-}P}$ = 53.0 Hz), 76.8 (d, $J_{C\text{-}P}$ = 301.1 Hz), 53.5 (d, $J_{C\text{-}P}$ = 5.5 Hz); ^{31}P NMR ($CDCl_3$, 163 MHz): δ -2.28; HRMS (ESI^+) calcd for $C_{10}H_{11}O_3P$ $[M+H]^+$: 211.0519, found: 211.0520.

Diethyl (phenylethynyl)phosphonate (**3c**):[1] colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.54 (d, J = 7.6 Hz, 2H), 7.46−7.40 (m, 1H), 7.39−7.31 (m, 2H), 4.27−4.15 (m, 4H), 1.38 (t, J = 7.0 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 132.7 (d, $J_{C\text{-}P}$ = 2.4 Hz), 130.7, 128.6, 119.6 (d, $J_{C\text{-}P}$ = 5.6 Hz), 99.1 (d, $J_{C\text{-}P}$ = 52.7 Hz), 78.4 (d, $J_{C\text{-}P}$ = 297.1 Hz), 63.3 (d, $J_{C\text{-}P}$ = 5.4 Hz), 16.2 (d, $J_{C\text{-}P}$ = 7.0 Hz); ^{31}P NMR ($CDCl_3$, 163 MHz): δ -5.52; HRMS (ESI^+) calcd for $C_{12}H_{15}O_3P$ $[M+H]^+$: 239.0832, found: 239.0836.

Dipropyl (phenylethynyl)phosphonate (**3d**): colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.51 (d, J = 7.6 Hz, 2H), 7.41 (t, J = 7.2 Hz, 1H), 7.33 (t, J = 7.6 Hz, 2H), 4.12−4.03 (m, 4H), 1.79−1.65 (m, 4H), 0.96 (t, J = 7.4 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 132.6 (d, $J_{C\text{-}P}$ = 2.4 Hz), 130.7, 128.6, 119.6 (d, $J_{C\text{-}P}$ = 5.6 Hz), 99.1 (d, $J_{C\text{-}P}$ = 52.4 Hz), 78.3 (d, $J_{C\text{-}P}$ = 301.5 Hz), 68.7 (d, $J_{C\text{-}P}$ = 5.9 Hz), 23.6 (d, $J_{C\text{-}P}$ = 7.0 Hz), 10.1; ^{31}P NMR ($CDCl_3$, 163 MHz): δ -5.14; HRMS (ESI^+) calcd for $C_{14}H_{19}O_3P$ $[M+H]^+$: 267.1145, found: 267.1150.

Dibutyl (phenylethynyl)phosphonate (**3e**):[1] colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.53 (d, J = 7.2 Hz, 2H), 7.46−7.39 (m, 1H), 7.38−7.31 (m, 2H), 4.16−4.07 (m, 4H), 1.73−1.66 (m, 4H), 1.47−1.38 (m, 4H), 0.92 (t, J = 7.4 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 132.6 (d, $J_{C\text{-}P}$ = 2.3 Hz), 130.7, 128.6, 119.6 (d, $J_{C\text{-}P}$ = 5.6 Hz), 99.1 (d, $J_{C\text{-}P}$ = 52.5 Hz), 79.8 (d, $J_{C\text{-}P}$ = 300.1 Hz), 66.9 (d, $J_{C\text{-}P}$ = 5.8 Hz), 32.2 (d, $J_{C\text{-}P}$ = 7.1 Hz), 18.7, 13.6; ^{31}P NMR ($CDCl_3$, 163 MHz): δ -5.14; HRMS (ESI^+) calcd for $C_{16}H_{23}O_3P$ $[M+H]^+$: 295.1458, found: 295.1462.

Dibenzyl (phenylethynyl)phosphonate (**3f**):[3] colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.46−7.32 (m, 15H), 5.18 (s, 2H), 5.16 (s, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 135.5 (d, $J_{C\text{-}P}$ = 7.2 Hz), 132.7 (d, $J_{C\text{-}P}$ = 2.4 Hz), 130.9, 128.6, 128.1, 127.6, 127.0, 119.3 (d, $J_{C\text{-}P}$ = 5.6 Hz), 100.0 (d, $J_{C\text{-}P}$ = 53.9 Hz), 78.1 (d, $J_{C\text{-}P}$ = 304.0 Hz) , 68.6 (d, $J_{C\text{-}P}$ = 5.2 Hz); ^{31}P NMR ($CDCl_3$, 163 MHz): δ -5.00; HRMS (ESI^+) calcd for $C_{22}H_{19}O_3P$ $[M+H]^+$: 363.1145, found: 363.1149.

Diisopropyl (*p*-tolylethynyl)phosphonate (**3g**): colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.44 (d, J = 8.0 Hz, 2H), 7.17 (d, J = 7.6 Hz, 2H), 4.87−4.70 (m, 2H), 2.37 (s, 3H), 1.40 (d, J = 6.0 Hz, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 140.0, 131.4 (d, $J_{C\text{-}P}$ =

2.3 Hz), 128.3, 115.7 (d, $J_{C\text{-}P}$ = 5.5 Hz), 97.7 (d, $J_{C\text{-}P}$ = 52.8 Hz), 78.2 (d, $J_{C\text{-}P}$ = 297.5 Hz), 71.3 (d, $J_{C\text{-}P}$ = 5.6 Hz), 22.9 (d, $J_{C\text{-}P}$ = 4.6 Hz), 22.6 (d, $J_{C\text{-}P}$ = 4.8 Hz), 20.7; ^{31}P NMR ($CDCl_3$, 163 MHz): δ -7.76; HRMS (ESI^+) calcd for $C_{15}H_{21}O_3P$ $[M+H]^+$: 281.1301, found: 281.1305.

Diisopropyl ((4-ethylphenyl)ethynyl)phosphonate (**3h**):[4] colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.40 (d, J = 8.0 Hz, 2H), 7.13 (d, J = 8.0 Hz, 1H), 4.78−4.68 (m, 2H), 2.65−2.55 (m, 2H), 1.33 (d, J = 6.0 Hz, 12H), 1.16 (t, J = 8.0 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 147.4, 132.6 (d, $J_{C\text{-}P}$ = 2.3 Hz), 128.2, 116.9 (d, $J_{C\text{-}P}$ = 5.6 Hz), 98.8 (d, $J_{C\text{-}P}$ = 53.0 Hz), 79.2 (d, $J_{C\text{-}P}$ = 298.1 Hz), 72.3 (d, $J_{C\text{-}P}$ = 5.5 Hz), 29.0, 24.0 (d, $J_{C\text{-}P}$ = 4.6 Hz), 23.7 (d, $J_{C\text{-}P}$ = 4.9 Hz), 15.2; ^{31}P NMR ($CDCl_3$, 163 MHz): δ -7.77; HRMS (ESI^+) calcd for $C_{16}H_{23}O_3P$ $[M+H]^+$: 295.1458, found: 295.1462.

Diisopropyl ((4-methoxyphenyl)ethynyl)phosphonate (**3i**):[5] colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.48 (d, J = 8.0 Hz, 2H), 6.86 (d, J = 8.0 Hz, 1H), 4.84−4.74 (m, 2H), 3.82 (s, 3H), 1.39 (d, J = 6.0 Hz, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 161.3, 134.3 (d, $J_{C\text{-}P}$ = 2.4 Hz), 114.2, 111.7 (d, $J_{C\text{-}P}$ = 5.8 Hz), 98.9 (d, $J_{C\text{-}P}$ = 53.5 Hz), 78.5 (d, $J_{C\text{-}P}$ = 299.4 Hz), 72.2 (d, $J_{C\text{-}P}$ = 5.4 Hz), 55.4, 24.0 (d, $J_{C\text{-}P}$ = 4.5 Hz), 23.7 (d, $J_{C\text{-}P}$ = 4.8 Hz); ^{31}P NMR ($CDCl_3$, 163 MHz): δ -7.50; HRMS (ESI^+) calcd for $C_{15}H_{21}O_4P$ $[M+H]^+$: 297.1250, found: 297.1255.

Diisopropyl ((4-(*tert*-butyl)phenyl)ethynyl)phosphonate (**3j**): colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.48 (d, J = 8.0 Hz, 2H), 7.38 (d, J = 8.4 Hz, 1H), 4.86−4.73 (m, 2H), 1.39 (d, J = 6.0 Hz, 12H), 1.30 (s, 9H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 154.2, 132.4 (d, $J_{C\text{-}P}$ = 2.4 Hz), 125.6, 116.7 (d, $J_{C\text{-}P}$ = 5.5 Hz), 98.7 (d, $J_{C\text{-}P}$ = 53.0 Hz), 79.2 (d, $J_{C\text{-}P}$ = 297.7 Hz), 72.3 (d, $J_{C\text{-}P}$ = 5.4 Hz), 35.1, 31.1, 24.0 (d, $J_{C\text{-}P}$ = 4.5 Hz), 23.7 (d, $J_{C\text{-}P}$ = 4.9 Hz); ^{31}P NMR ($CDCl_3$, 163 MHz): δ -7.78; HRMS (ESI^+) calcd for $C_{18}H_{27}O_3P$ $[M+H]^+$: 323.1771, found: 323.1775.

Diisopropyl (naphthalen-1-ylethynyl)phosphonate (**3k**): colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.27 (d, J = 8.4 Hz, 1H), 7.93 (d, J = 8.0 Hz, 1H), 7.87 (d, J = 8.0 Hz, 1H), 7.80 (d, J = 6.8 Hz, 1H), 7.64−7.52 (m, 2H), 7.45 (t, J = 7.7 Hz, 1H), 4.95−4.82 (m, 2H), 1.46−1.41 (m, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 133.4 (d, $J_{C\text{-}P}$ = 1.8 Hz), 133.0, 132.6 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.2, 128.6, 127.7, 127.0, 125.6, 125.1, 117.4 (d, $J_{C\text{-}P}$ = 5.7 Hz), 96.6 (d, $J_{C\text{-}P}$ = 52.3 Hz), 84.5 (d, $J_{C\text{-}P}$ = 295.5 Hz), 72.5 (d, $J_{C\text{-}P}$ = 5.5 Hz), 24.0 (d, $J_{C\text{-}P}$ = 4.5 Hz), 23.8 (d, $J_{C\text{-}P}$ = 4.7 Hz); ^{31}P NMR ($CDCl_3$, 163 MHz): δ -8.14; HRMS (ESI^+) calcd for $C_{18}H_{21}O_3P$ $[M+H]^+$: 317.1301, found: 317.1306.

Diisopropyl (phenanthren-9-ylethynyl)phosphonate (**3l**): colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.73−8.64 (m, 2H), 8.40−8.34 (m, 1H), 8.16 (s, 1H), 7.89 (d, J = 7.9 Hz, 1H), 7.77−7.67 (m, 3H), 7.66−7.60 (m, 1H), 4.99−4.86 (m, 2H), 1.49−1.46 (m, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 133.9 (d, $J_{C\text{-}P}$ = 2.9 Hz), 130.1, 129.5, 128.9, 128.0, 127.7, 126.6, 126.5, 126.3, 125.4, 121.9, 121.7, 115.3 (d, $J_{C\text{-}P}$ = 5.6 Hz), 95.7 (d, $J_{C\text{-}P}$ = 52.3 Hz), 83.1 (d, $J_{C\text{-}P}$ = 295.6 Hz), 71.5 (d, $J_{C\text{-}P}$ = 5.5 Hz), 23.0 (d, $J_{C\text{-}P}$ = 4.6 Hz), 22.8 (d, $J_{C\text{-}P}$ = 4.9 Hz); ^{31}P NMR ($CDCl_3$, 163 MHz): δ -8.19; HRMS (ESI^+) calcd for $C_{18}H_{27}O_3P$ $[M+H]^+$: 317.1301, found: 317.1306; HRMS (ESI^+) calcd for $C_{22}H_{23}O_3P$ $[M+H]^+$: 367.1458, found: 367.1461.

Diisopropyl ((2-chlorophenyl)ethynyl)phosphonate (**3m**): colorless oil; ^{1}H NMR

($CDCl_3$, 400 MHz): δ 7.56 (d, J = 8.0 Hz, 1H), 7.42 (d, J = 8.0 Hz, 1H), 7.39−7.33 (m, 1H), 7.29−7.23 (m, 1H), 4.88−4.77 (m, 2H), 1.41 (d, J = 6.4 Hz, 6H), 1.40 (d, J = 6.4 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 137.0 (d, $J_{C\text{-}P}$ = 2.5 Hz), 134.3 (d, $J_{C\text{-}P}$ = 2.3 Hz), 131.5, 129.6, 126.7, 120.2 (d, $J_{C\text{-}P}$ = 5.6 Hz), 94.3 (d, $J_{C\text{-}P}$ = 51.8 Hz), 84.7 (d, $J_{C\text{-}P}$ = 291.9 Hz), 72.7 (d, $J_{C\text{-}P}$ = 5.6 Hz), 24.0 (d, $J_{C\text{-}P}$ = 4.5 Hz), 23.7 (d, $J_{C\text{-}P}$ = 4.8 Hz); ^{31}P NMR ($CDCl_3$, 163 MHz): δ -8.92; HRMS (ESI^+) calcd for $C_{14}H_{18}ClO_3P$ $[M+H]^+$: 301.0755, found: 301.0758.

Diisopropyl ((4-bromophenyl)ethynyl)phosphonate (**3n**): colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.50 (d, J = 8.4 Hz, 2H), 7.39 (d, J = 8.4 Hz, 2H), 4.87−4.73 (m, 2H), 1.39 (d, J = 6.0 Hz, 6H), 1.38 (d, J = 6.0 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 132.8 (d, $J_{C\text{-}P}$ = 2.4 Hz), 130.9, 124.2, 117.7 (d, $J_{C\text{-}P}$ = 5.7 Hz), 95.7 (d, $J_{C\text{-}P}$ = 52.4 Hz), 80.1 (d, $J_{C\text{-}P}$ = 296.0 Hz), 71.5 (d, $J_{C\text{-}P}$ = 5.6 Hz), 22.9 (d, $J_{C\text{-}P}$ = 4.5 Hz), 22.6 (d, $J_{C\text{-}P}$ = 4.9 Hz); ^{31}P NMR ($CDCl_3$, 163 MHz): δ -8.46; HRMS (ESI^+) calcd for $C_{14}H_{18}BrO_3P$ $[M+H]^+$: 345.0250, found: 345.0251.

Diisopropyl ((4-fluorophenyl)ethynyl)phosphonate (**3o**): colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.55−7.50 (m, 2H), 7.06−7.02 (m, 2H), 4.85−4.73 (m, 2H), 1.38 (d, J = 6.4 Hz, 6H), 1.37 (d, J = 6.0 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 163.8 (d, $J_{C\text{-}F}$ = 251.8 Hz), 134.8 (dd, $J_{C\text{-}F}$ = 8.8 Hz, $J_{C\text{-}P}$ = 2.4 Hz), 116.1 (d, $J_{C\text{-}F}$ = 22.3 Hz), 115.9 (d, $J_{C\text{-}P}$ = 3.7 Hz), 97.0 (d, $J_{C\text{-}P}$ = 52.8 Hz), 79.8 (d, $J_{C\text{-}P}$ = 297.2 Hz), 72.4 (d, $J_{C\text{-}P}$ = 5.5 Hz), 23.9 (d, $J_{C\text{-}P}$ = 4.6 Hz), 23.7 (d, $J_{C\text{-}P}$ = 4.9 Hz); ^{31}P NMR ($CDCl_3$, 163 MHz): δ -8.25; HRMS (ESI^+) calcd for $C_{14}H_{18}FO_3P$ $[M+H]^+$: 285.1050, found: 285.1075.

Diisopropyl ((3-chloro-4-fluorophenyl)ethynyl)phosphonate (**3p**): colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.58 (d, J = 6.8 Hz, 1H), 7.45−7.40 (m, 1H), 7.16−7.12 (m, 1H), 4.85−4.73 (m, 2H), 1.39 (d, J = 6.4 Hz, 12H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 159.4 (d, $J_{C\text{-}F}$ = 254.1 Hz), 134.7 (q, $J_{C\text{-}P}$ = 2.4 Hz), 132.7 (dd, $J_{C\text{-}F}$ = 7.8 Hz, $J_{C\text{-}P}$ = 2.4 Hz), 121.8 (d, $J_{C\text{-}F}$ = 1.9 Hz), 117.2 (d, $J_{C\text{-}F}$ = 22.0 Hz), 117.1 (d, $J_{C\text{-}P}$ = 5.6 Hz), 95.2 (d, $J_{C\text{-}P}$ = 52.4 Hz), 80.9 (d, $J_{C\text{-}P}$ = 295.1 Hz), 72.6 (d, $J_{C\text{-}P}$ = 5.6 Hz), 23.9 (d, $J_{C\text{-}P}$ = 4.5 Hz), 23.7 (d, $J_{C\text{-}P}$ = 4.8 Hz); ^{31}P NMR ($CDCl_3$, 163 MHz): δ -8.84; HRMS (ESI^+) calcd for $C_{14}H_{17}ClFO_3P$ $[M+H]^+$: 319.0661, found: 319.0664.

Diisopropyl ((4-acetylphenyl)ethynyl)phosphonate (**3q**): colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.93 (d, J = 8.4 Hz, 2H), 7.61 (d, J = 8.0 Hz, 1H), 4.85−4.74 (m, 2H), 2.59 (s, 3H), 1.39 (d, J = 6.4 Hz, 6H), 1.38 (d, J = 6.0 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 197.0, 138.0, 132.7 (d, $J_{C\text{-}P}$ = 2.3 Hz), 128.3, 124.4 (d, $J_{C\text{-}P}$ = 5.5 Hz), 96.5 (d, $J_{C\text{-}P}$ = 51.8 Hz), 82.7 (d, $J_{C\text{-}P}$ = 294.2 Hz), 72.7 (d, $J_{C\text{-}P}$ = 5.6 Hz), 26.7, 23.9 (d, $J_{C\text{-}P}$ = 4.6 Hz), 23.7 (d, $J_{C\text{-}P}$ = 4.8 Hz); ^{31}P NMR ($CDCl_3$, 163 MHz): δ -8.85; HRMS (ESI^+) calcd for $C_{16}H_{21}O_4P$ $[M+H]^+$: 309.1250, found: 309.1255.

Diisopropyl ((4-(trifluoromethyl)phenyl)ethynyl)phosphonate (**3r**): colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.68−7.60 (m, 4H), 4.87−4.74 (m, 2H), 1.40 (d, J = 6.0 Hz, 6H), 1.39 (d, J = 6.4 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 132.8 (d, $J_{C\text{-}P}$ = 2.4 Hz), 132.1 (q, $J_{C\text{-}F}$ = 32.8 Hz), 125.6 (q, $J_{C\text{-}F}$ = 3.8 Hz), 123.6 (d, $J_{C\text{-}P}$ = 5.5 Hz), 123.5 (q, $J_{C\text{-}F}$ = 270.9 Hz), 95.9 (d, $J_{C\text{-}P}$ = 51.9 Hz), 82.2 (d, $J_{C\text{-}P}$ = 294.1 Hz), 72.7 (d, $J_{C\text{-}P}$ = 5.6 Hz), 23.9 (d, $J_{C\text{-}P}$ = 4.6 Hz), 23.7 (d, $J_{C\text{-}P}$ = 4.9 Hz); ^{31}P NMR ($CDCl_3$, 163 MHz): δ -8.98; HRMS (ESI^+) calcd for $C_{15}H_{18}F_3O_3P$ $[M+H]^+$: 335.1018, found:

335.1021.

Diisopropyl ((3-nitrophenyl)ethynyl)phosphonate (**3s**): colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 8.37 (s, 1H), 8.29 (d, J = 8.8 Hz, 1H), 7.85 (d, J = 8.0 Hz, 1H), 7.59 (t, J = 8.0 Hz, 1H), 4.87−4.77 (m, 2H), 1.41 (d, J = 6.0 Hz, 6H), 1.40 (d, J = 6.0 Hz, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 148.1, 138.0 (d, $J_{C\text{-}P}$ = 2.2 Hz), 129.9, 127.3 (d, $J_{C\text{-}P}$ = 2.5 Hz), 125.2, 121.7 (d, $J_{C\text{-}P}$ = 5.7 Hz), 94.4 (d, $J_{C\text{-}P}$ = 51.6 Hz), 82.6 (d, $J_{C\text{-}P}$ = 292.6 Hz), 72.9 (d, $J_{C\text{-}P}$ = 5.6 Hz), 24.0 (d, $J_{C\text{-}P}$ = 4.5 Hz), 23.7 (d, $J_{C\text{-}P}$ = 4.8 Hz); ^{31}P NMR ($CDCl_3$, 163 MHz): δ -9.42; HRMS (ESI^+) calcd for $C_{14}H_{18}NO_5P$ $[M+H]^+$: 312.0995, found: 312.1001.

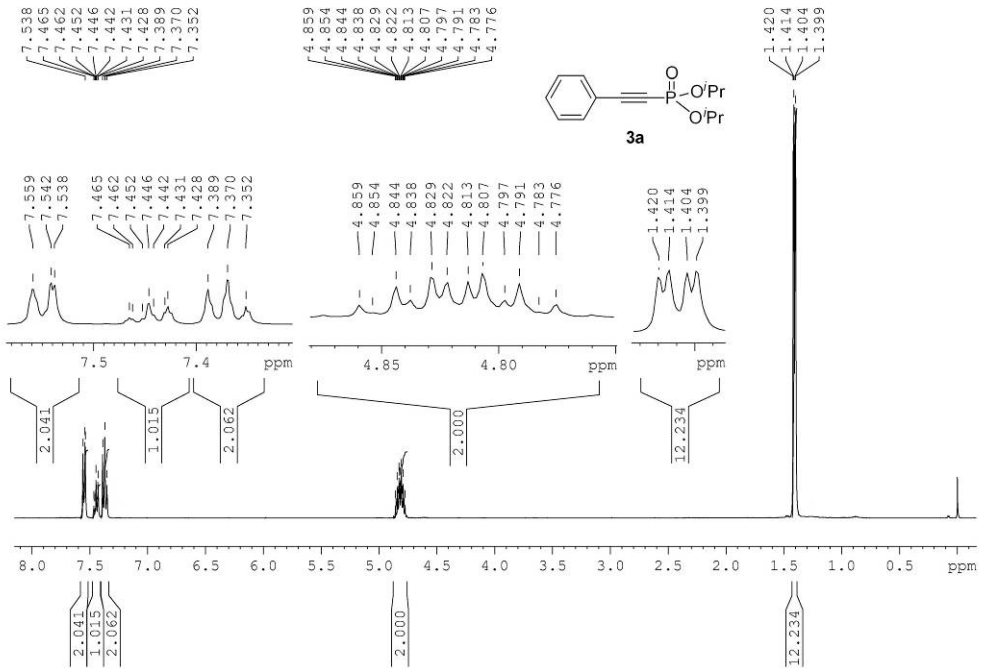
Diisopropyl (thiophen-3-ylethynyl)phosphonate (**3t**): colorless oil; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.72−7.70 (m, 1H), 7.35−7.31 (m, 1H), 7.22−7.20 (m, 1H), 4.88−4.73 (m, 2H), 1.42 (s, 6H), 1.40 (s, 6H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 133.0 (d, $J_{C\text{-}P}$ = 2.8 Hz), 129.9 (d, $J_{C\text{-}P}$ = 1.6 Hz), 126.1, 119.1 (d, $J_{C\text{-}P}$ = 6.0 Hz), 93.5 (d, $J_{C\text{-}P}$ = 53.7 Hz), 79.8 (d, $J_{C\text{-}P}$ = 297.7 Hz), 72.3 (d, $J_{C\text{-}P}$ = 5.6 Hz), 23.9 (d, $J_{C\text{-}P}$ = 4.4 Hz), 23.7 (d, $J_{C\text{-}P}$ = 4.8 Hz); ^{31}P NMR ($CDCl_3$, 163 MHz): δ -8.43; HRMS (ESI^+) calcd for $C_{12}H_{17}O_3PS$ $[M+H]^+$: 255.1146, found: 312.1149.

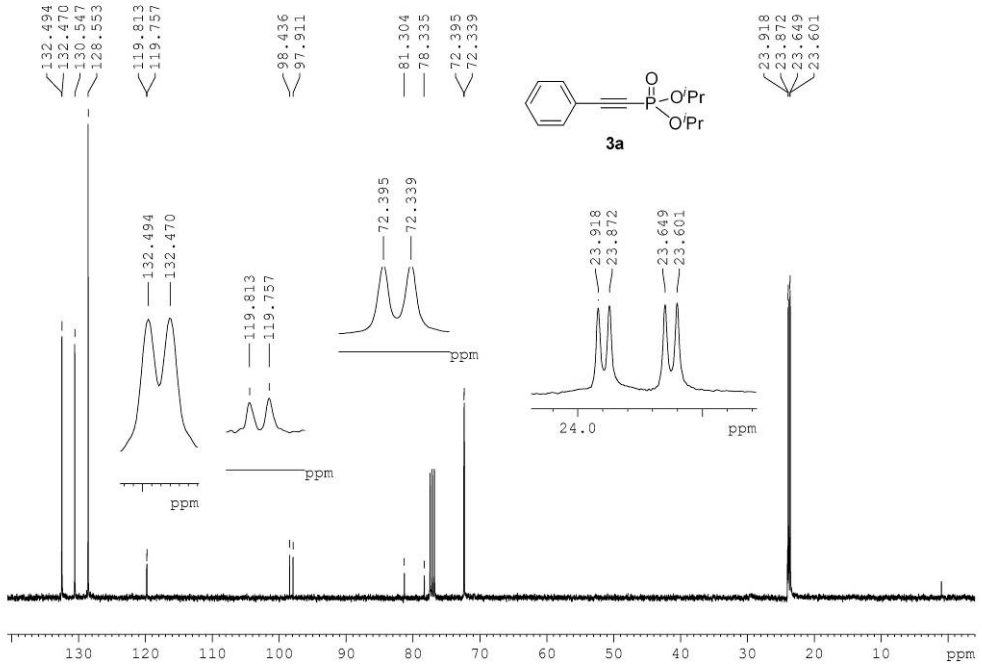
Diphenyl(phenylethynyl)phosphine oxide (**3v**):[5] white solid, mp 101−102 ^{o}C; ^{1}H NMR ($CDCl_3$, 400 MHz): δ 7.95−7.84 (m, 4H), 7.60−7.55 (m, 2H), 7.55−7.40 (m, 7H), 7.38−7.31 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 133.6, 132.6 (d, $J_{C\text{-}P}$ = 1.8 Hz), 132.4, 132.3 (d, $J_{C\text{-}P}$ = 1.9 Hz), 131.0 (d, $J_{C\text{-}P}$ = 11.1 Hz), 130.8, 128.7 (d, $J_{C\text{-}P}$ = 13.5 Hz), 119.9 (d, $J_{C\text{-}P}$ = 4.0 Hz), 105.5 (d, $J_{C\text{-}P}$ = 29.9 Hz), 82.9 (d, $J_{C\text{-}P}$ = 168.7 Hz); ^{31}P NMR ($CDCl_3$, 163 MHz): δ 8.87; HRMS (ESI^+) calcd for $C_{14}H_{18}NO_5P$ $[M+H]^+$: 303.0933, found: 303.0938.

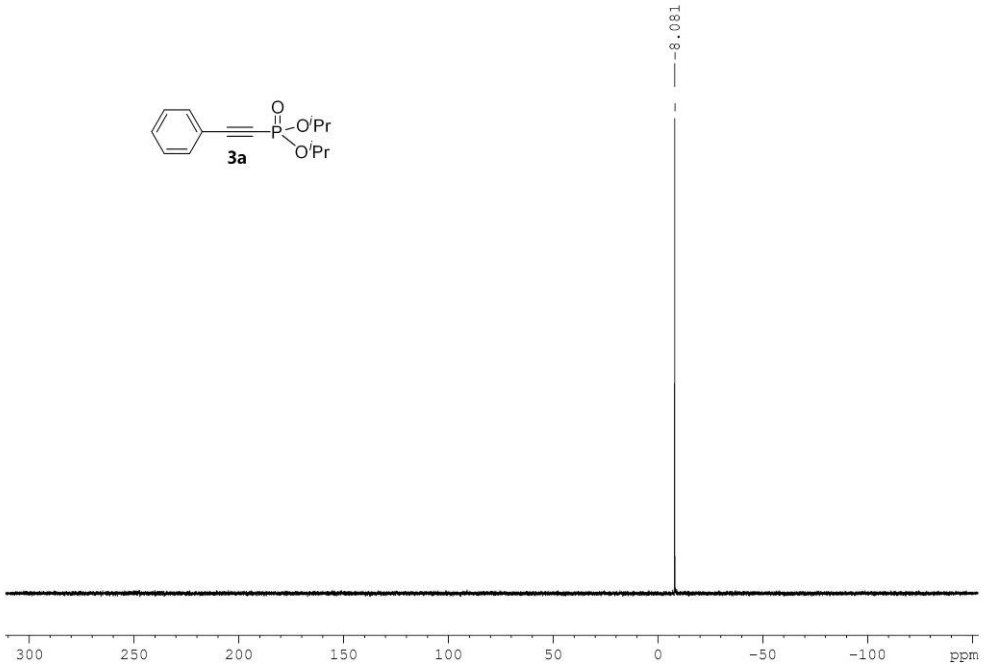
Reference:

1. Gao, Y.; Wang, G.; Chen, L.; Xu, P.; Zhao, Y., Zhou, Y.; Han, L.-B. *J. Am. Chem. Soc.* **2009**, *131*, 7956.
2. Cockburn, N.; Karimi, E.; Tam, W. *J. Org. Chem.* **2009**, *74*, 5762.
3. Li, X.; Hu, G.; Luo, P.; Tang, G.; Gao, Y.; Xu, P.; Zhao Y. *Adv. Synth. Catal.* **2012**, *354*, 2427.
4. Qu, Z.; Chen, X.; Yuan, J.; Qu, L.; Li, X.; Wang, F.; Ding, X.; Zhao, Y. *Can. J. Chem.* **2012**, *90*,747.
5. Hu, J.; Zhao, N.; Yang, B.; Wang, G.; Guo, L.-N.; Liang, Y.-M.; Yang, S.-D. *Chem.-Eur. J.* **2011**, *17*, 5516.

Copies of ^{1}H NMR, ^{13}C NMR, and ^{31}P NMR spectra of products



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200
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100
50
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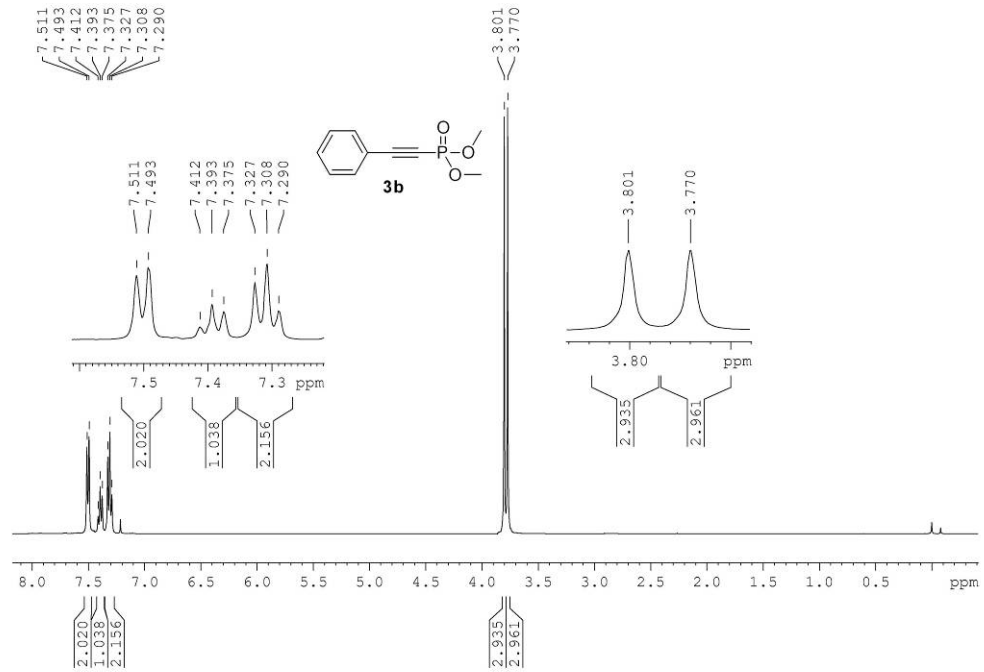
-100
ppm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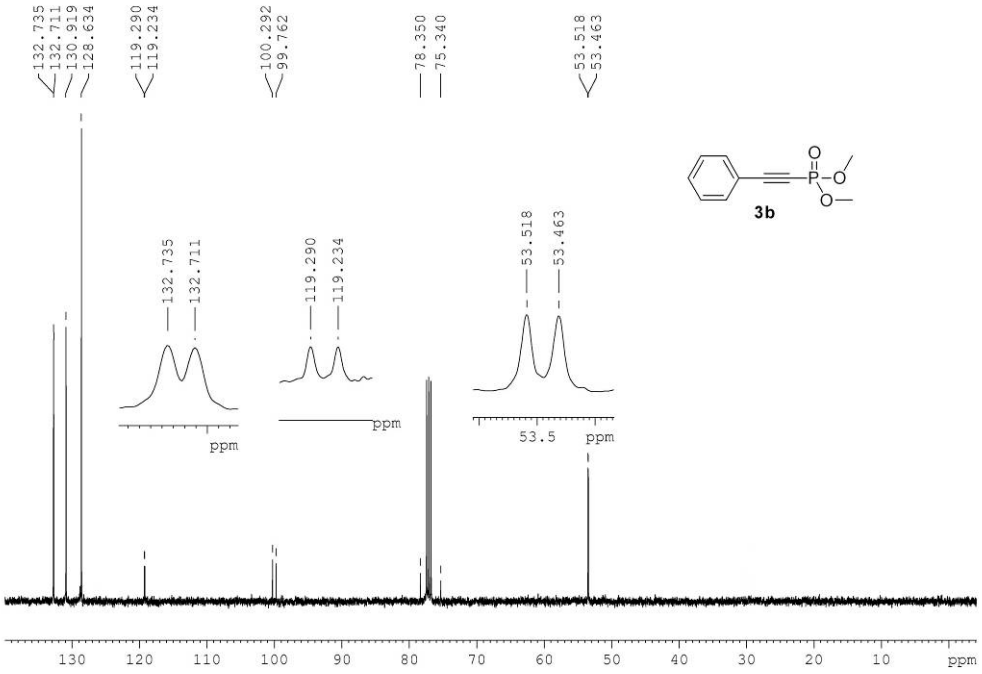
7.493
7.412
7.393
7.375
7.327
7.308
7.290
3.801
3.770
3b
7.5
7.4
7.3 ppm
2.020
1.038
2.156
3.80
ppm
2.935
2.961
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm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130.919
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99.762
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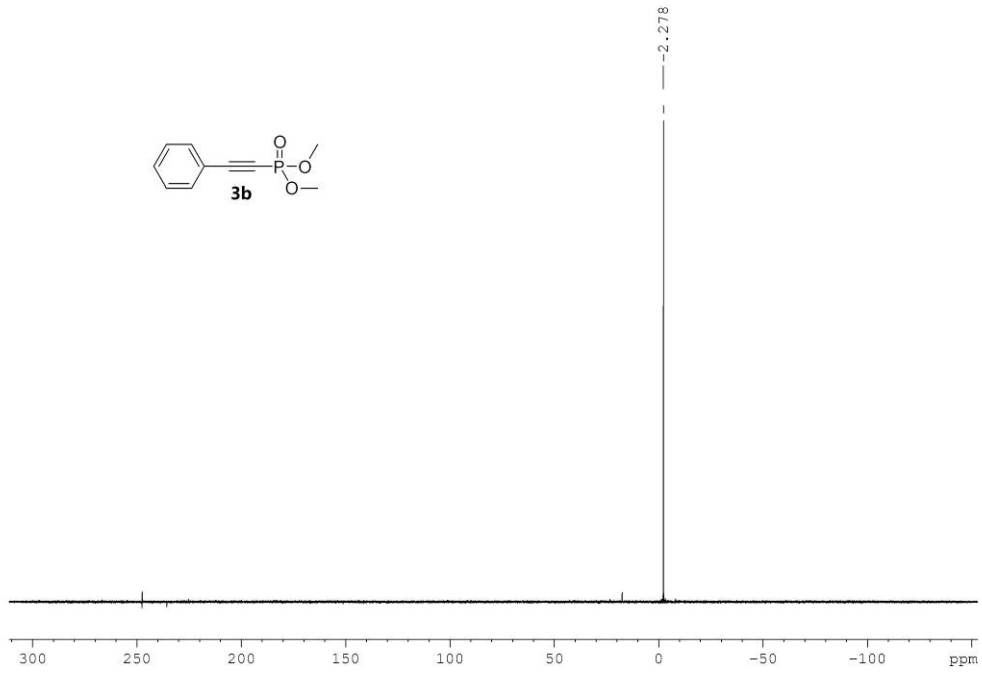
75.340
53.518
53.463
3b
ppm
53.5
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm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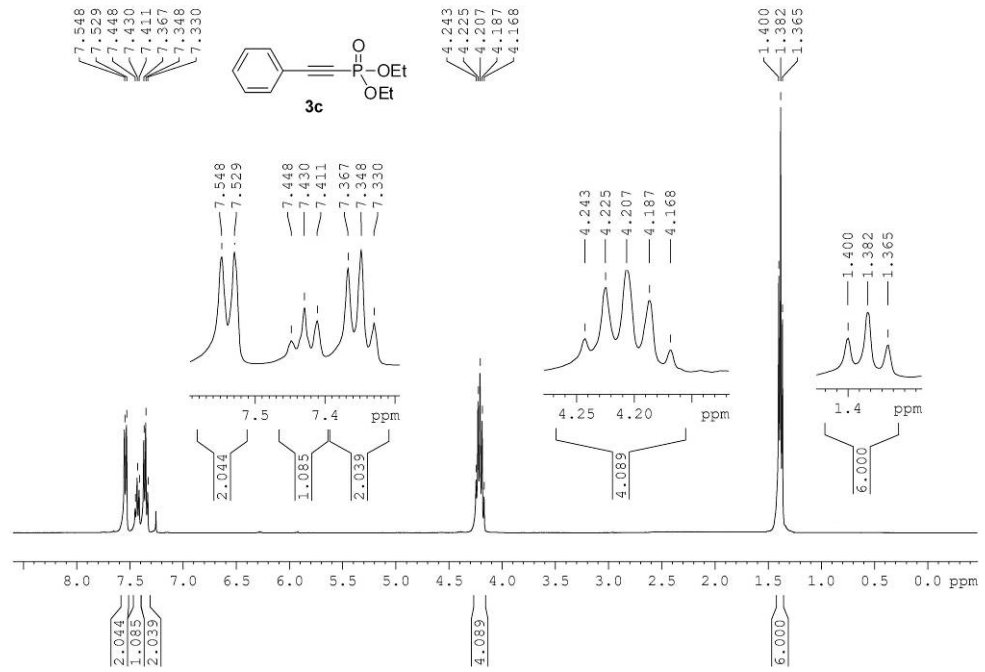
200
150
100
50
0
-50
-100
ppm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7.448
7.430
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7.367
7.348
7.330
4.243
4.225
4.207
4.187
4.168
1.400
1.382
1.365
O
P
OEt
OEt
3c
7.5
7.4
ppm
4.25
4.20
1.4
2.044
1.085
2.039
4.089
6.000
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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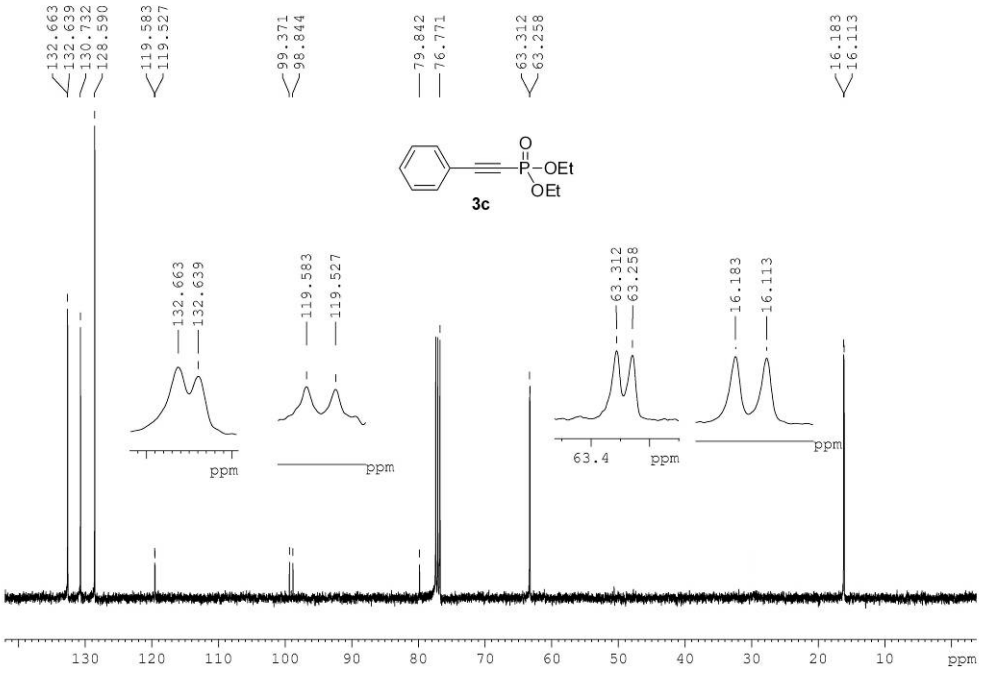
3.0
2.5
2.0
1.5
1.0
0.5
0.0 ppm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98.844
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63.258
16.183
16.113
O
P
OEt
OEt
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63.4
ppm
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120
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100
90
80
70
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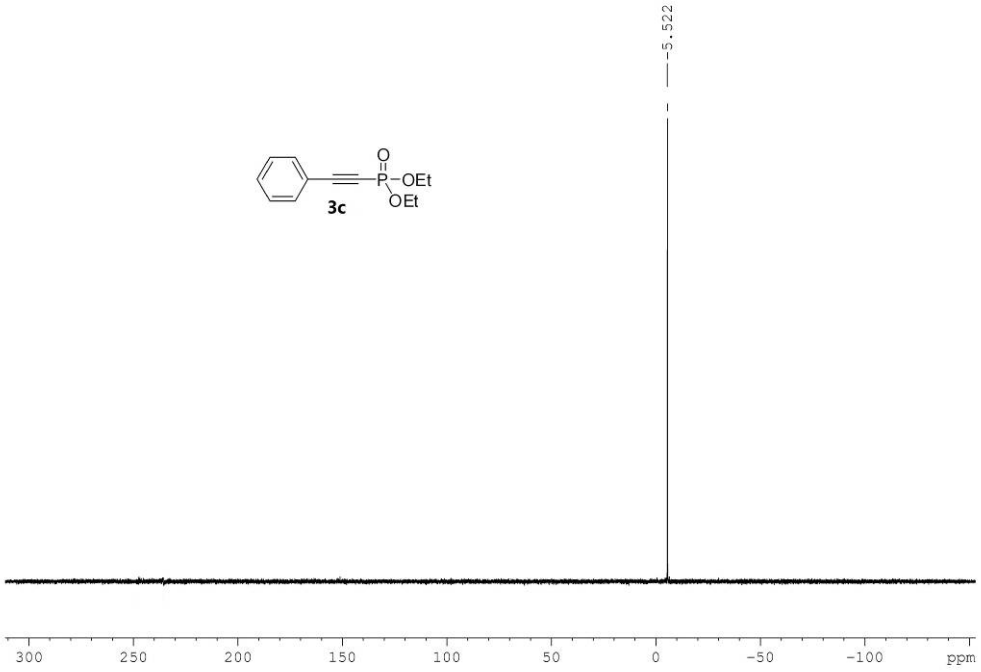
40
30
20
10
ppm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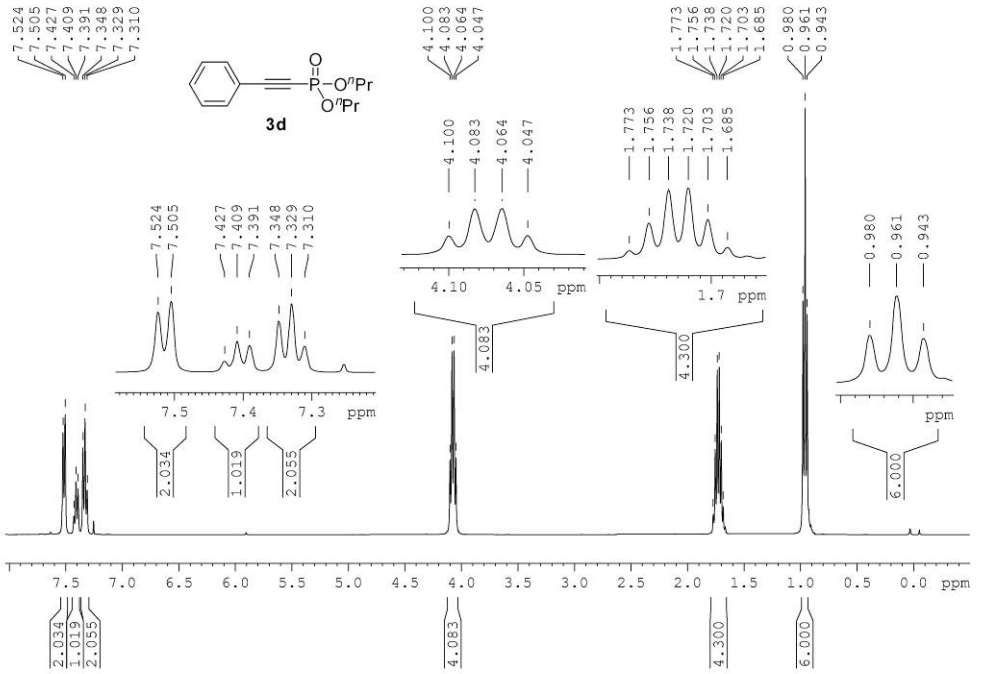
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7.348
7.329
7.310
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4.064
4.047
1.773
1.756
1.738
1.720
1.703
1.685
0.980
0.961
0.943
3d
4.10
4.05
ppm
1.7
ppm
7.5
7.4
7.3
ppm
2.034
1.019
2.055
4.083
4.300
6.000
7.5
7.0
6.5
6.0
5.5
5.0
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4.0
3.5
3.0
2.5
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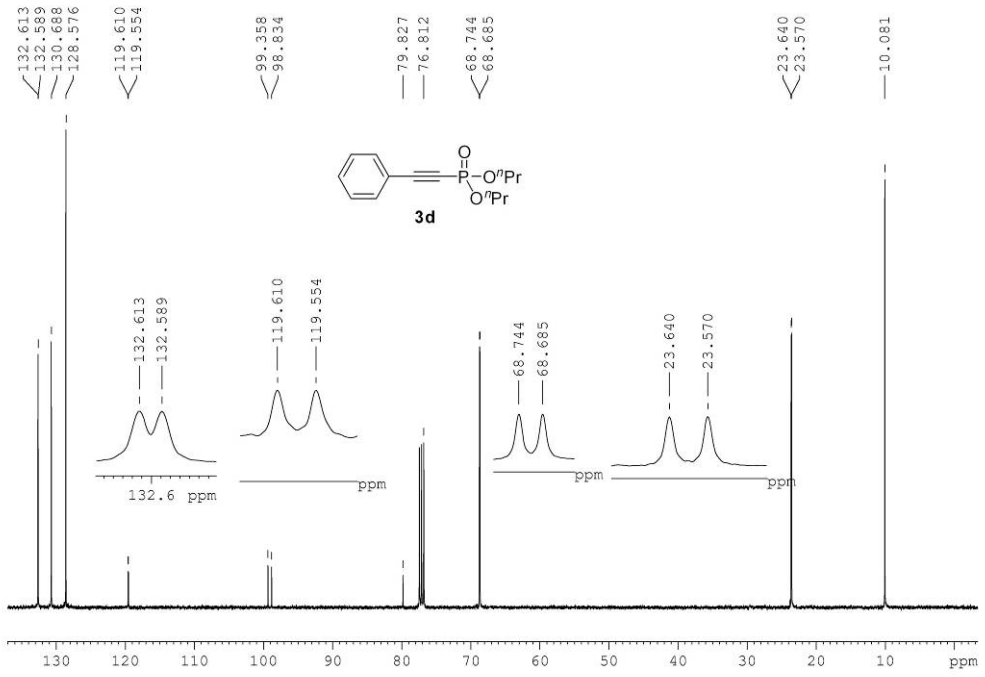
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23.570
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ppm
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110
100
90
80
70
60
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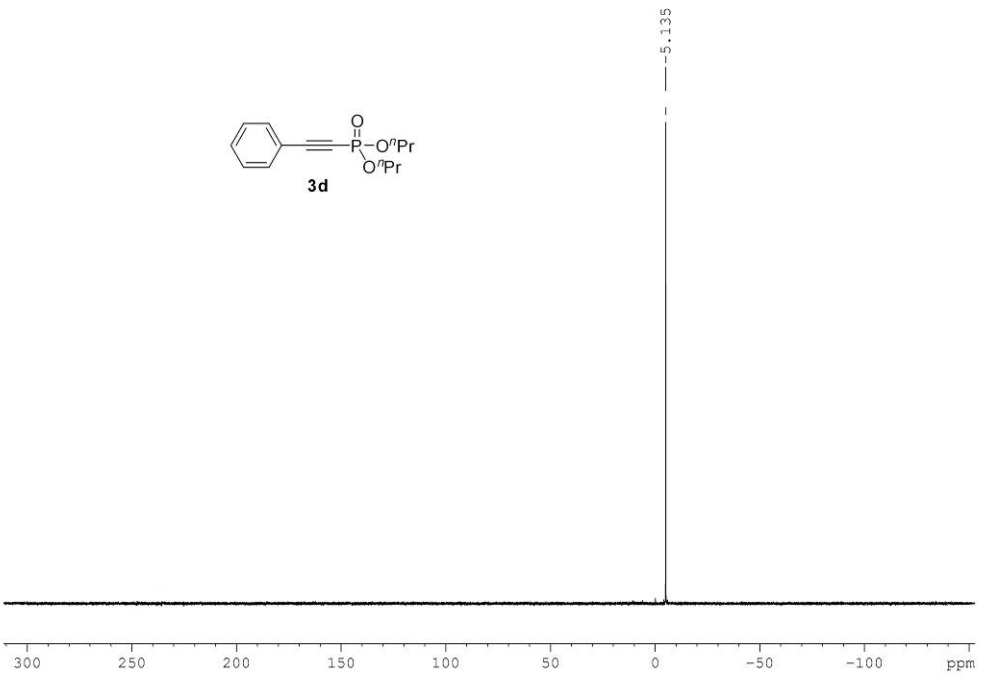
40
30
20
10
ppm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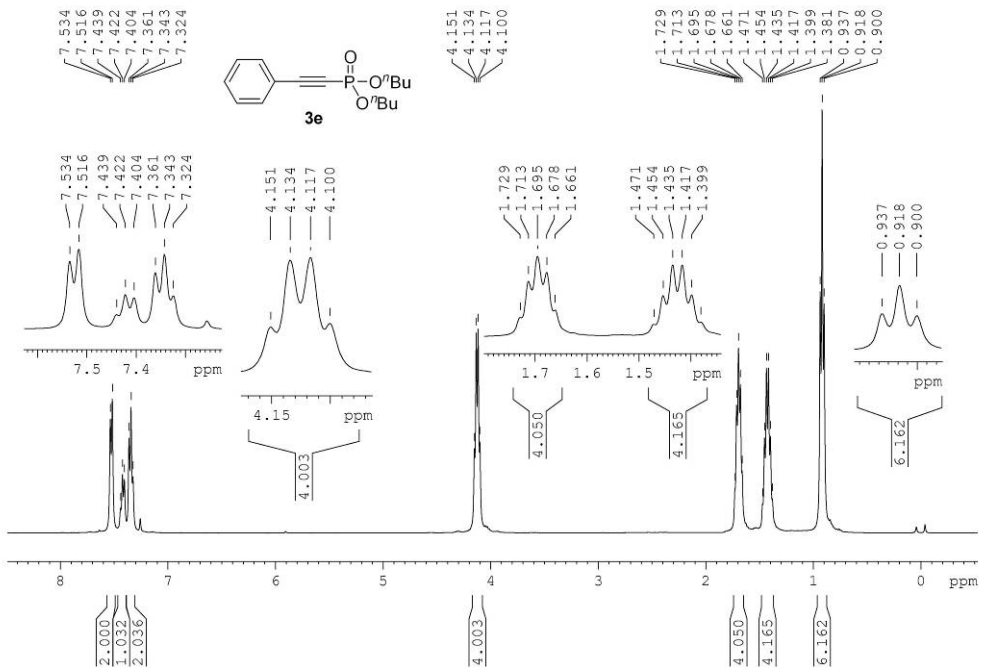
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7.324
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1.713
1.695
1.678
1.661
1.471
1.454
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ppm
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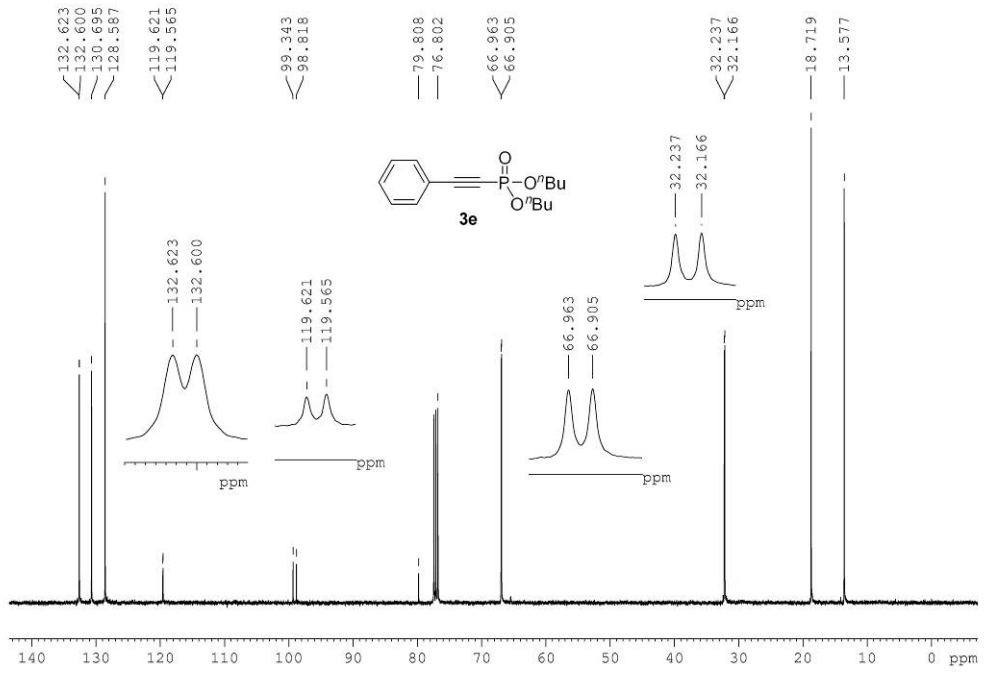
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13.577
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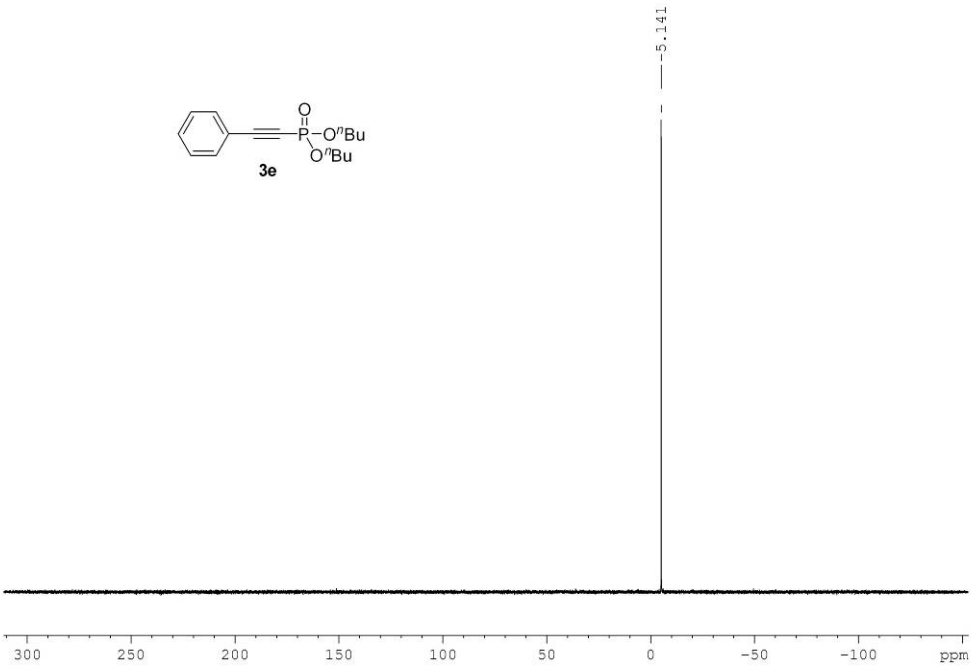
ppm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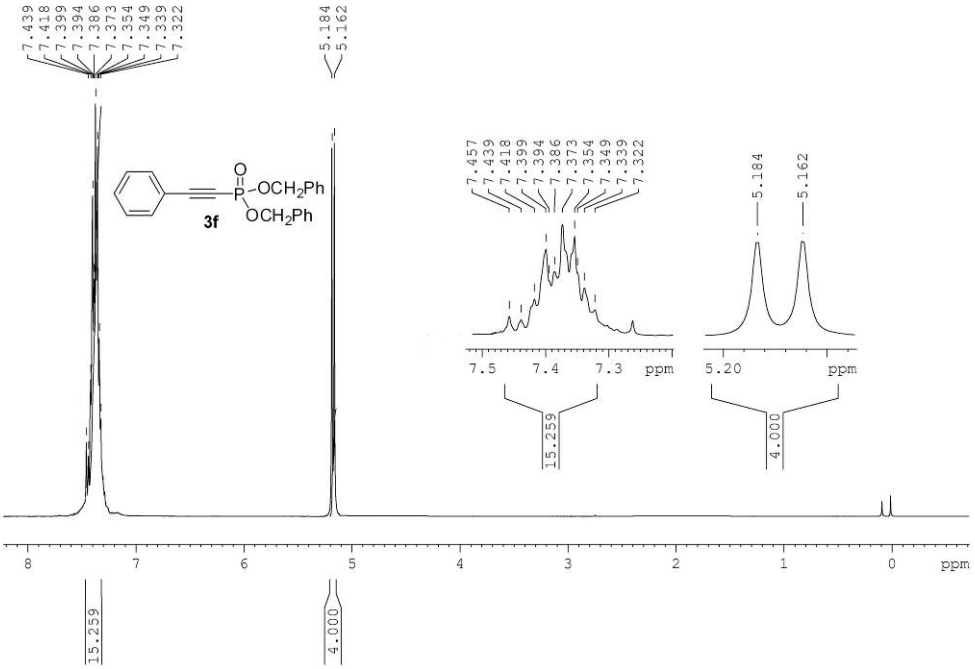
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7.322
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7.4
7.3
ppm
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ppm
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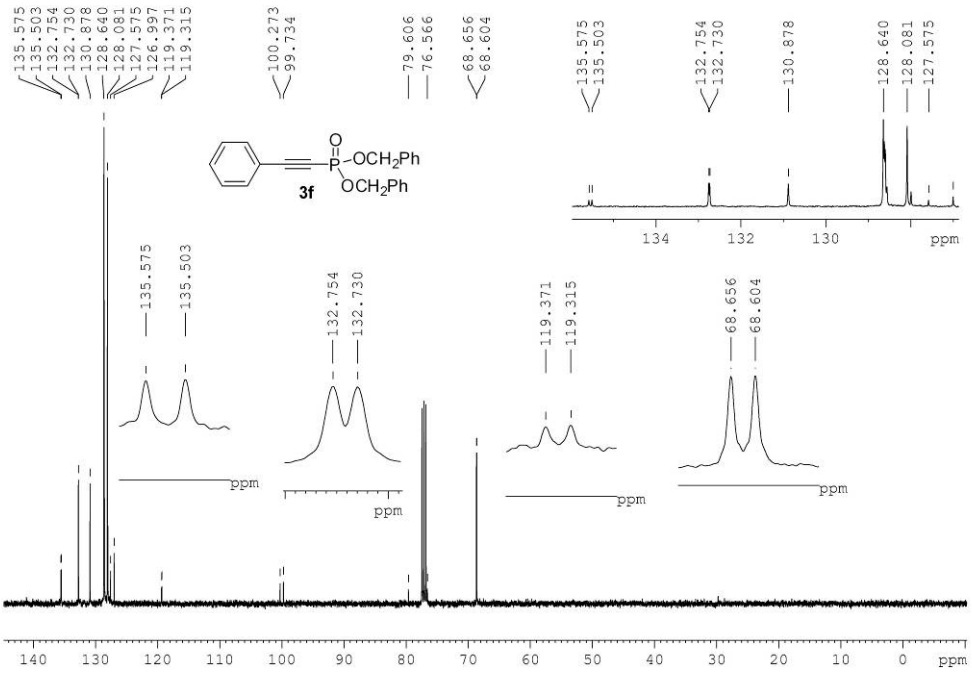
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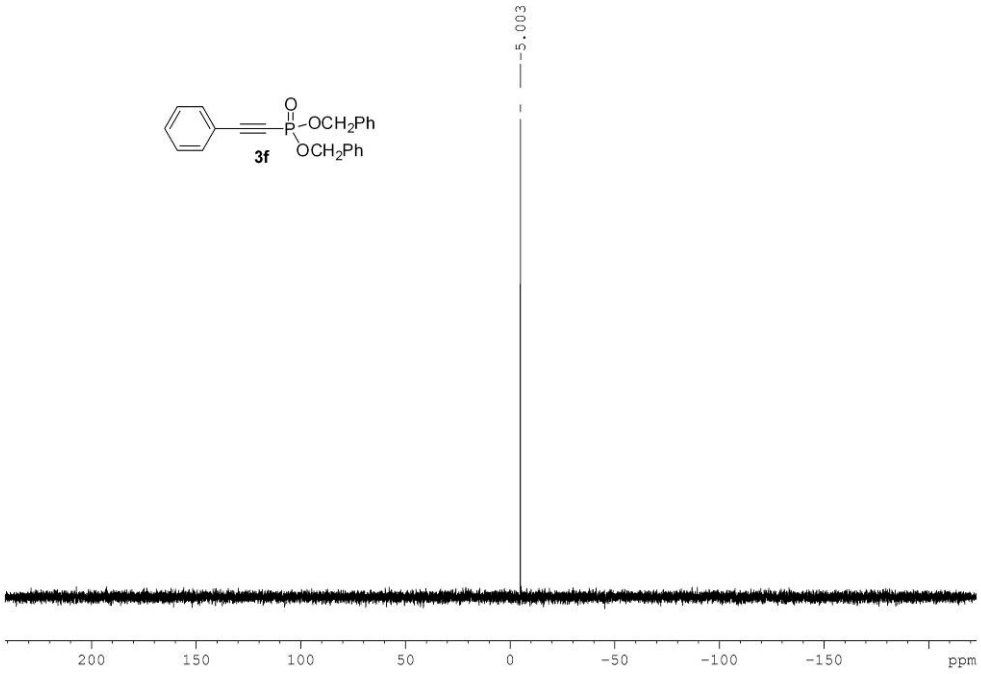
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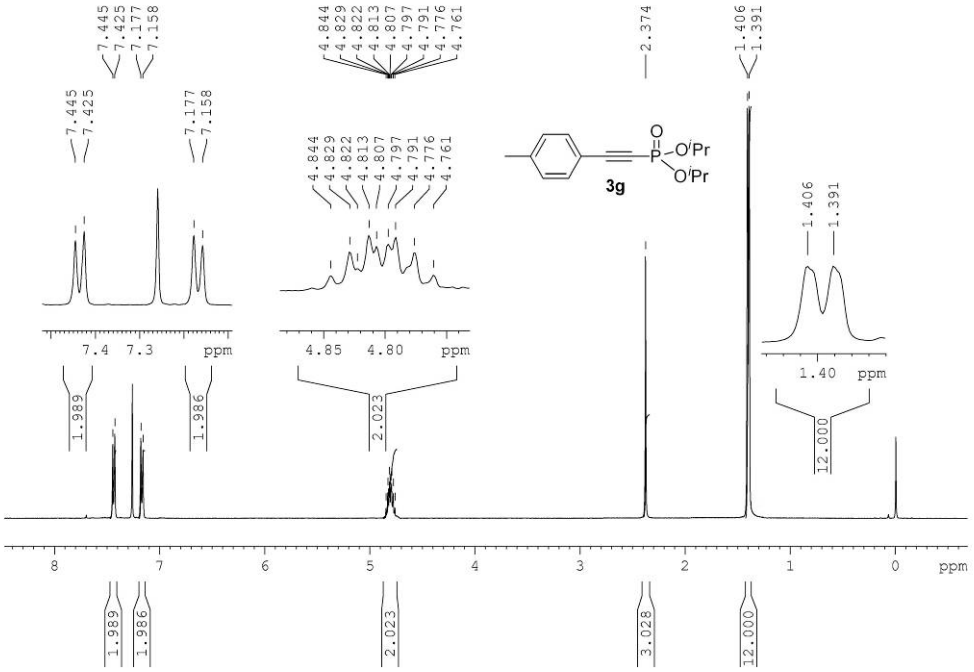
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2.374
1.406
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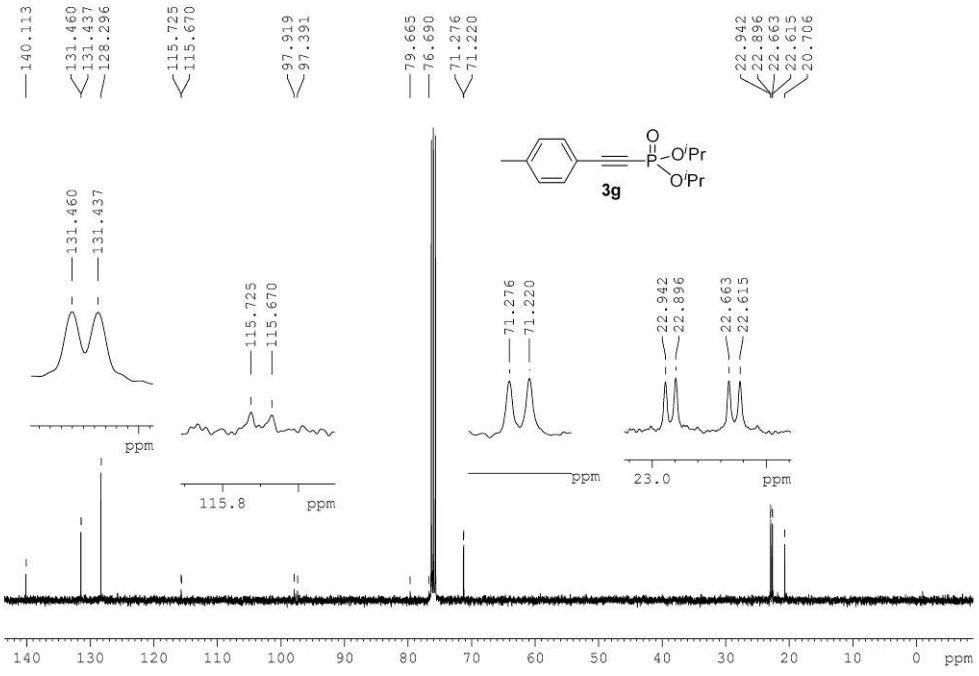
3.028
ppm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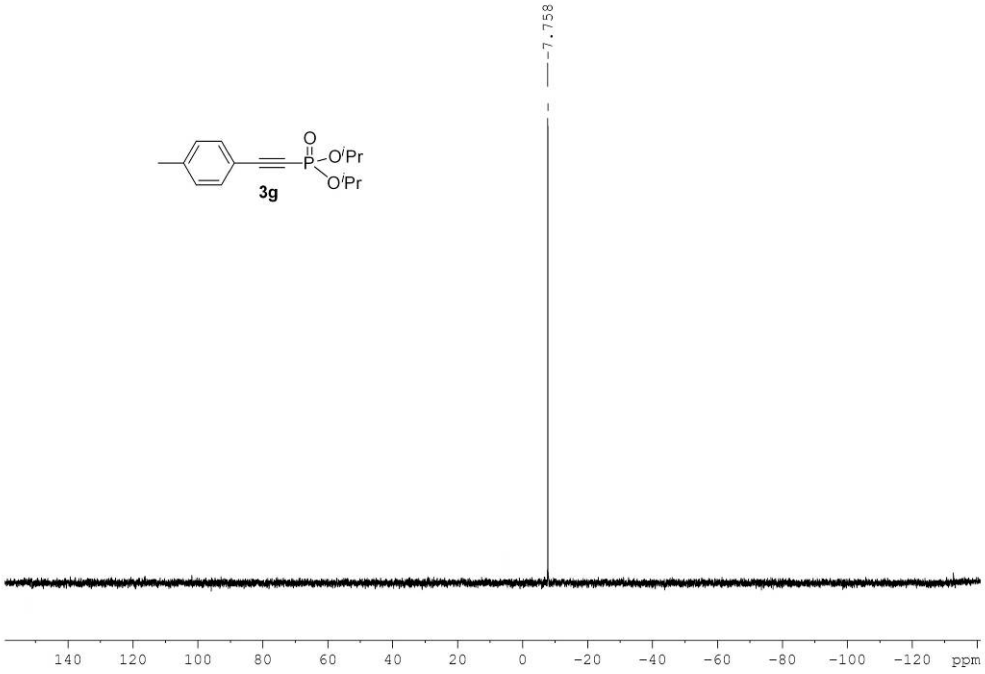
ppm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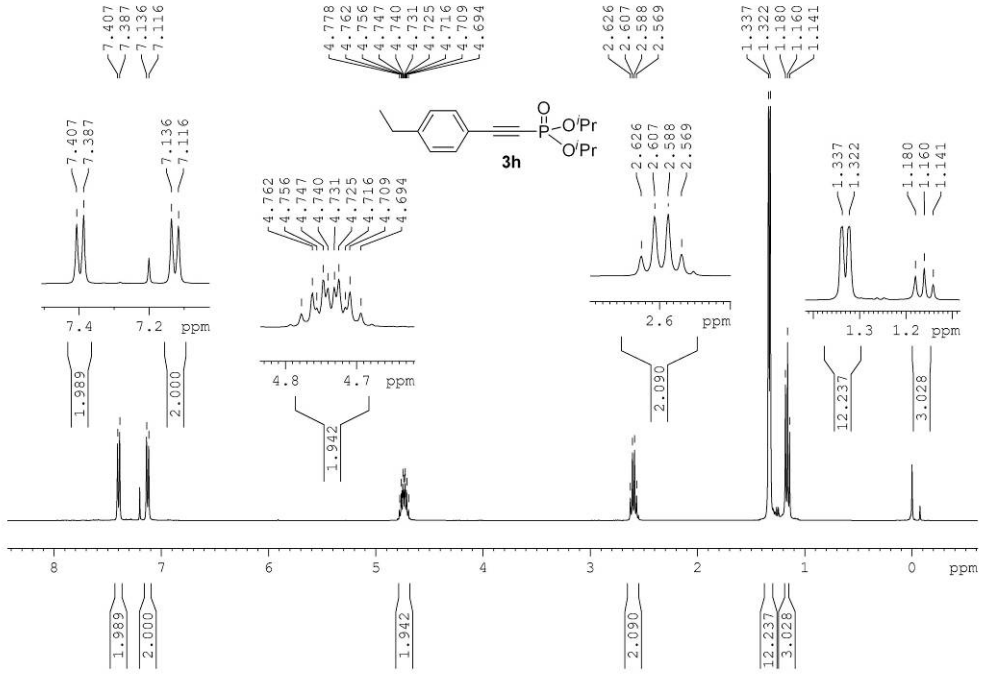
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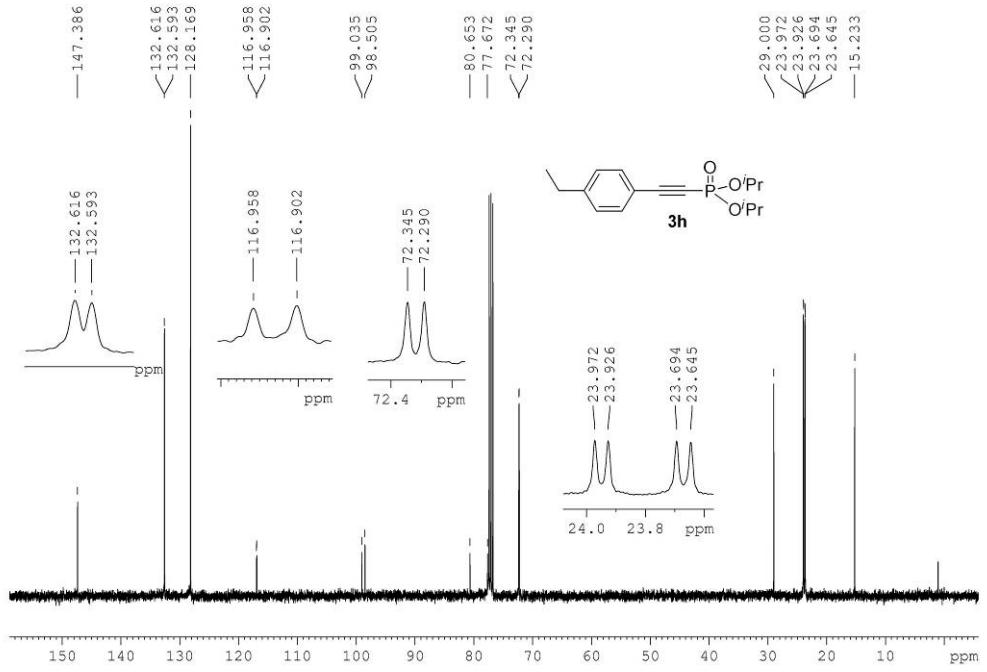
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3h
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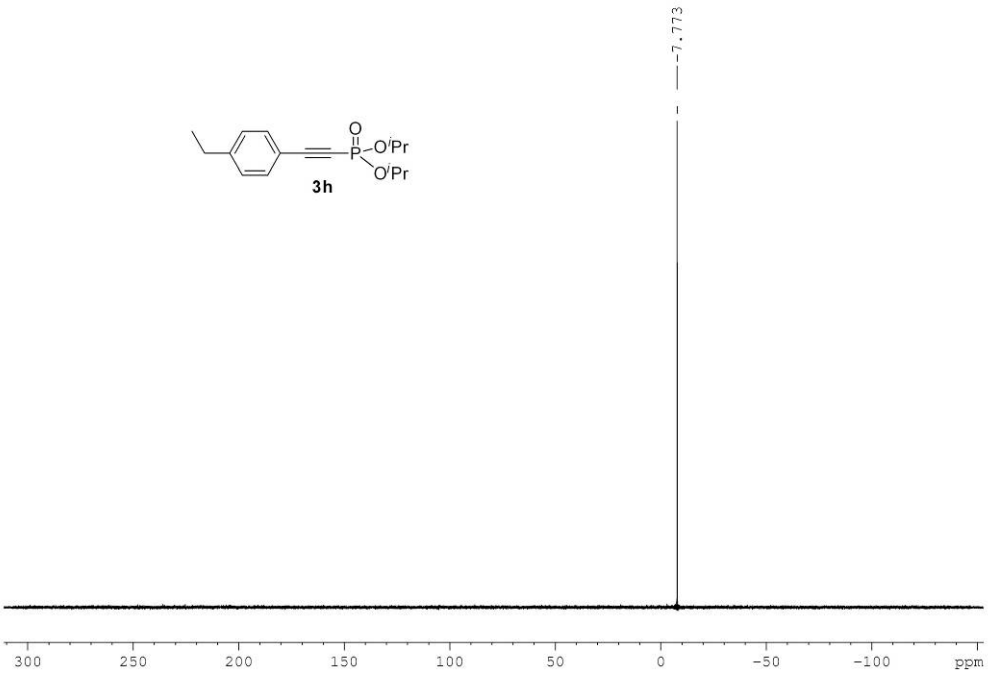
23.8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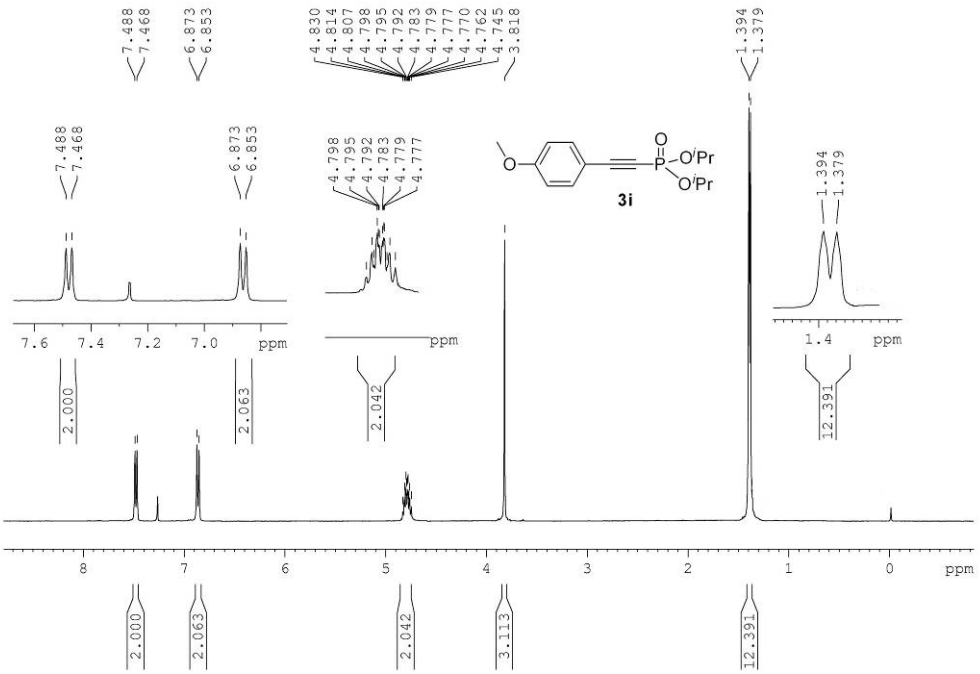
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4.777
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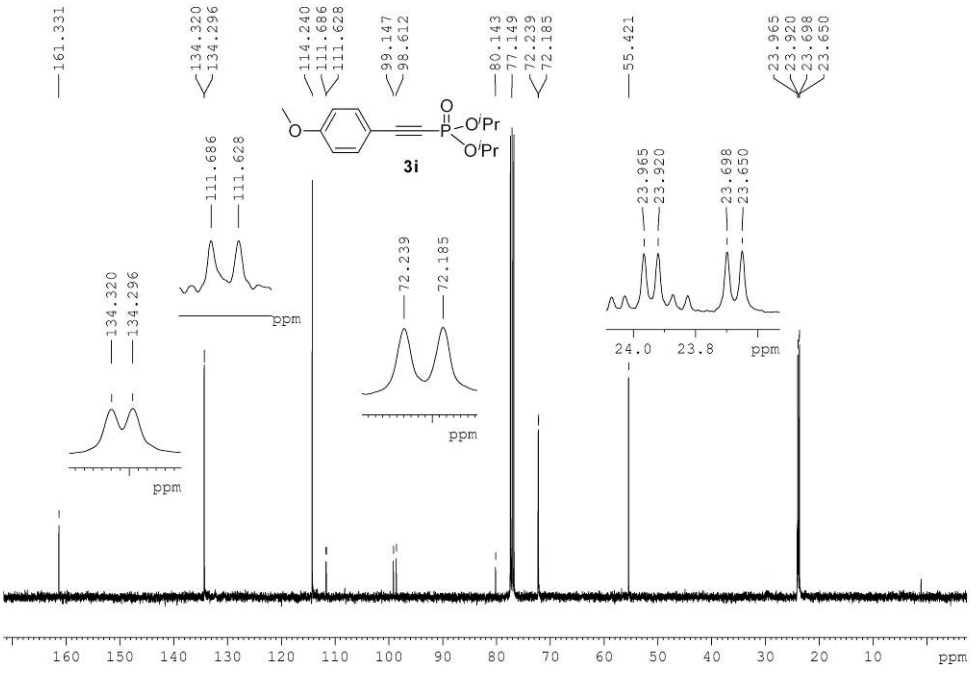
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3i
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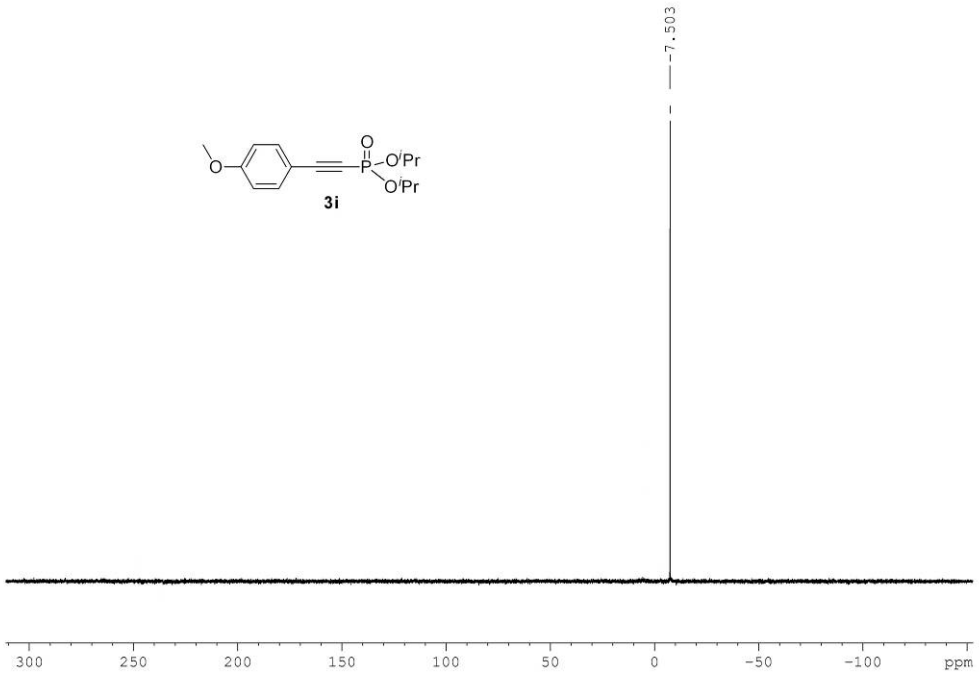
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O
P
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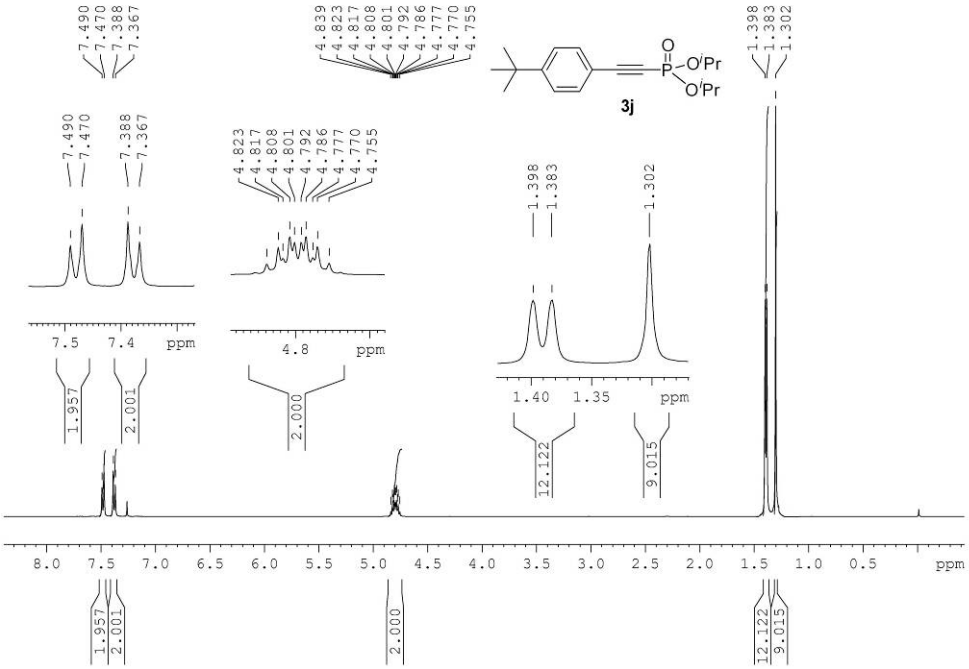
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5.0
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3.0
2.5
2.0
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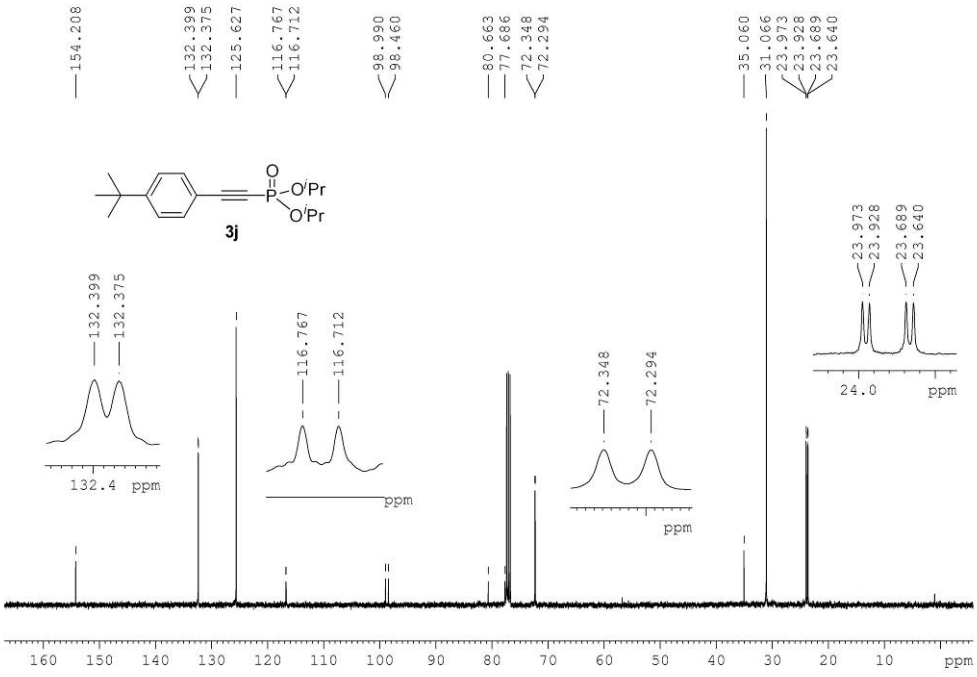
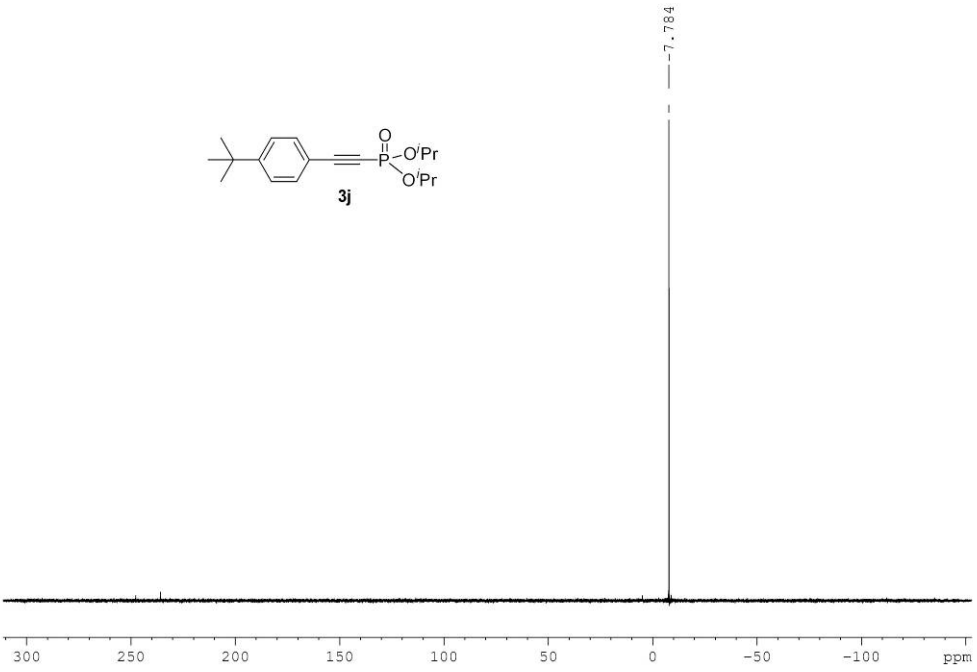
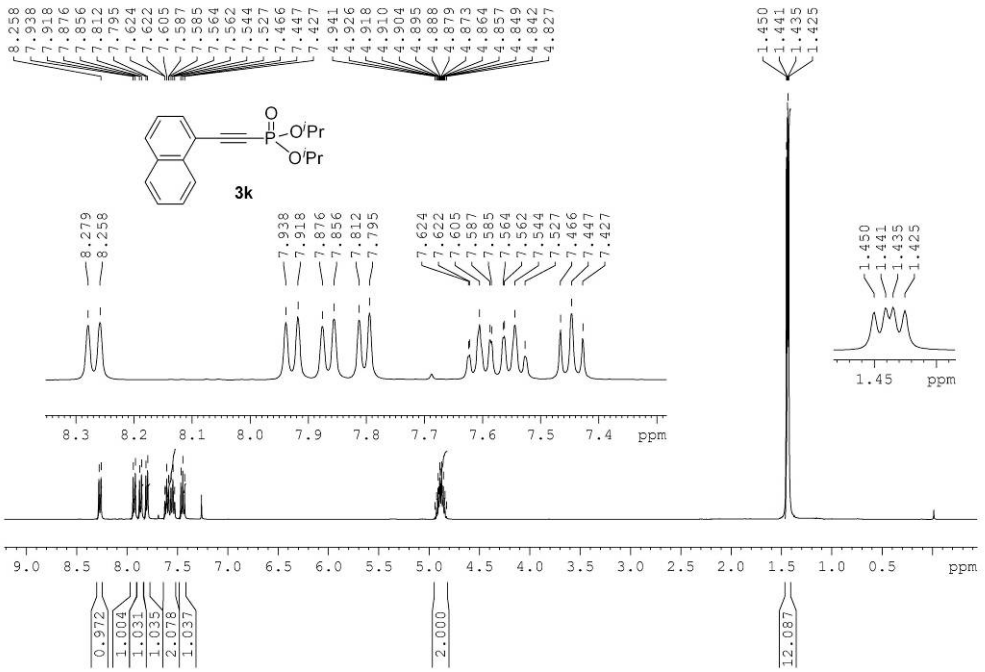
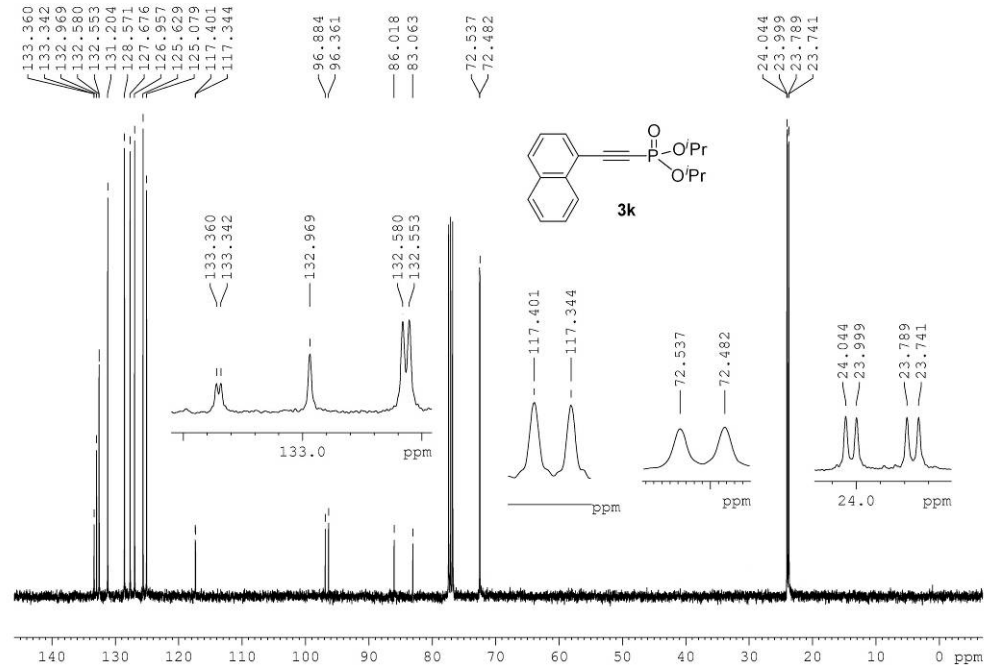
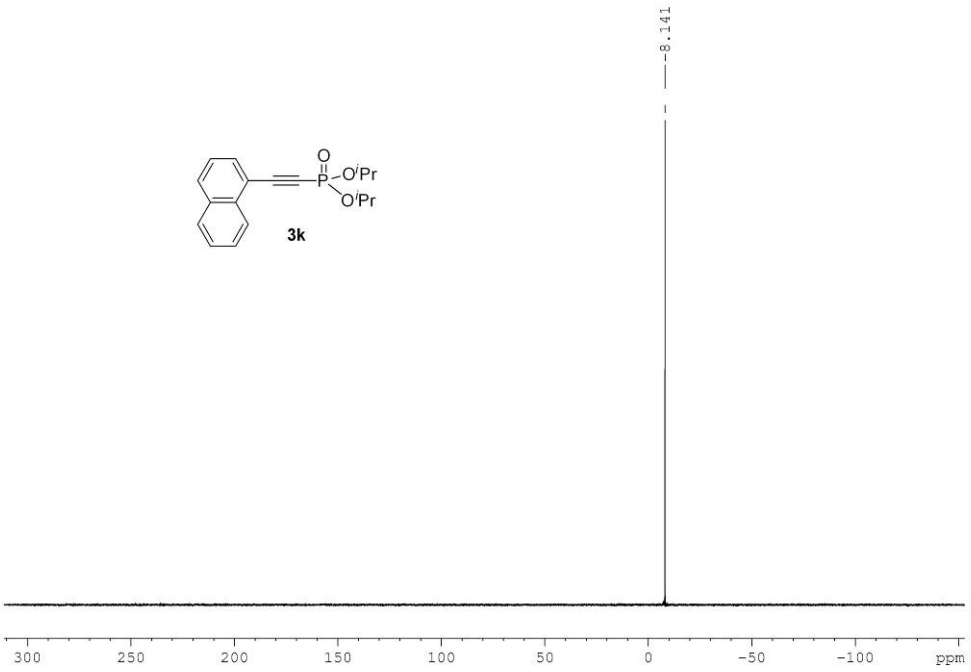
1.0
0.5
ppm

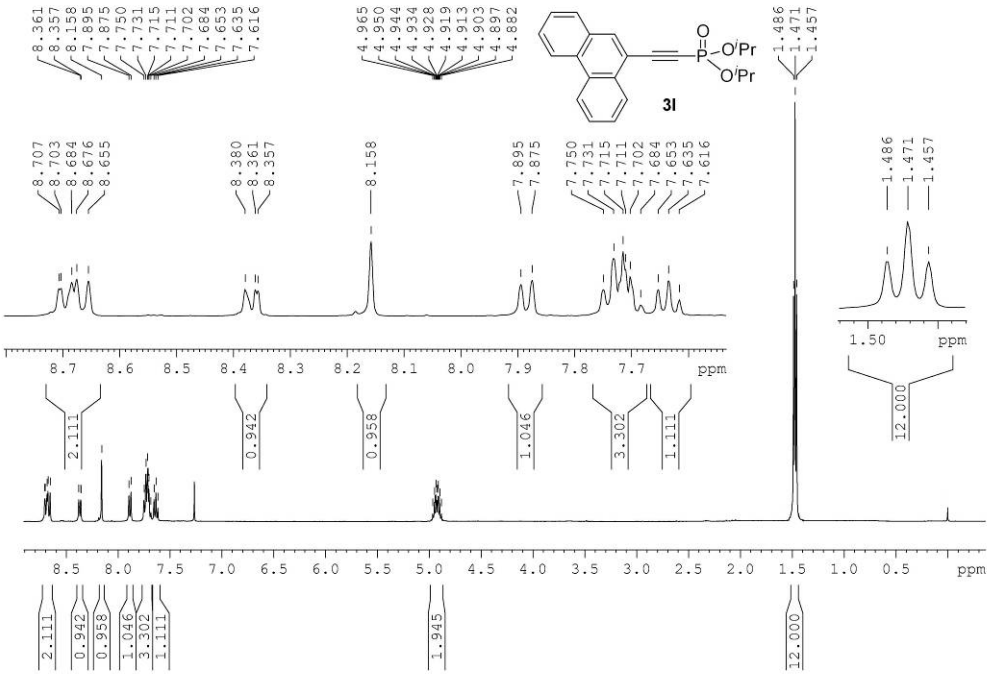

154.208
132.399
132.375
125.627
116.767
116.712
98.990
98.460
80.663
77.686
72.348
72.294
35.060
31.066
23.973
23.928
23.689
23.640
3j
132.4 ppm
24.0 ppm
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

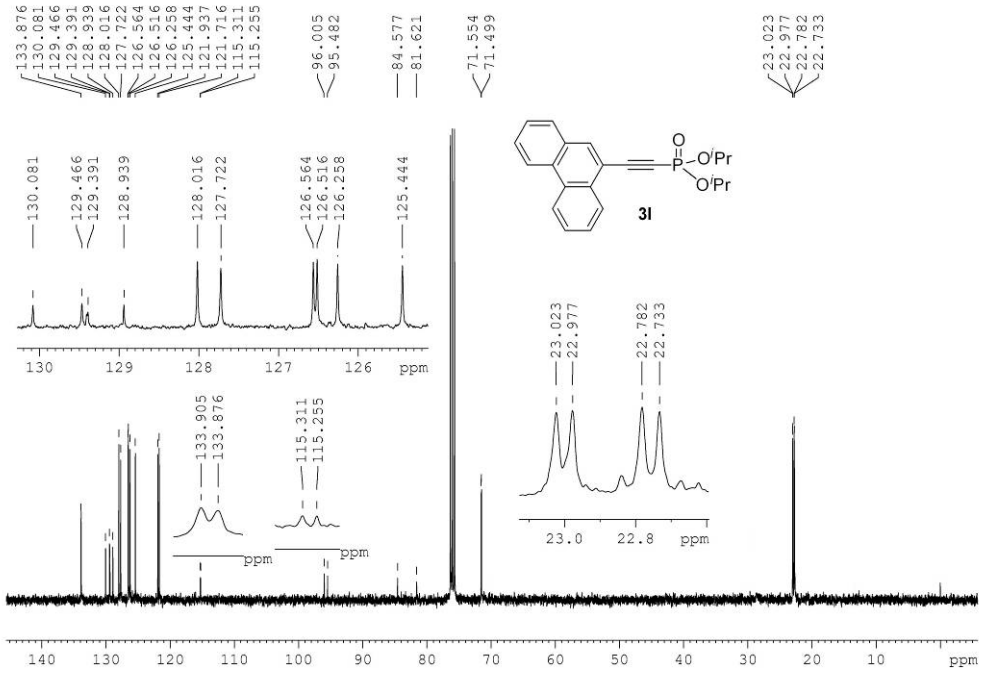

-7.784
3j
300 250 200 150 100 50 0 -50 -100 ppm

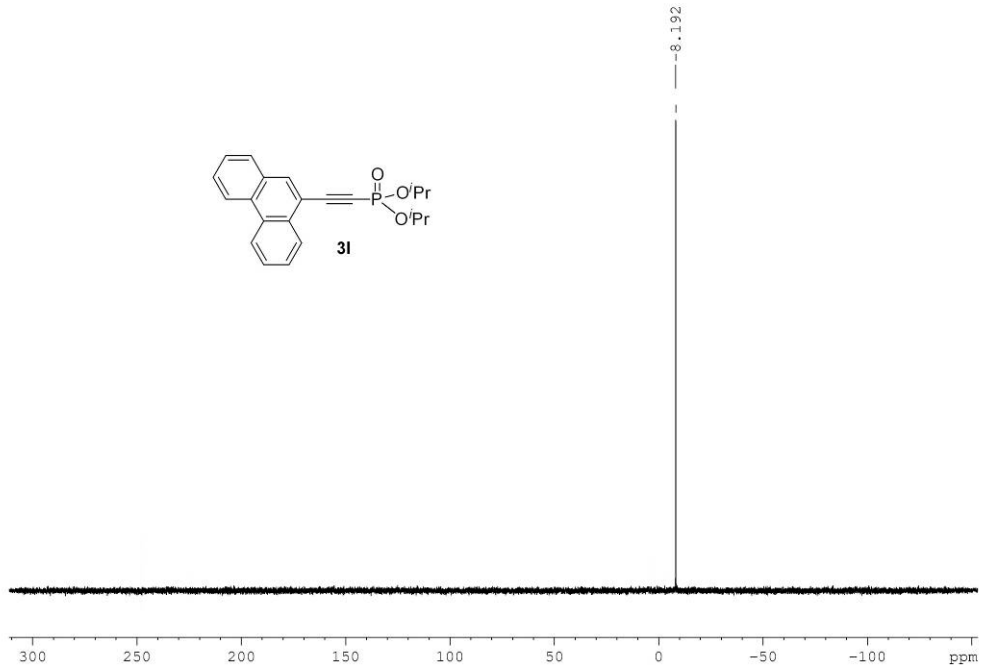

8.258
7.938
7.918
7.876
7.856
7.812
7.795
7.624
7.622
7.605
7.587
7.585
7.564
7.562
7.544
7.527
7.466
7.447
7.427
4.941
4.926
4.918
4.910
4.904
4.895
4.888
4.879
4.873
4.864
4.857
4.849
4.842
4.827
1.450
1.441
1.435
1.425
O
P
O^{i}Pr
O^{i}Pr
3k
8.279
8.258
7.938
7.918
7.876
7.856
7.812
7.795
7.624
7.622
7.605
7.587
7.585
7.564
7.562
7.544
7.527
7.466
7.447
7.427
1.450
1.441
1.435
1.425
1.45 ppm
8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 ppm
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.972
1.004
1.031
1.035
2.078
1.037
2.000
12.087

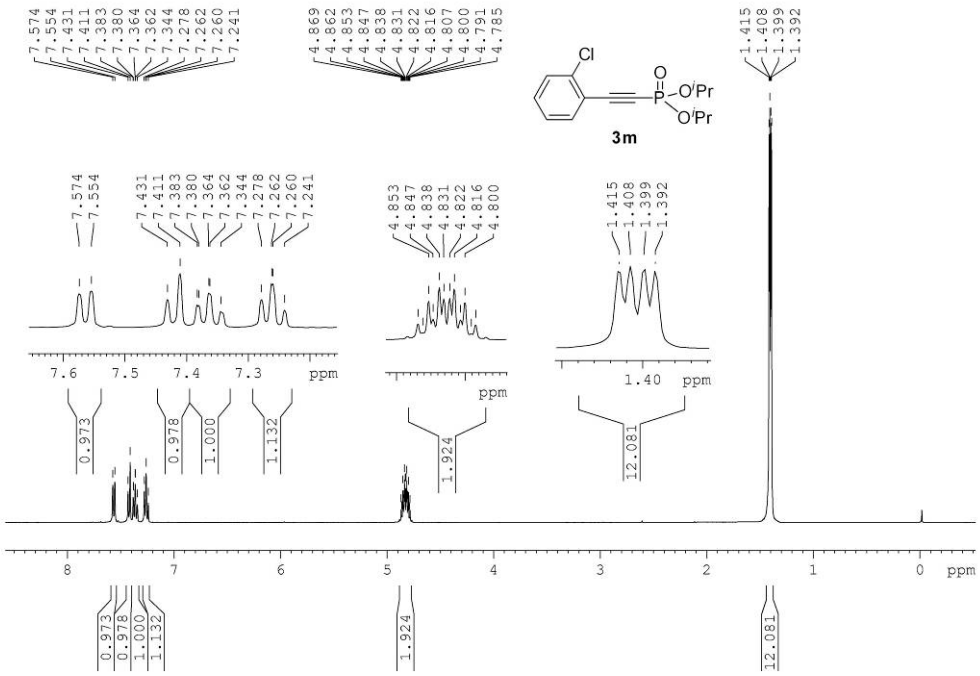

133.360
133.342
132.969
132.580
132.553
131.204
128.571
127.676
126.957
125.629
125.079
117.401
117.344
96.884
96.361
86.018
83.063
72.537
72.482
24.044
23.999
23.789
23.741
O
P
O^{i}Pr
O^{i}Pr
3k
133.360
133.342
132.969
132.580
132.553
133.0 ppm
117.401
117.344
ppm
72.537
72.482
ppm
24.044
23.999
23.789
23.741
24.0 ppm
140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

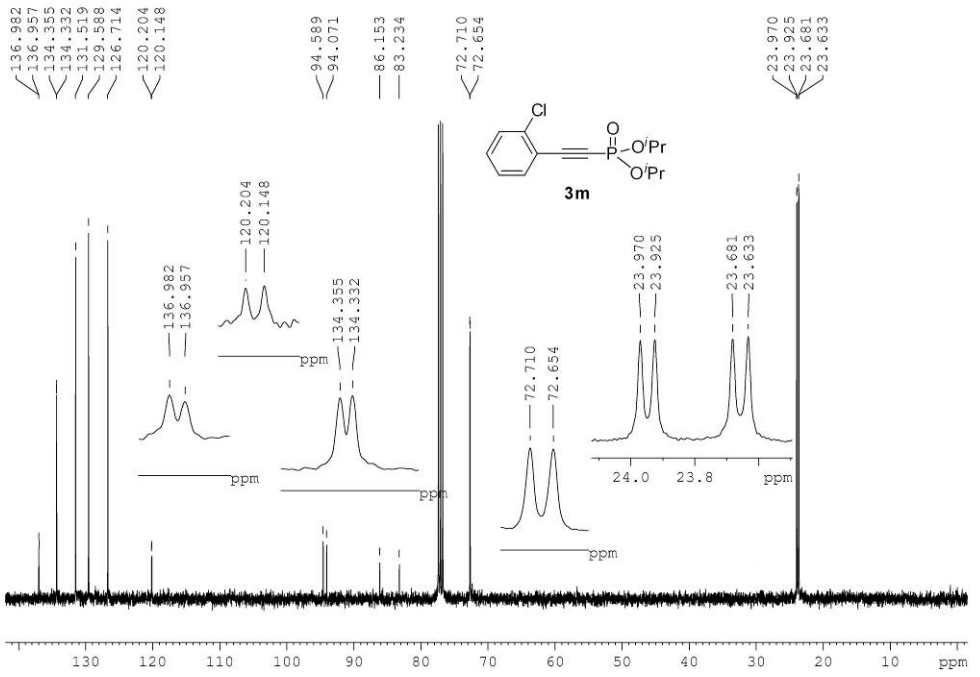

8.141
3k
300
250
200
150
100
50
0
-50
-100
ppm

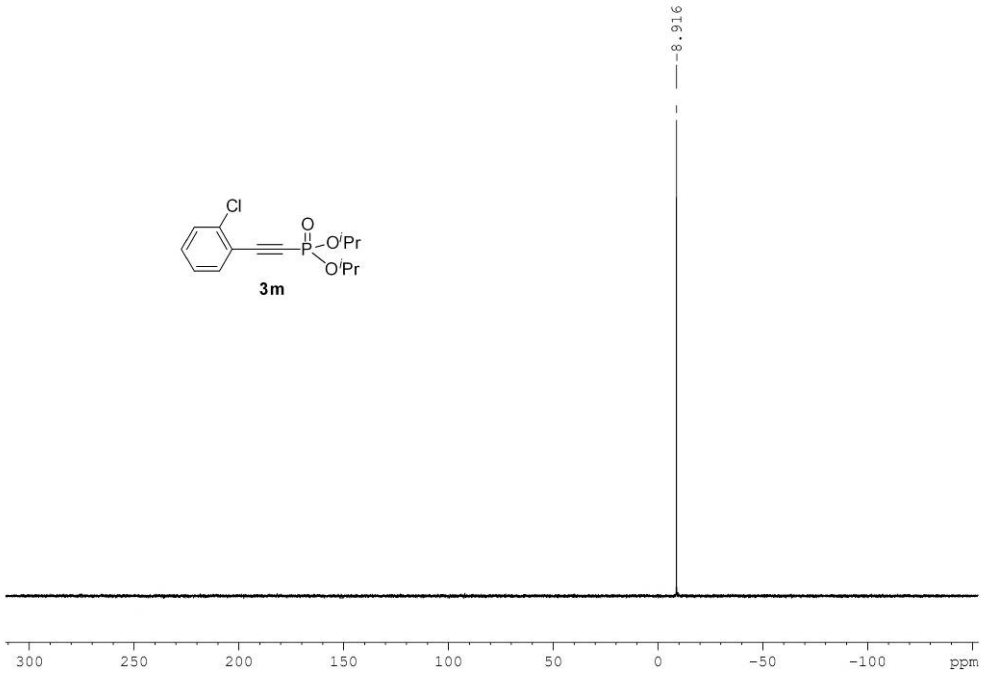

8.361
8.357
8.158
7.895
7.875
7.750
7.731
7.715
7.711
7.702
7.684
7.653
7.635
7.616
4.965
4.950
4.944
4.934
4.928
4.919
4.913
4.903
4.897
4.882
1.486
1.471
1.457
3l
8.7
8.6
8.5
8.4
8.3
8.2
8.1
8.0
7.9
7.8
7.7
ppm
2.111
0.942
0.958
1.046
3.302
1.111
1.50
12.000
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
1.945

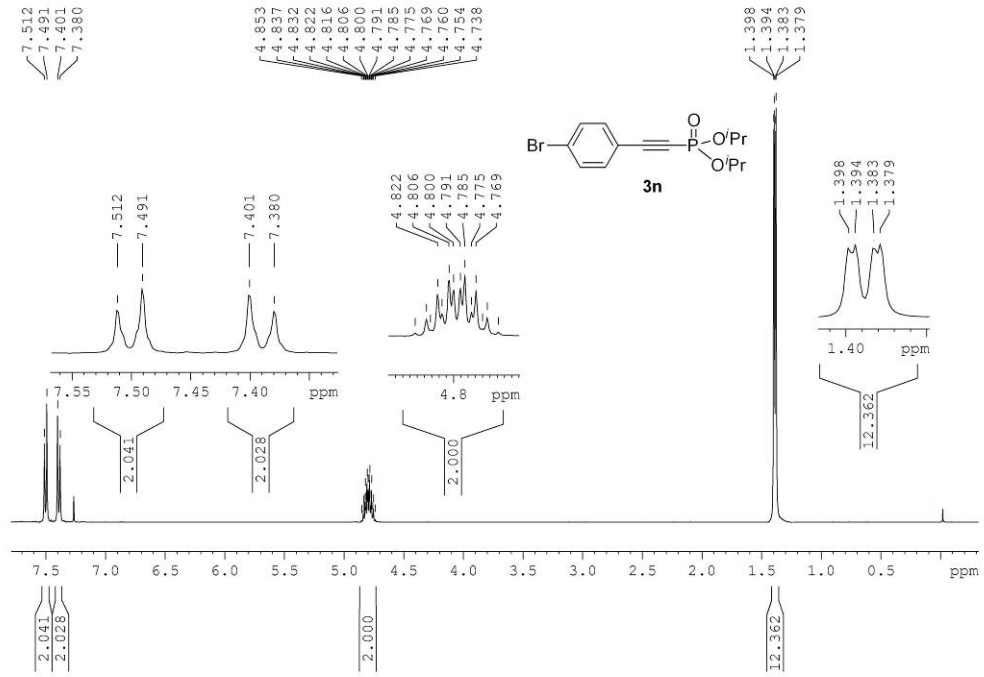

133.876
130.081
129.466
129.391
128.939
128.016
127.722
126.564
126.516
126.258
125.444
121.937
121.716
115.311
115.255
96.005
95.482
84.577
81.621
71.554
71.499
23.023
22.977
22.782
22.733
3l
ppm

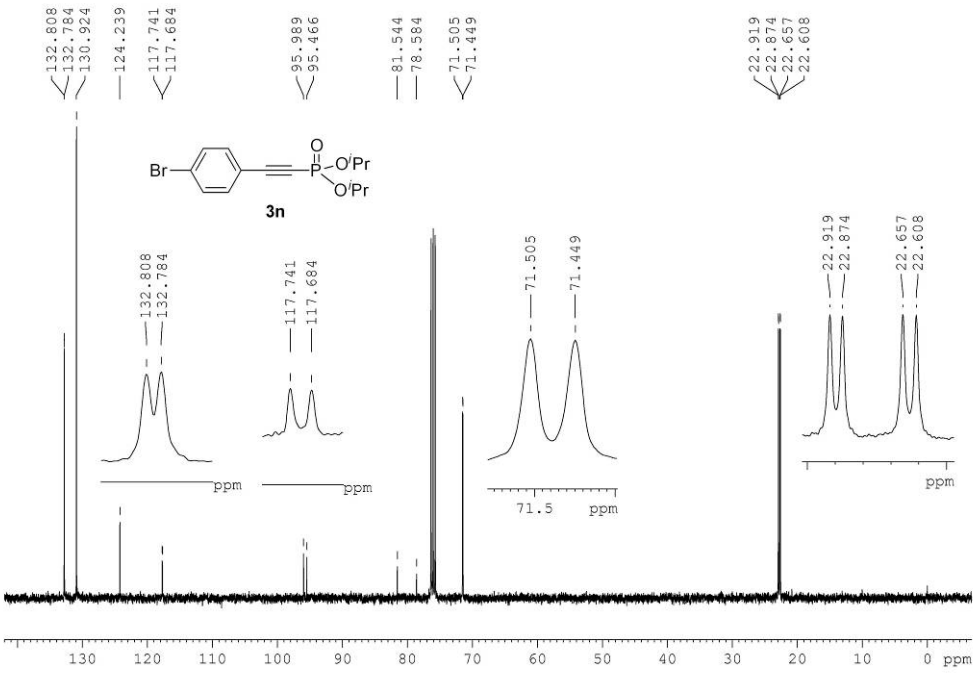

-8.192
3l
ppm

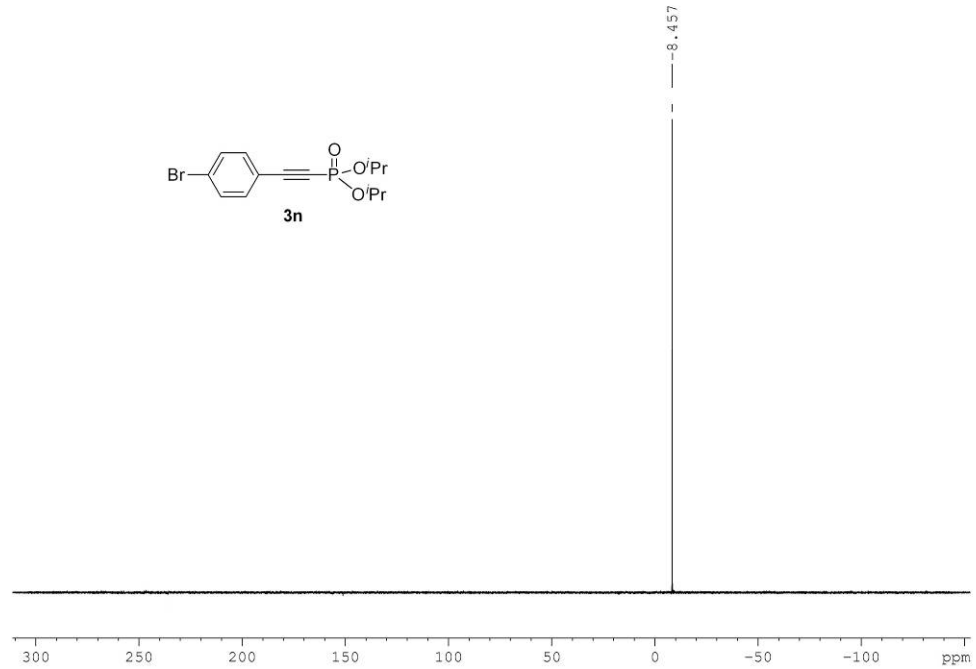

3m
7.574
7.554
7.431
7.411
7.383
7.380
7.364
7.362
7.344
7.278
7.262
7.260
7.241
4.869
4.862
4.853
4.847
4.838
4.831
4.822
4.816
4.807
4.800
4.791
4.785
1.415
1.408
1.399
1.392
0.973
0.978
1.000
1.132
1.924
12.081
ppm

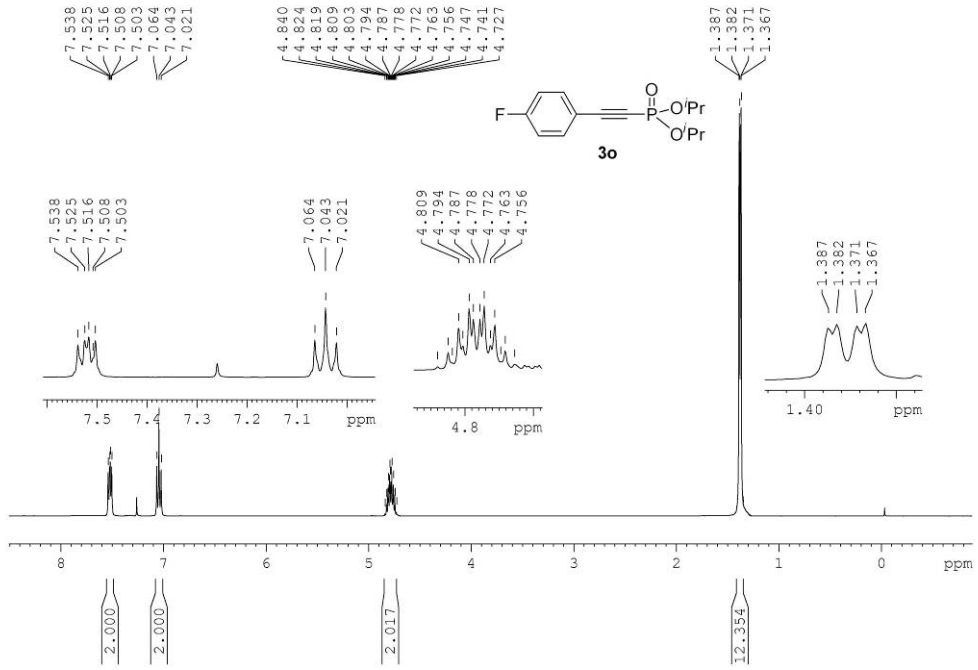

3m
136.982
136.957
134.355
134.332
131.519
129.588
126.714
120.204
120.148
94.589
94.071
86.153
83.234
72.710
72.654
23.970
23.925
23.681
23.633
ppm

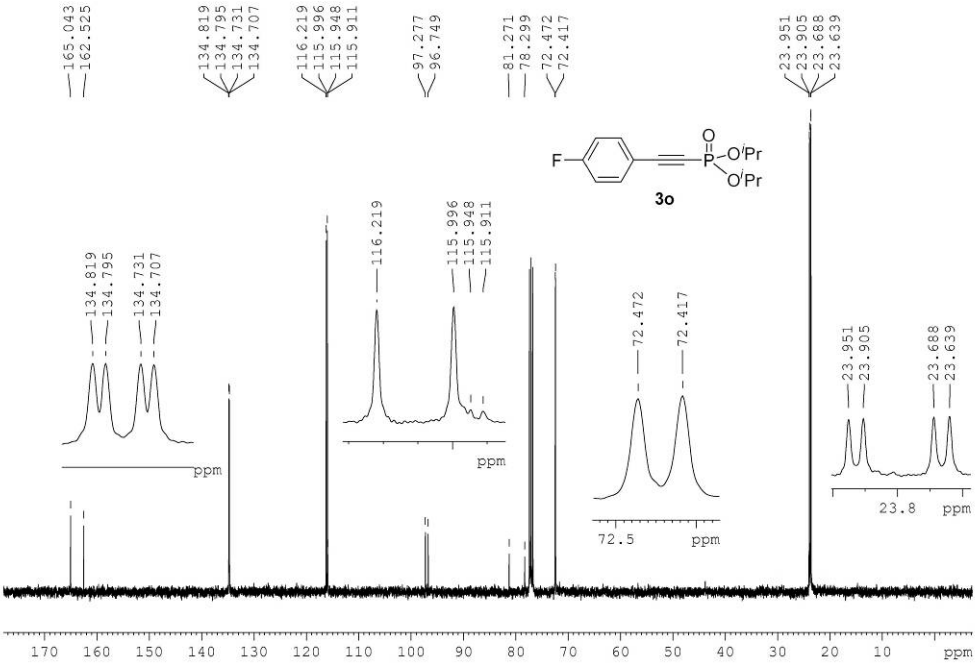

Cl
O
P
O^{i}Pr
O^{i}Pr
3m
8.916
300
250
200
150
100
50
0
-50
-100
ppm

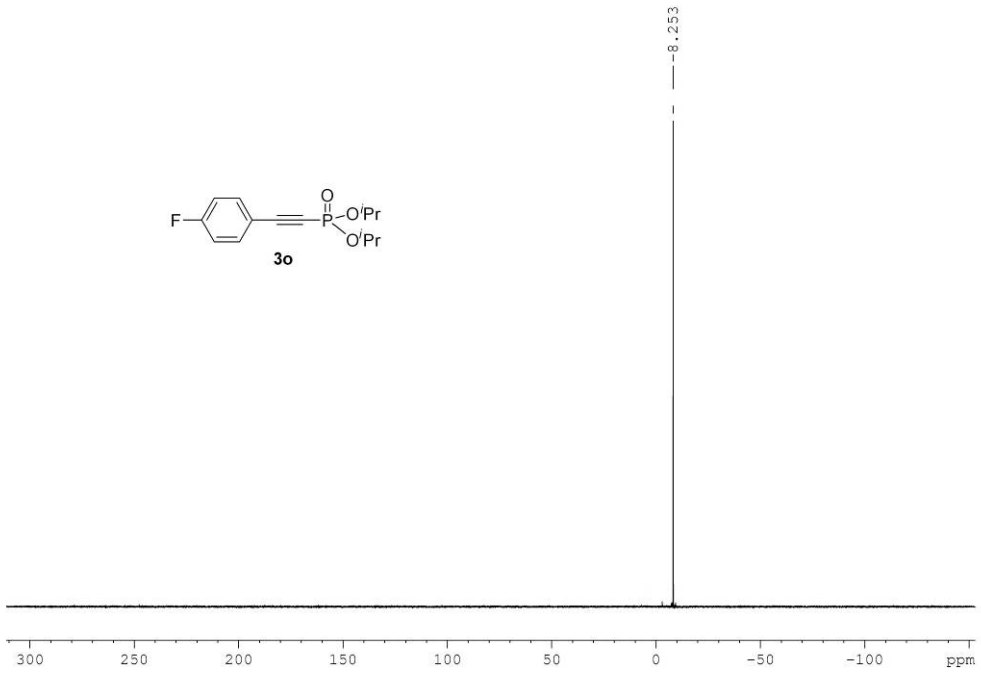

7.512
7.491
7.401
7.380
4.853
4.837
4.832
4.822
4.816
4.806
4.800
4.791
4.785
4.775
4.769
4.760
4.754
4.738
1.398
1.394
1.383
1.379
Br
O
P
O^{i}Pr
O^{i}Pr
3n
7.55
7.50
7.45
7.40
ppm
2.041
2.028
4.8
ppm
2.000
1.40
ppm
12.362
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
2.041
2.028
2.000
12.362

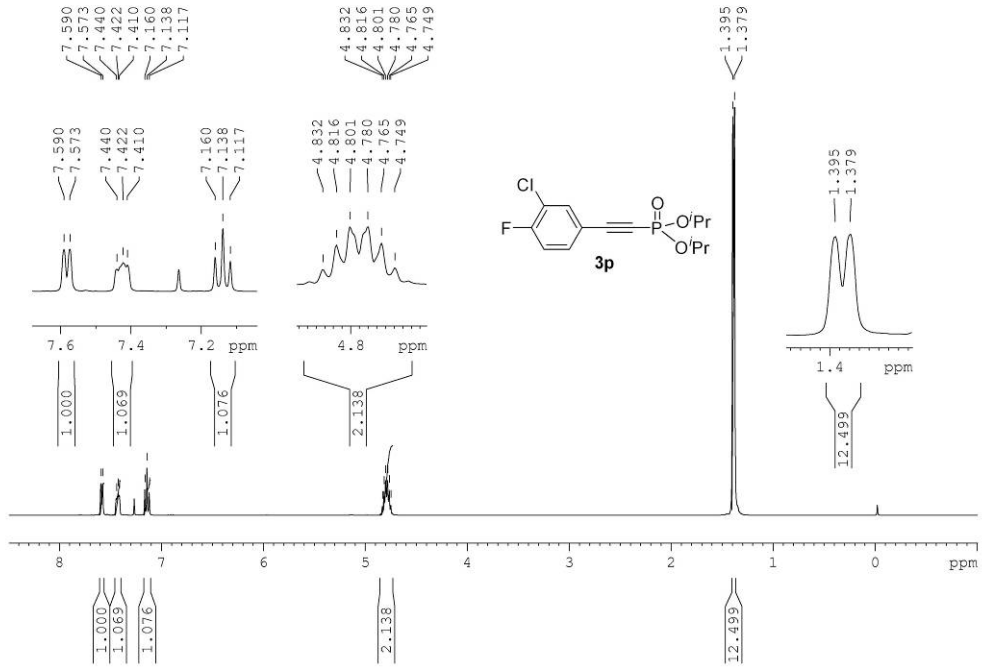

132.808
132.784
130.924
124.239
117.741
117.684
95.989
95.466
81.544
78.584
71.505
71.449
22.919
22.874
22.657
22.608
Br
O
P
O^{i}Pr
O^{i}Pr
3n
ppm
71.5
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

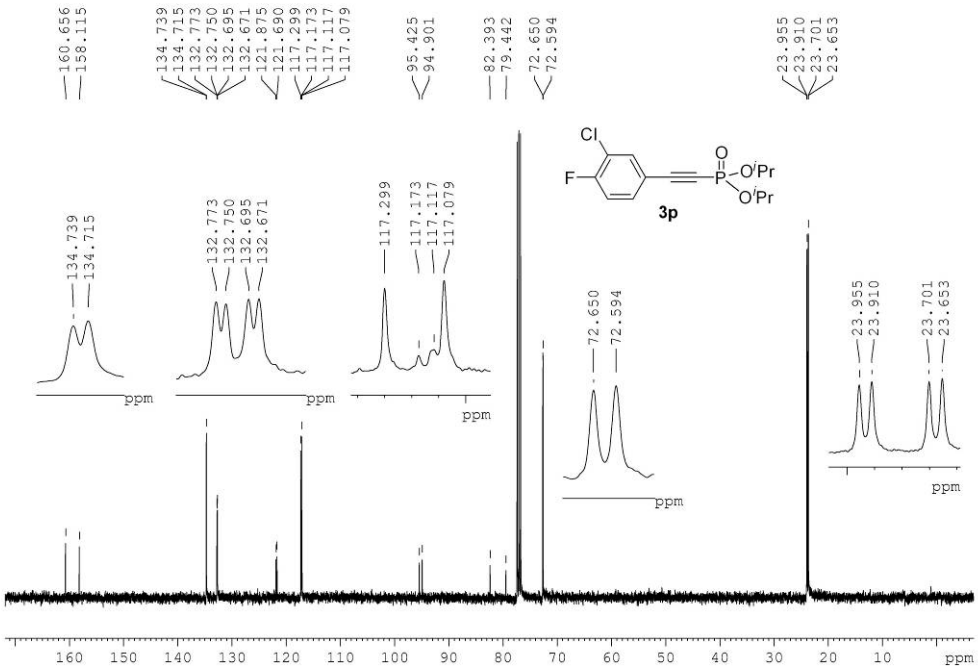

-8.457
Br
O
P
O^{i}Pr
O^{i}Pr
3n
300
250
200
150
100
50
0
-50
-100
ppm

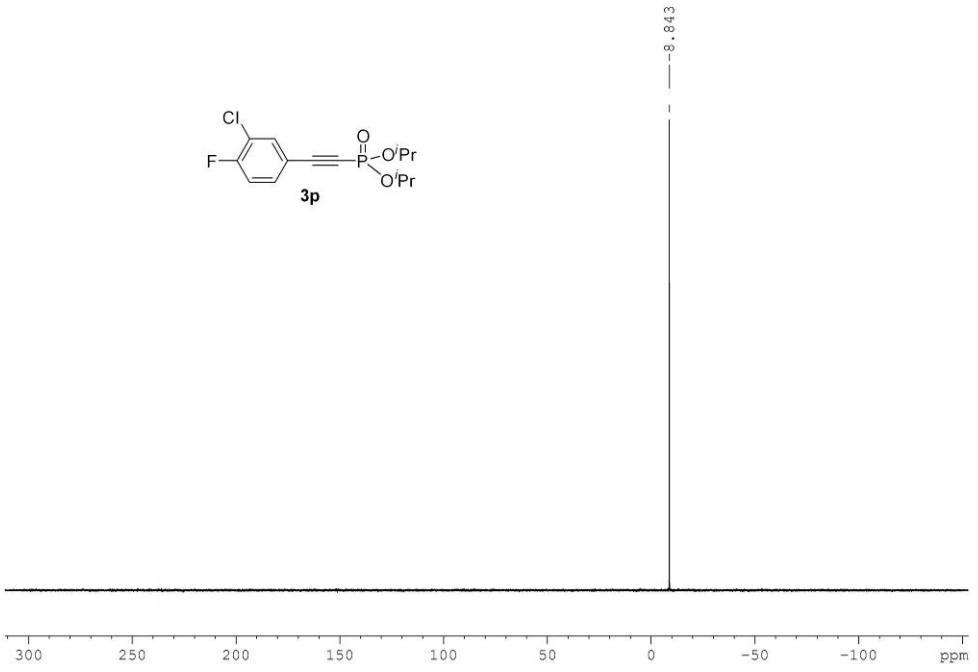

7.538
7.525
7.516
7.508
7.503
7.064
7.043
7.021
4.840
4.824
4.819
4.809
4.803
4.794
4.787
4.778
4.772
4.763
4.756
4.747
4.741
4.727
1.387
1.382
1.371
1.367
F
O
P
O^{i}Pr
O^{i}Pr
3o
7.5
7.4
7.3
7.2
7.1
ppm
4.8
ppm
1.40
ppm
8
7
6
5
4
3
2
1
0
ppm
2.000
2.000
2.017
12.354

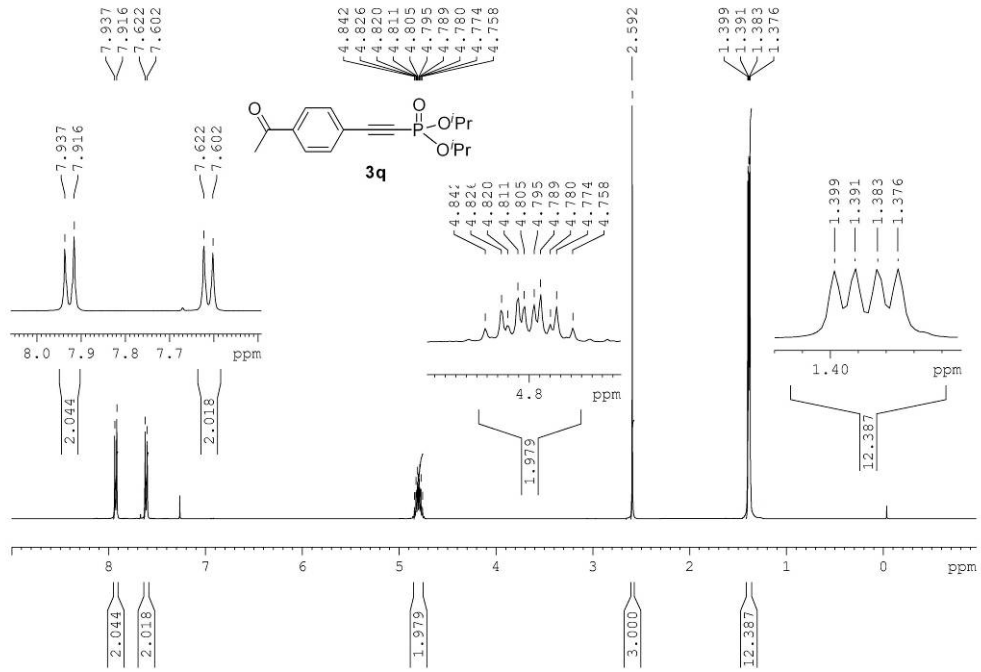

165.043
162.525
134.819
134.795
134.731
134.707
116.219
115.996
115.948
115.911
97.277
96.749
81.271
78.299
72.472
72.417
23.951
23.905
23.688
23.639
F
O
P
O^{i}Pr
O^{i}Pr
3o
ppm
ppm
72.5
ppm
23.8
ppm
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
ppm

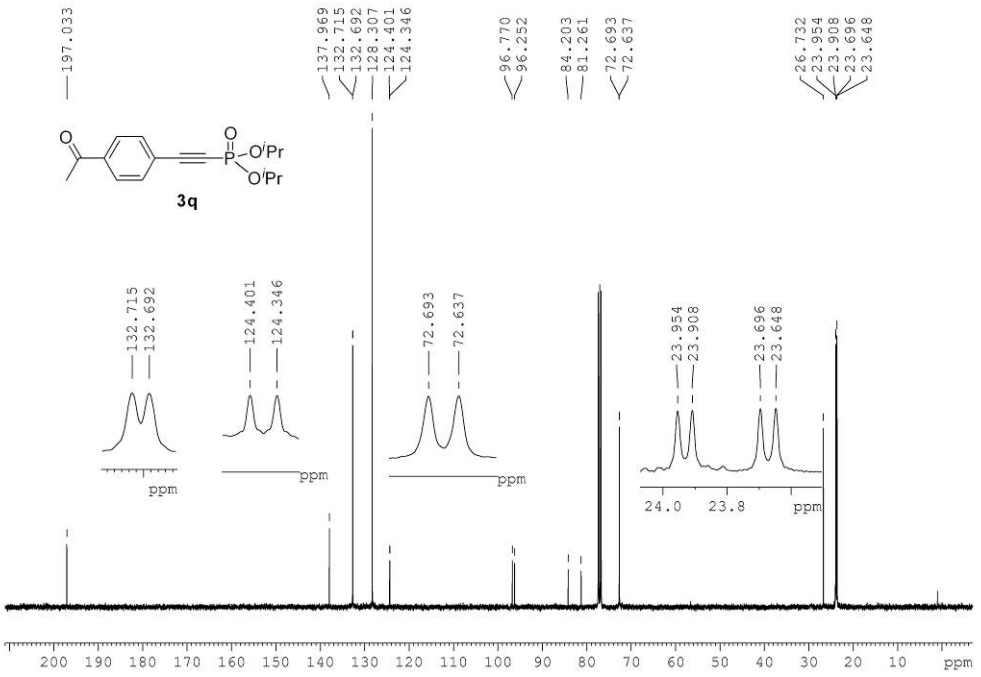

8.253
F
O
P
O^{i}Pr
O^{i}Pr
3o
300
250
200
150
100
50
0
-50
-100
ppm

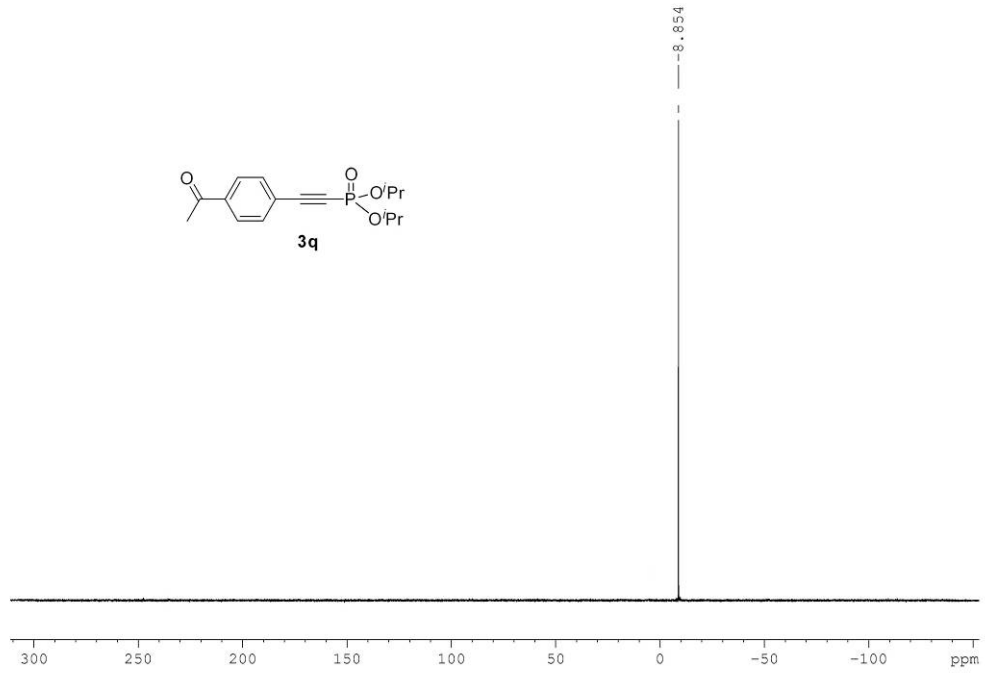

7.590
7.573
7.440
7.422
7.410
7.160
7.138
7.117
4.832
4.816
4.801
4.780
4.765
4.749
1.395
1.379
Cl
F
O
P
O^{i}Pr
O^{i}Pr
3p
7.6
7.4
7.2
ppm
4.8
ppm
1.4
ppm
1.000
1.069
1.076
2.138
12.499
8
7
6
5
4
3
2
1
0
ppm

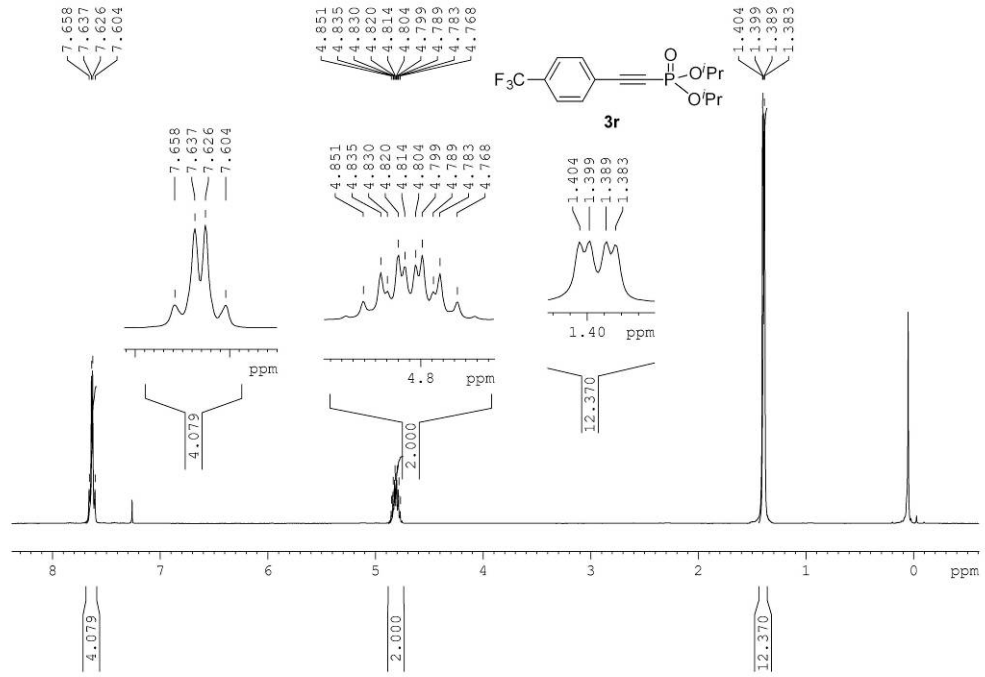

160.656
158.115
134.739
134.715
132.773
132.750
132.695
132.671
121.875
121.690
117.299
117.173
117.117
117.079
95.425
94.901
82.393
79.442
72.650
72.594
23.955
23.910
23.701
23.653
Cl
F
O
P
O^{i}Pr
O^{i}Pr
3p
ppm
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10

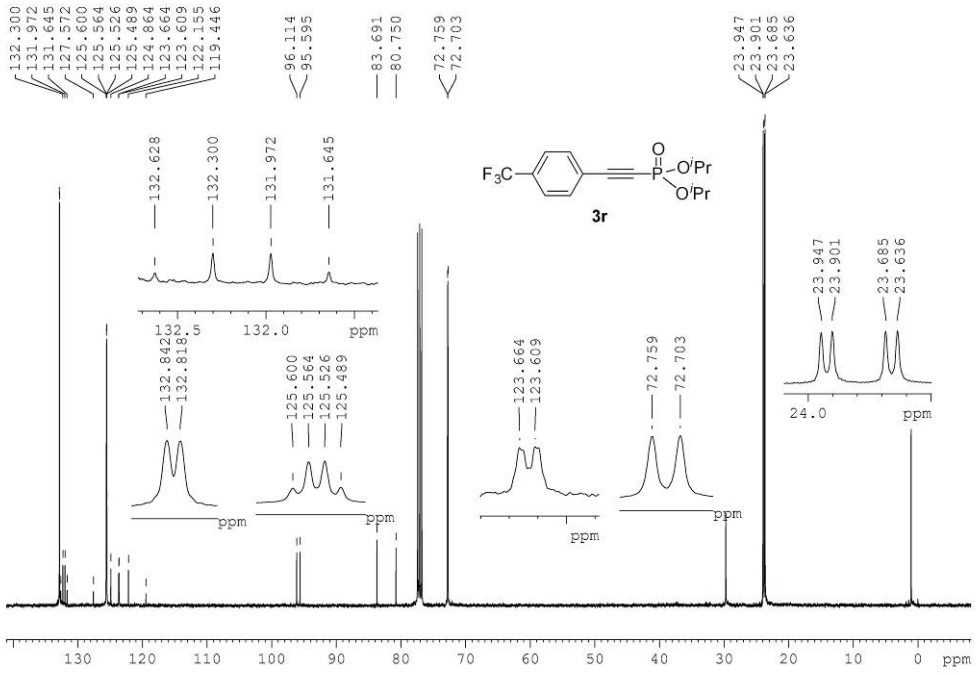

-8.843
Cl
F
O
P
O^{i}Pr
O^{i}Pr
3p
300
250
200
150
100
50
0
-50
-100
ppm

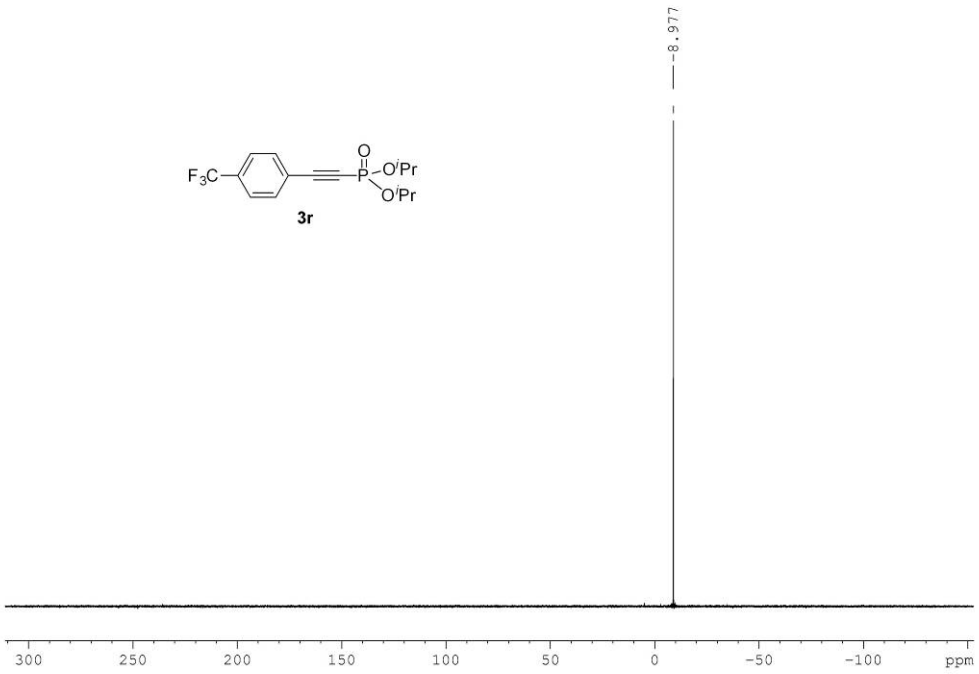

7.937
7.916
7.622
7.602
4.842
4.826
4.820
4.811
4.805
4.795
4.789
4.780
4.774
4.758
2.592
1.399
1.391
1.383
1.376
3q
8.0 7.9 7.8 7.7 ppm
4.8 ppm
1.40 ppm
2.044
2.018
1.979
12.387
3.000
8 7 6 5 4 3 2 1 0 ppm

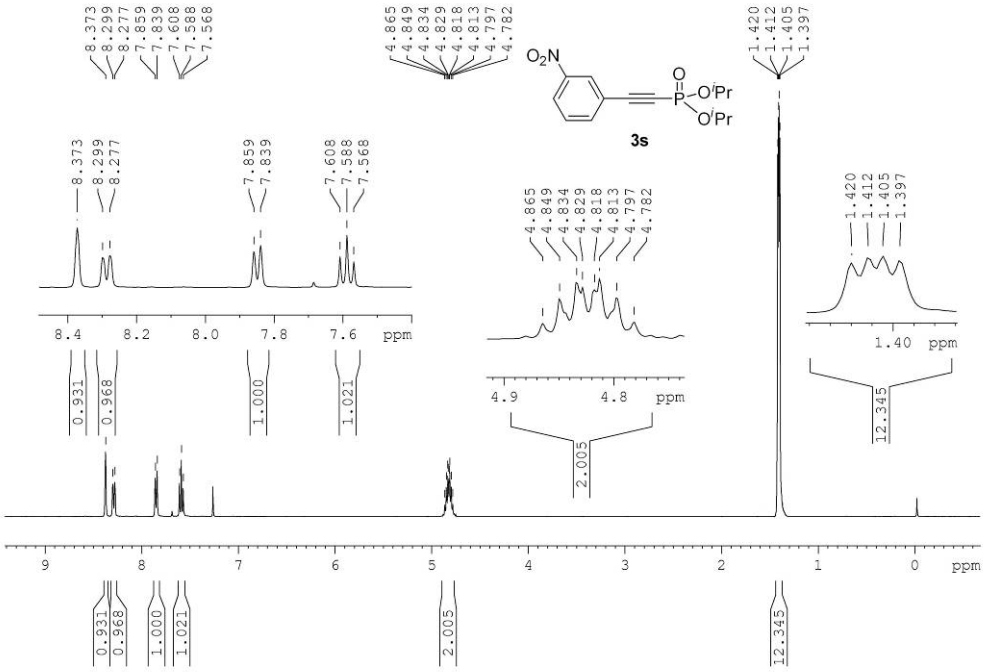

197.033
137.969
132.715
132.692
128.307
124.401
124.346
96.770
96.252
84.203
81.261
72.693
72.637
26.732
23.954
23.908
23.696
23.648
3q
ppm
24.0 23.8 ppm
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

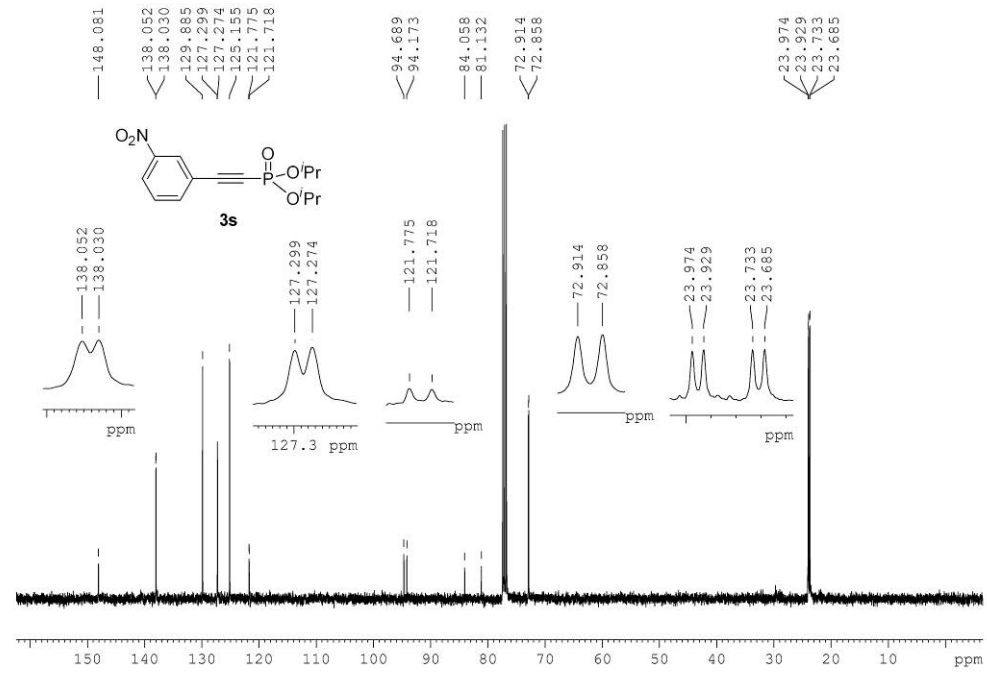

8.854
O
O
P
O^{i}Pr
O^{i}Pr
3q
300
250
200
150
100
50
0
−50
−100
ppm

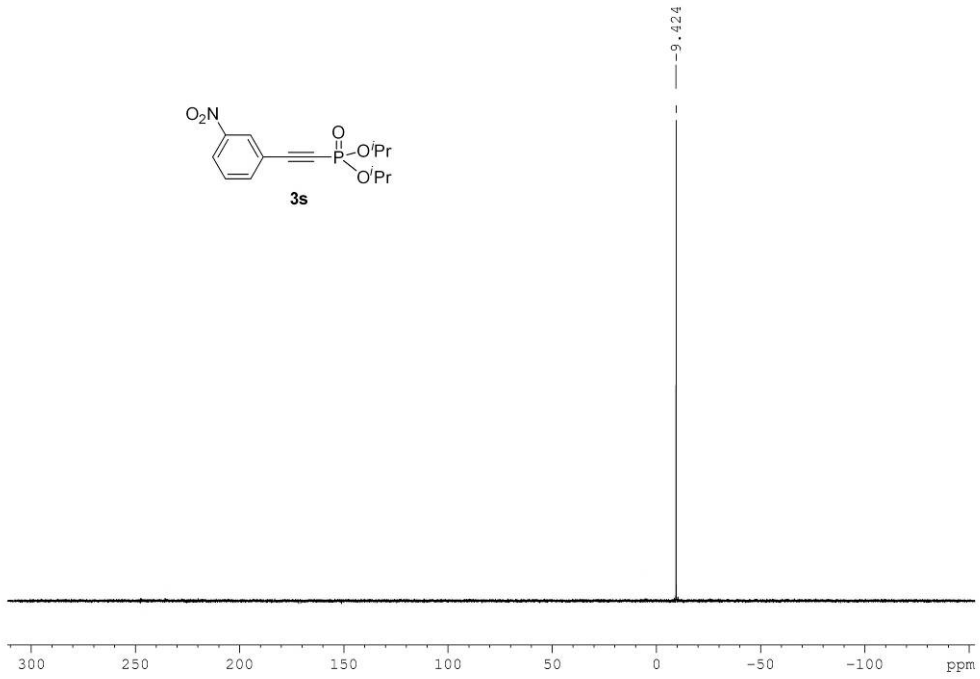

7.658
7.637
7.626
7.604
4.851
4.835
4.830
4.820
4.814
4.804
4.799
4.789
4.783
4.768
1.404
1.399
1.389
1.383
F$_3$C
O
P
O^{i}Pr
O^{i}Pr
3r
1.40
ppm
4.8
ppm
4.079
2.000
12.370
8
7
6
5
4
3
2
1
0
ppm

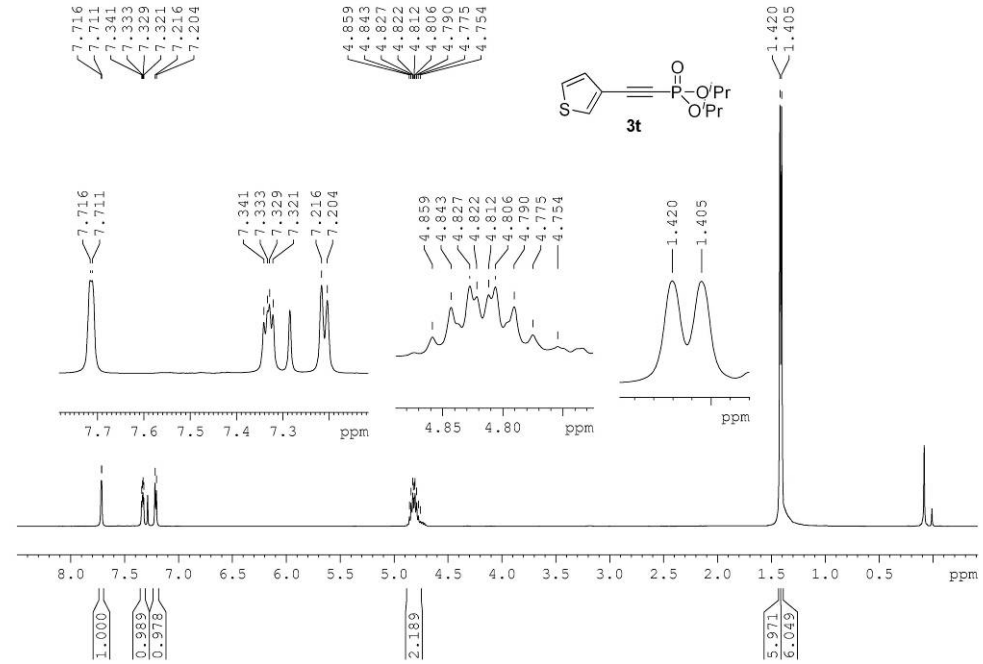

132.300
131.972
131.645
127.572
125.600
125.564
125.526
125.489
124.864
123.664
123.609
122.155
119.446
96.114
95.595
83.691
80.750
72.759
72.703
23.947
23.901
23.685
23.636
132.628
132.300
131.972
131.645
132.5
132.0
ppm
F3C
O
P
O^iPr
O^iPr
3r
23.947
23.901
23.685
23.636
24.0
ppm
132.842
132.818
ppm
125.600
125.564
125.526
125.489
ppm
123.664
123.609
ppm
72.759
72.703
ppm
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

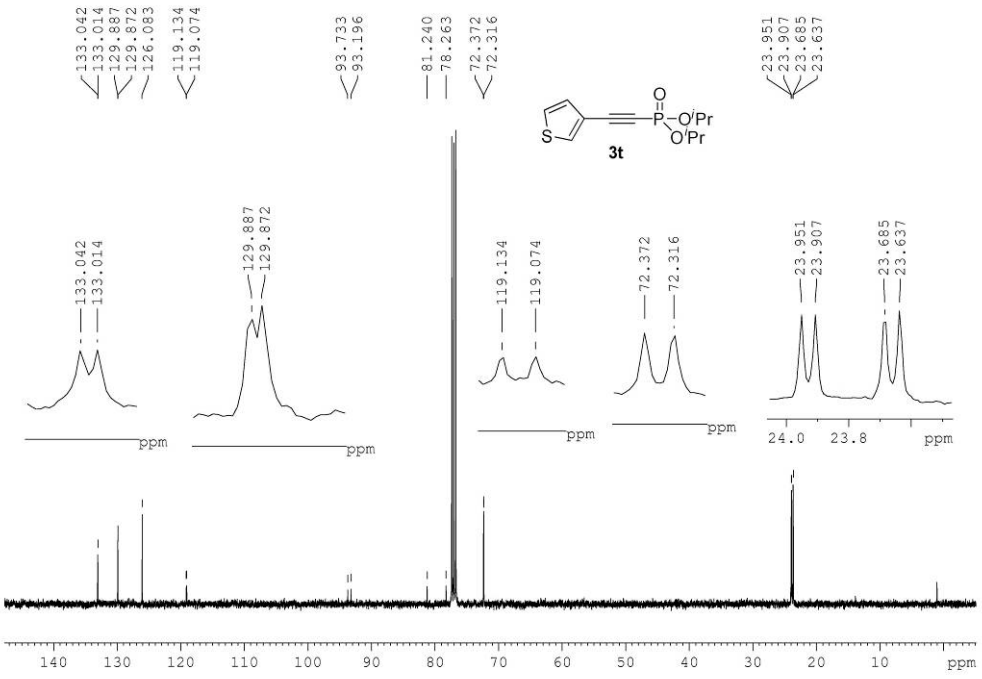

-8.977
F3C
O
P
O^iPr
O^iPr
3r
300
250
200
150
100
50
0
-50
-100
ppm

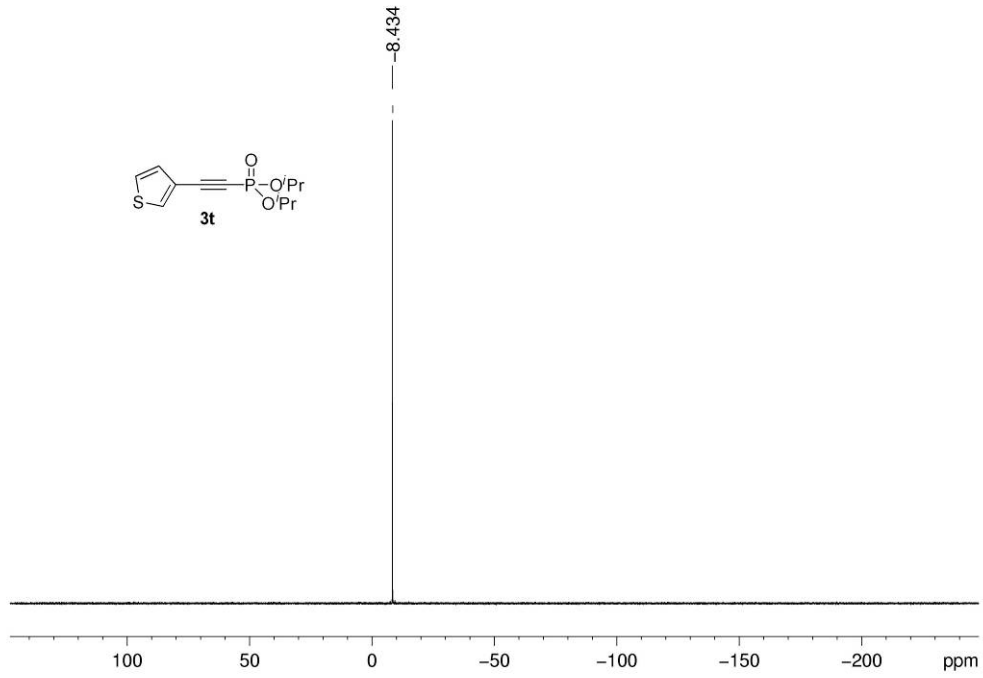

8.373
8.299
8.277
7.859
7.839
7.608
7.588
7.568
4.865
4.849
4.834
4.829
4.818
4.813
4.797
4.782
1.420
1.412
1.405
1.397
O2N
O
P
O iPr
O iPr
3s
ppm
0.931
0.968
1.000
1.021
2.005
12.345

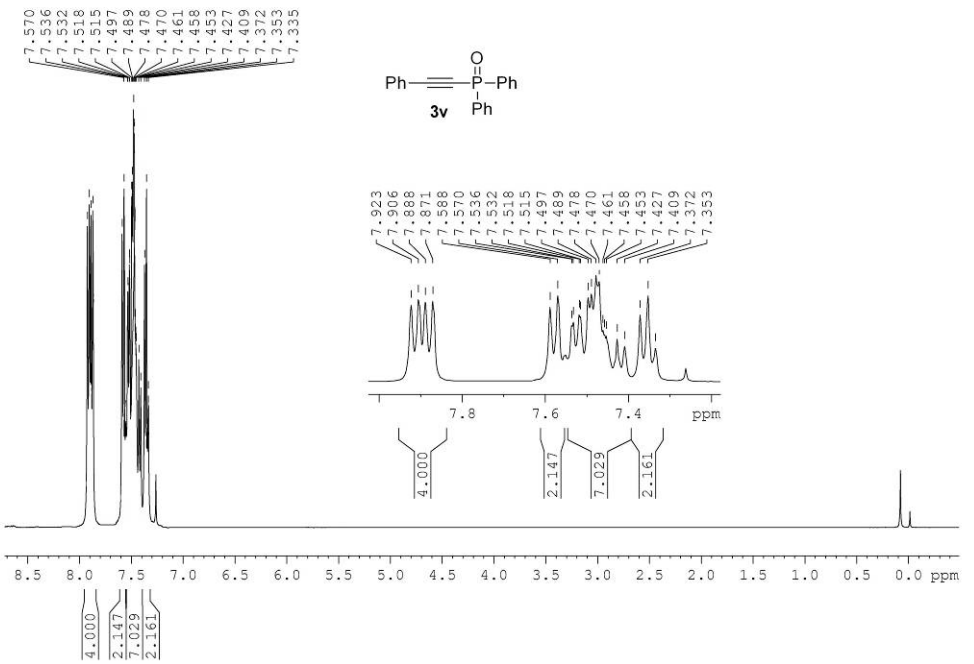

148.081
138.052
138.030
129.885
127.299
127.274
125.155
121.775
121.718
94.689
94.173
84.058
81.132
72.914
72.858
23.974
23.929
23.733
23.685
O2N
O
P
O iPr
O iPr
3s
127.3 ppm
ppm

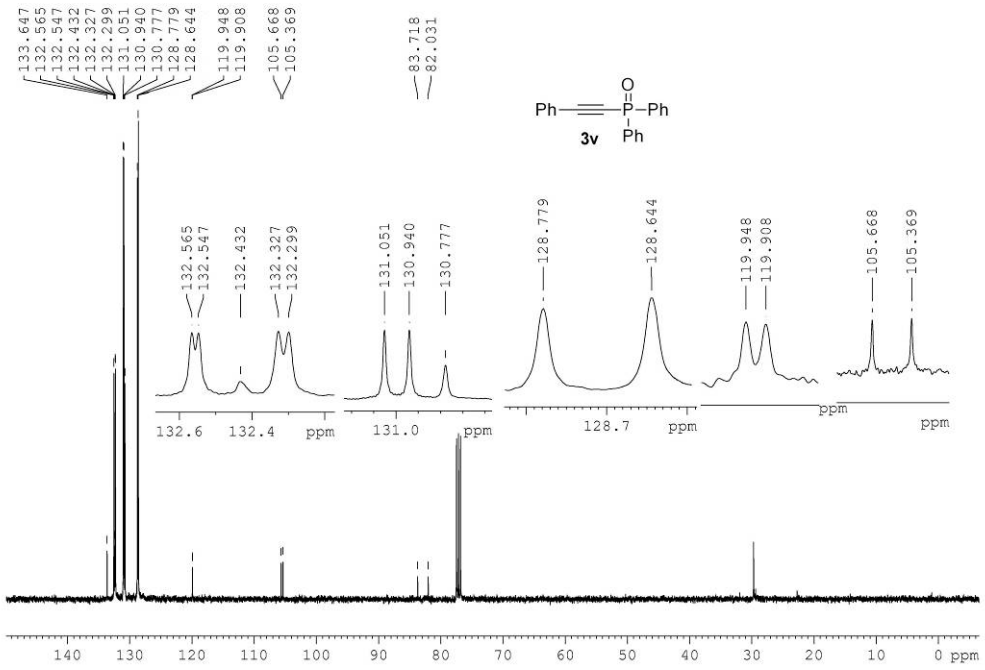

-9.424
O2N
O
P
O^{i}Pr
O^{i}Pr
3s
300
250
200
150
100
50
0
-50
-100
ppm

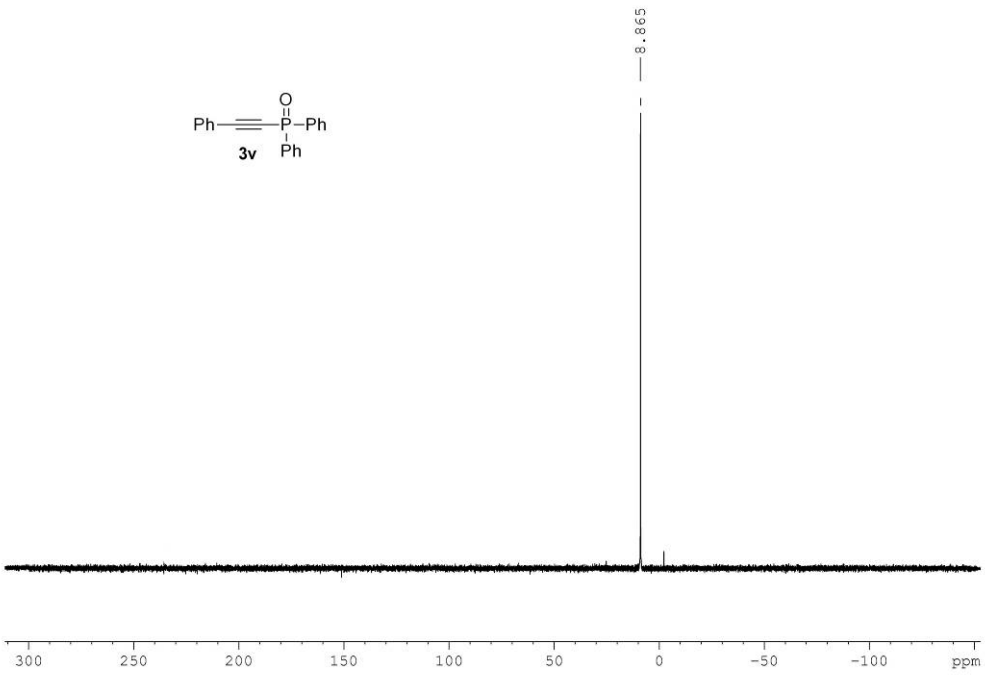

7.716
7.711
7.341
7.333
7.329
7.321
7.216
7.204
4.859
4.843
4.827
4.822
4.812
4.806
4.790
4.775
4.754
1.420
1.405
S
O
P
O^{i}Pr
O^{i}Pr
3t
7.7
7.6
7.5
7.4
7.3
4.85
4.80
ppm
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
1.000
0.989
0.978
2.189
5.971
6.049


133.042
133.014
129.887
129.872
126.083
119.134
119.074
93.733
93.196
81.240
78.263
72.372
72.316
23.951
23.907
23.685
23.637
O
P
O^{i}Pr
O^{i}Pr
S
3t
ppm
24.0
23.8
140
130
120
110
100
90
80
70
60
50
40
30
20
10


−8.434
O
P
O^{i}Pr
O^{i}Pr
S
3t
100
50
0
−50
−100
−150
−200
ppm


7.570
7.536
7.532
7.518
7.515
7.497
7.489
7.478
7.470
7.461
7.458
7.453
7.427
7.409
7.372
7.353
7.335
Ph
O
P
Ph
Ph
3v
7.923
7.906
7.888
7.871
7.588
7.570
7.536
7.532
7.518
7.515
7.497
7.489
7.478
7.470
7.461
7.458
7.453
7.427
7.409
7.372
7.353
7.8
7.6
7.4
ppm
4.000
2.147
7.029
2.161
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
4.000
2.147
7.029
2.161


133.647
132.565
132.547
132.432
132.327
132.299
131.051
130.940
130.777
128.779
128.644
119.948
119.908
105.668
105.369
83.718
82.031
Ph
O
P
Ph
Ph
3v
132.565
132.547
132.432
132.327
132.299
131.051
130.940
130.777
128.779
128.644
119.948
119.908
105.668
105.369
132.6
132.4
ppm
131.0
ppm
128.7
ppm
ppm
ppm
140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.865
O
Ph
P
Ph
Ph
3v
300
250
200
150
100
50
0
−50
−100
ppm