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Supporting Information

Pd-catalysed Suzuki coupling of α-bromo ethenylphosphonates with organotrifluoroborates: a general protocol for the synthesis of terminal α-substituted vinylphosphonates

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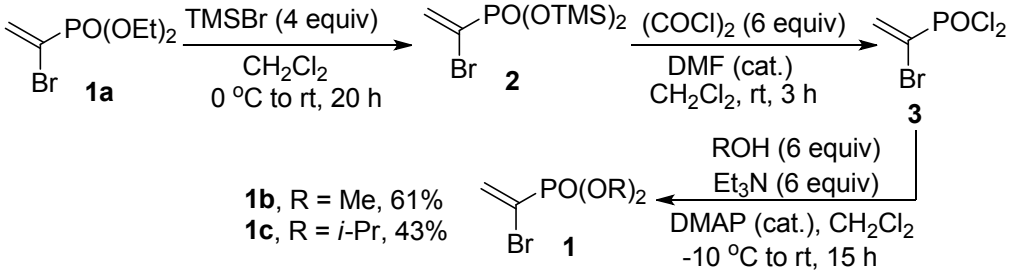
Table of contents

General information

All reactions were carried out under an atmosphere of nitrogen in oven-dried glassware otherwise mentioned elsewhere. Toluene was distilled from calcium hydride. Potassium organotrifluoroborates[1] and diethyl 1-bromovinylphosphonate[2] were synthesized according to the reported procedure. All organoboronic acids and other reagents were obtained from commercial suppliers and used without further purification. NMR spectra were recorded in $CDCl_3$ on a Bruker Avance 500 spectrometer. High resolution mass spectra (HRMS) were recorded on ESI-Q-TOF spectrometer (Bruker micrOTOF-Q II).

General Procedure A.[3] **Synthesis of dialkyl 1-bromovinylphosphonates 1b-c from diethyl 1-bromovinylphosphonate 1a**



Diethyl (1-bromovinyl)phosphonate **1a** (2.43 g, 10 mmol) was dissolved in CH_2Cl_2 (30 mL) and cooled to 0 °C. Then, bromotrimethylsilane (5.3 mL, 40 mmol) was added dropwise. After 2 h at 0 °C, the mixture was warmed to rt and stirred overnight. The volatile compounds were evaporated and the residue containing compound bis(trimethylsilyl) (1-bromovinyl)phosphonate **2** was used for the next step without further purification.

The crude bis(trimethylsilyl) (1-bromovinyl)phosphonate **2** was dissolved in CH_2Cl_2 (15 mL) and a few drops of DMF. With vigorous stirring, oxalyl chloride (5.1 mL, 60 mmol) was added dropwise. After the addition was completed, the mixture was stirred for 3 h at rt. Subsequently, the solvents were evaporated, resulting in brownish oil of crude (1-bromovinyl)phosphonic dichloride **3**.

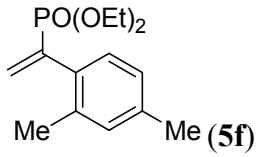
Alcohol (60 mmol) and triethylamine (8.3 mL, 60 mmol) were dissolved in CH_2Cl_2 (15 mL) containing 4-(dimethylamino)pyridine (DMAP) (24.2 mg, 0.2 mmol). The solution was stirred at -10 °C for 30 min. The crude (1-bromovinyl)phosphonic dichloride **3** was dissolved in CH_2Cl_2 (8 mL) and added dropwise. After the addition was completed, the mixture was warmed to rt and stirred overnight. Subsequently, the solvents were evaporated and the crude product was purified by chromatography on silica gel with petroleum ether/EtOAc (1/1, v/v) as the eluent to yield the product as a pale yellow oil.

General procedure B. Synthesis of 5a-r, 5u-y, 7, 9, 12, 13, 15, 16 via Suzuki coupling of *α*-bromvinylphosphonates with potassium organotrifluoroborates

A Schlenk flask was loaded with *α*-bromvinylphosphonate (97.2 mg, 0.4 mmol), $Pd_2(dba)_3$ (7.3 mg or 18.3 mg, 2 mol % or 5 mol %), SPhos (13.1 mg or 32.8 mg, 8 mol % or 20 mol %), potassium organotrifluoroborate (0.6 mmol), Cs_2CO_3 (261 mg, 0.8 mmol) and held under vacuum for 10 min and filled with nitrogen. Then toluene/water (4/1, v/v) (2.5 mL) was introduced and the mixture was stirred at rt-90 °C for 10-20 h. Upon completion of the reaction, the resulting mixture was cooled down to rt and extracted with EtOAc (4 × 10 mL). The combined organic phase was

dried over anhydrous $MgSO_4$. After the removal of solvent under reduced pressure, the crude product was purified by column chromatography on silica gel with petroleum ether/EtOAc (1/1, v/v) as the eluent to yield the product usually as a pale yellow oil.

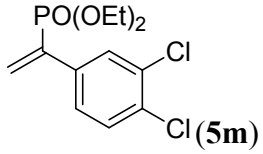
Diethyl (1-(2,4-dimethylphenyl)vinyl)phosphonate (**5f**)



(5f)

The compound **5f** was synthesized from general procedure B in the presence of 2 mol % of $Pd_2(dba)_3$ and 8 mol % of SPhos at 60 °C for 15 h. The title compound **5f** was isolated as pale yellow oil (106.1 mg, 99% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.27 (t, *J* = 7.1 Hz, 6H), 2.27 (s, 3H), 2.31 (s, 3H), 4.03-4.10 (m, 4H), 5.81 (dd, *J* = 47.8 Hz, 2.1 Hz, 1H), 6.41 (dd, *J* = 22.5 Hz, 2.1 Hz, 1H), 6.97 (d, *J* = 7.8 Hz, 1H), 7.03 (s, 1H), 7.07 (dd, *J* = 7.8 Hz, 1.5 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 16.2 (d, *J* = 6.3 Hz), 19.8, 21.0, 62.1 (d, *J* = 6.3 Hz), 126.0 (d, *J* = 1.5 Hz), 128.9 (d, *J* = 3.7 Hz), 131.0, 132.9 (d, *J* = 8.7 Hz), 133.6 (d, *J* = 9.9 Hz), 135.8 (d, *J* = 5.4 Hz), 137.4 (d, *J* = 1.8 Hz), 139.9 (d, *J* = 175.3 Hz); ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 16.2; IR (KBr): ν (cm^{-1}) 3545, 2982, 2928, 1259, 1236, 1051, 1025, 965; GC-MS: *m*/*z* (rel intensity) 268 (M^+, 16), 239 (8), 211 (58), 193 (3), 157 (4), 129 (100), 115 (34), 91 (16); HRMS (ESI) calcd for $C_{14}H_{21}NaO_3P$ $[M+Na]^+$ 291.1121, found 291.1136.

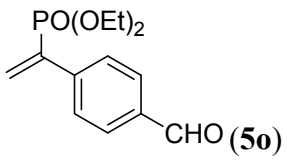
Diethyl (1-(3,4-dichlorophenyl)vinyl)phosphonate (**5m**)



(5m)

The compound **5m** was synthesized from general procedure B in the presence of 2 mol % of $Pd_2(dba)_3$ and 8 mol % of SPhos at 60 °C for 15 h. The title compound **5m** was isolated as pale yellow oil (121.9 mg, 99% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.31 (t, *J* = 7.1 Hz, 6H), 4.07-4.17 (m, 4H), 6.15 (dd, *J* = 44.9 Hz, 1.1 Hz, 1H), 6.36 (dd, *J* = 21.8 Hz, 1.1 Hz, 1H), 7.37-7.43 (m, 2H), 7.61-7.62 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 16.2 (d, *J* = 6.0 Hz), 62.4 (d, *J* = 5.6 Hz), 126.8 (d, *J* = 5.3 Hz), 129.3 (d, *J* = 6.1 Hz), 130.3, 132.4, 132.5, 132.6 (d, *J* = 7.8 Hz), 136.6 (d, *J* = 12.0 Hz), 138.0 (d, *J* = 176.8 Hz); ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 15.7; IR (KBr): ν (cm^{-1}) 3545, 2983, 2906, 1472, 1392, 1255, 1235, 1050, 1024, 968; GC-MS: *m*/*z* (rel intensity) 308 (M^+, 15), 280 (29), 236 (23), 199 (24), 171 (69), 163 (75), 136 (100), 109 (26), 81 (28); HRMS (ESI) calcd for $C_{12}H_{15}Cl_2NaO_3P$ $[M+Na]^+$ 331.0028, found 331.0028.

Diethyl (1-(4-formylphenyl)vinyl)phosphonate (**5o**)

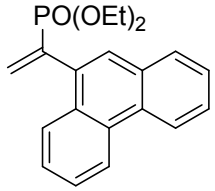


(5o)

The compound **5o** was synthesized from general procedure B in the presence of 2 mol % of $Pd_2(dba)_3$ and 8 mol % of SPhos at 60 °C for 15 h. The title compound **5o** was isolated as pale

yellow oil (99.7 mg, 93% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.30 (t, *J* = 7.1 Hz, 6H), 4.06-4.19 (m, 4H), 6.24 (d, *J* = 45.0 Hz, 1H), 6.44 (d, *J* = 22.0 Hz, 1H), 7.70 (d, *J* = 7.7 Hz, 2H), 7.87 (d, *J* = 8.0 Hz, 2H), 10.03 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 16.2 (d, *J* = 6.1 Hz), 62.4 (d, *J* = 5.7 Hz), 128.1 (d, *J* = 5.6 Hz), 129.7, 133.3 (d, *J* = 7.3 Hz), 135.9, 139.2 (d, *J* = 175.8 Hz), 142.7 (d, *J* = 11.7 Hz), 191.6; ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 15.8; IR (KBr): ν (cm^{-1}) 3474, 2983, 2837, 1702, 1606, 1392, 1235, 1172, 1023, 969; GC-MS: *m*/*z* (rel intensity) 268 (M^+, 58), 240 (56), 211 (26), 196 (23), 158 (53), 131 (100), 103 (54), 91 (17); HRMS (ESI) calcd for $C_{13}H_{17}NaO_4P$ $[M+Na]^+$ 291.0757, found 291.0756.

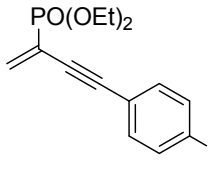
Diethyl (1-(phenanthren-9-yl)vinyl)phosphonate (**5v**)



(**5v**)

The compound **5v** was synthesized from general procedure B in the presence of 2 mol % of $Pd_2(dba)_3$ and 8 mol % of SPhos at 60 °C for 15 h. The title compound **5v** was isolated as pale yellow oil (131.9 mg, 97% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.20 (t, *J* = 7.1 Hz, 6H), 4.05-4.10 (m, 4H), 6.09 (dd, *J* = 47.0 Hz, 2.0 Hz, 1H), 6.70 (dd, *J* = 22.5 Hz, 2.0 Hz, 1H), 7.60-7.62 (m, 2H), 7.65-7.69 (m, 3H), 7.88 (d, *J* = 7.8 Hz, 1H), 8.08 (d, *J* = 8.2 Hz, 1H), 8.69 (d, *J* = 8.3 Hz, 1H), 8.73 (d, *J* = 8.2 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 16.2 (d, *J* = 6.2 Hz), 62.4 (d, *J* = 6.3 Hz), 122.4, 122.7, 126.4, 126.5, 126.6, 126.7, 126.8, 127.3 (d, *J* = 5.5 Hz), 128.7, 130.1, 130.5, 130.6 (d, *J* = 4.3 Hz), 131.0 (d, *J* = 1.7 Hz), 133.2 (d, *J* = 9.2 Hz), 134.6 (d, *J* = 7.9 Hz), 139.1 (d, *J* = 177.2 Hz); ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 15.6; IR (KBr): ν (cm^{-1}) 3527, 2982, 2905, 1450, 1254, 1052, 1024, 966; GC-MS: *m*/*z* (rel intensity) 340 (M^+, 4), 283 (4), 265 (2), 202 (100), 176 (3), 142 (2), 101 (3), 81 (2); HRMS (ESI) calcd for $C_{20}H_{21}NaO_3P$ $[M+Na]^+$ 363.1121, found 363.1121.

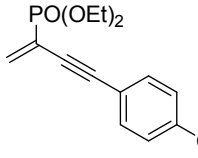
Diethyl (4-(p-tolyl)but-1-en-3-yn-2-yl)phosphonate (**9b**)



(**9b**)

The compound **9b** was synthesized from general procedure B in the presence of 5 mol % of $Pd_2(dba)_3$ and 20 mol % of SPhos at 50 °C for 10 h. The title compound **9b** was isolated as pale yellow oil (91.2 mg, 82% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.37 (t, *J* = 7.0 Hz, 6H), 2.35 (s, 3H), 4.13-4.24 (m, 4H), 6.31 (dd, *J* = 43.9 Hz, 1.8 Hz, 1H), 6.46 (dd, *J* = 20.7 Hz, 1.8 Hz, 1H), 7.13 (d, *J* = 7.9 Hz, 2H), 7.39-7.41 (d, *J* = 8.0 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 16.2 (d, *J* = 6.3 Hz), 21.4, 62.8 (d, *J* = 5.8 Hz), 83.8 (d, *J* = 10.9 Hz), 94.0 (d, *J* = 8.4 Hz), 119.2 (d, *J* = 2.6 Hz), 122.7 (d, *J* = 185.6 Hz), 129.0, 131.4 (d, *J* = 2.5 Hz), 137.8 (d, *J* = 6.6 Hz), 139.0; ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 12.7; IR (KBr): ν (cm^{-1}) 3545, 2983, 2198, 1508, 1392, 1253, 1165, 1023, 970; GC-MS: *m*/*z* (rel intensity) 278 (M^+, 9), 249 (7), 233 (17), 207 (13), 170 (17), 142 (100), 115 (62), 91 (18); HRMS (ESI) calcd for $C_{15}H_{19}NaO_3P$ $[M+Na]^+$ 301.0964, found 301.0969.

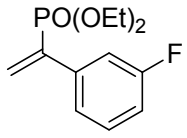
Diethyl (4-(4-methoxyphenyl)but-1-en-3-yn-2-yl)phosphonate (9c)



(9c)

The compound **9c** was synthesized from general procedure B in the presence of 5 mol % of $Pd_2(dba)_3$ and 20 mol % of SPhos at 50 °C for 10 h. The title compound **9c** was isolated as pale yellow oil (91.7 mg, 78% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.38 (td, *J* = 7.1 Hz, 0.4 Hz, 6H), 3.82 (s, 3H), 4.15-4.23 (m, 4H), 6.30 (dd, *J* = 43.9 Hz, 2.0 Hz, 1H), 6.44 (dd, *J* = 20.7 Hz, 2.0 Hz, 1H), 6.85-6.87 (m, 2H), 7.39-7.42 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 16.3 (d, *J* = 6.3 Hz), 55.3, 62.9 (d, *J* = 5.7 Hz), 83.4 (d, *J* = 10.9 Hz), 94.0 (d, *J* = 8.4 Hz), 114.0 (d, *J* = 4.4 Hz), 114.5 (d, *J* = 2.7 Hz), 122.8 (d, *J* = 185.4 Hz), 133.2 (d, *J* = 2.6 Hz), 137.5 (d, *J* = 6.5 Hz), 160.0; ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 12.9; IR (KBr): ν (cm^{-1}) 3545, 2983, 2196, 1606, 1509, 1299, 1250, 1026, 972; GC-MS: *m*/*z* (rel intensity) 294 (M^+, 14), 265 (8), 251 (9), 206 (10), 184 (7), 158 (100), 142 (21), 114 (44), 81 (21); HRMS (ESI) calcd for $C_{15}H_{19}NaO_4P$ $[M+Na]^+$ 317.0913, found 317.0912.

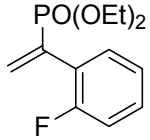
Diethyl (1-(3-fluorophenyl)vinyl)phosphonate (12b)



(12b)

The compound **12b** was synthesized from general procedure B in the presence of 2 mol % of $Pd_2(dba)_3$ and 8 mol % of SPhos at rt for 15 h. The title compound **12b** was isolated as pale yellow oil (92.9 mg, 90% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.28 (t, *J* = 7.1 Hz, 6H), 4.05-4.15 (m, 4H), 6.16 (dd, *J* = 45.2 Hz, 1.2 Hz, 1H), 6.35 (dd, *J* = 21.9 Hz, 1.1 Hz, 1H), 7.00-7.02 (m, 1H), 7.22-7.25 (m, 1H), 7.29-7.31 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 16.2 (d, *J* = 6.1 Hz), 62.3 (d, *J* = 5.8 Hz), 114.3-114.5 (m), 115.1 (d, *J* = 21.1 Hz), 123.1 (m), 129.9 (d, *J* = 8.3 Hz), 132.4 (d, *J* = 7.6 Hz), 138.6-138.8 (m), 138.8 (d, *J* = 175.4 Hz), 162.5 (d, *J* = 244.4 Hz); ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 16.3; ^{19}F NMR (470 MHz, $CDCl_3$): δ -112.8; IR (KBr): ν (cm^{-1}) 3566, 2984, 2907, 1580, 1486, 1243, 1050, 1023, 967; GC-MS: *m*/*z* (rel intensity) 258 (M^+, 11), 230 (24), 186 (11), 148 (52), 133 (11), 121 (88), 101 (100), 81 (24); HRMS (ESI) calcd for $C_{12}H_{16}FNaO_3P$ $[M+Na]^+$ 281.0713, found 281.0713.

Diethyl (1-(2-fluorophenyl)vinyl)phosphonate (12c)

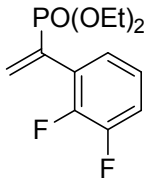


(12c)

The compound **12c** was synthesized from general procedure B in the presence of 2 mol % of $Pd_2(dba)_3$ and 8 mol % of SPhos at 60 °C for 15 h. The title compound **12c** was isolated as pale yellow oil (88.8 mg, 86% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.29 (t, *J* = 7.1 Hz, 6H), 4.05-4.16 (m, 4H), 6.15 (d, *J* = 46.1 Hz, 1H), 6.54 (d, *J* = 22.3 Hz, 1H), 7.07-7.14 (m, 2H), 7.29 (d, *J* =

6.1 Hz, 1H), 7.42-7.45 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 16.2 (d, *J* = 6.4 Hz), 62.3 (d, *J* = 5.8 Hz), 115.8 (d, *J* = 22.4 Hz), 123.8 (d, *J* = 3.5 Hz), 124.6 (m), 129.7 (dd, *J* = 8.1 Hz, 1.4 Hz, 1H), 130.7 (m), 133.8 (d, *J* = 178.5 Hz), 135.4 (m), 159.6 (dd, *J* = 7.0 Hz, 246.6 Hz); ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 15.6; ^{19}F NMR (470 MHz, $CDCl_3$): δ -115.5; IR (KBr): ν (cm^{-1}) 3566, 2983, 2907, 1491, 1260, 1239, 1050, 1024, 968; GC-MS: *m*/*z* (rel intensity) 258 (M^+, 26), 230 (32), 210 (24), 186 (51), 148 (32), 121 (64), 101 (100), 82 (19); HRMS (ESI) calcd for $C_{12}H_{16}FNaO_3P$ $[M+Na]^+$ 281.0713, found 281.0718.

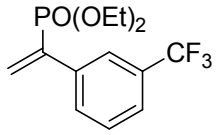
Diethyl (1-(2,3-difluorophenyl)vinyl)phosphonate (**12d**)



(**12d**)

The compound **12d** was synthesized from general procedure B in the presence of 5 mol % of $Pd_2(dba)_3$ and 20 mol % of SPhos at 60 °C for 10 h. The title compound **12d** was isolated as pale yellow oil (96.0 mg, 87% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.29 (t, *J* = 7.0 Hz, 6H), 4.08-4.15 (m, 4H), 6.16 (d, *J* = 45.6 Hz, 1H), 6.57 (d, *J* = 22.2 Hz, 1H), 7.05-7.07 (m, 1H), 7.09-7.14 (m, 1H), 7.18-7.20 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 16.2 (d, *J* = 6.3 Hz), 62.4 (d, *J* = 5.7 Hz), 116.8 (d, *J* = 16.9 Hz), 123.6-123.7 (m), 125.3 (m), 126.7 (t, *J* = 11.5 Hz), 132.9 (dd, *J* = 180.2 Hz, 2.1 Hz), 135.9 (m), 147.8 (dq, *J* = 248.3 Hz, 7.0 Hz), 150.6 (dd, *J* = 240.3 Hz, 13.3 Hz); ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 14.8; ^{19}F NMR (470 MHz, $CDCl_3$): δ -137.5 (d, *J* = 20.6 Hz), -141.0 (d, *J* = 20.8 Hz); IR (KBr): ν (cm^{-1}) 3481, 2985, 2909, 1589, 1475, 1393, 1241, 1023, 960; GC-MS: *m*/*z* (rel intensity) 276 (M^+, 18), 248 (34), 200 (61), 184 (26), 166 (26), 139 (75), 119 (100), 99 (49), 81 (43); HRMS (ESI) calcd for $C_{12}H_{16}F_2O_3P$ $[M+H]^+$ 277.0800, found 277.0799.

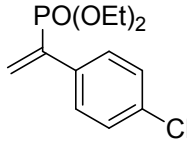
Diethyl (1-(3-(trifluoromethyl)phenyl)vinyl)phosphonate (**12e**)



(**12e**)

The compound **12e** was synthesized from general procedure B in the presence of 5 mol % of $Pd_2(dba)_3$ and 20 mol % of SPhos at 80 °C for 15 h. The title compound **12e** was isolated as pale yellow oil (89.9 mg, 73% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.30 (t, *J* = 7.0 Hz, 6H), 4.07-4.19 (m, 4H), 6.20 (d, *J* = 45.1 Hz, 1H), 6.41 (d, *J* = 21.9 Hz, 1H), 7.47-7.50 (m, 1H), 7.59 (d, *J* = 7.4 Hz, 1H), 7.73 (d, *J* = 7.4 Hz, 1H), 7.77 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 16.2 (d, *J* = 6.2 Hz), 62.4 (d, *J* = 5.9 Hz), 123.9 (d, *J* = 270.8 Hz), 124.3 (d, *J* = 3.6 Hz), 127.9 (d, *J* = 3.5 Hz), 128.9, 130.83 (d, *J* = 4.8 Hz), 130.84 (d, *J* = 32.2 Hz), 132.8 (d, *J* = 7.7 Hz), 137.6 (d, *J* = 11.9 Hz), 139.0 (d, *J* = 176.1 Hz); ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 15.9; ^{19}F NMR (470 MHz, $CDCl_3$): δ -62.8; IR (KBr): ν (cm^{-1}) 3482, 2986, 2909, 1331, 1232, 1167, 1127, 1023, 969; GC-MS: *m*/*z* (rel intensity) 308 (M^+, 13), 280 (23), 259 (13), 236 (14), 199 (14), 178 (67), 151 (100), 129 (33), 82 (33); HRMS (ESI) calcd for $C_{13}H_{17}F_3O_3P$ $[M+H]^+$ 309.0862, found 309.0853.

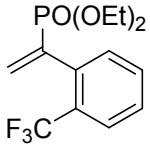
Diethyl (1-(4-(trifluoromethyl)phenyl)vinyl)phosphonate (**12f**)



(**12f**)

The compound **12f** was synthesized from general procedure B in the presence of 5 mol % of $Pd_2(dba)_3$ and 20 mol % of SPhos at 80 °C for 15 h. The title compound **12f** was isolated as pale yellow oil (114.6 mg, 93% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.30 (t, *J* = 7.1 Hz, 6H), 4.06-4.19 (m, 4H), 6.20 (dd, *J* = 45.1 Hz, 1.2 Hz, 1H), 6.41 (dd, *J* = 21.9 Hz, 1.3 Hz, 1H), 7.60-7.65 (m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 16.2 (d, *J* = 6.1 Hz), 62.4 (d, *J* = 5.8 Hz), 124.0 (d, *J* = 270.2 Hz), 125.3 (d, *J* = 3.6 Hz), 127.8 (d, *J* = 5.5 Hz), 130.2 (d, *J* = 32.1 Hz), 133.0 (d, *J* = 7.4 Hz), 139.0 (d, *J* = 175.8 Hz), 140.3 (d, *J* = 12.2 Hz); ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 15.9; ^{19}F NMR (470 MHz, $CDCl_3$): δ -62.7; IR (KBr): ν (cm^{-1}) 3545, 2985, 2908, 1327, 1238, 1170, 1123, 1019, 968; GC-MS: *m*/*z* (rel intensity) 308 (M^+, 11), 280 (28), 252 (6), 198 (22), 171 (70), 151 (100), 129 (50), 102 (29), 82 (33); HRMS (ESI) calcd for $C_{13}H_{16}F_3NaO_3P$ $[M+Na]^+$ 331.0681, found 331.0682.

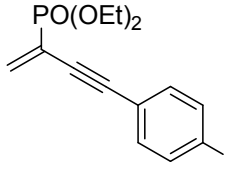
Diethyl (1-(2-(trifluoromethyl)phenyl)vinyl)phosphonate (**12g**)



(**12g**)

The compound **12g** was synthesized from general procedure B in the presence of 5 mol % of $Pd_2(dba)_3$ and 20 mol % of SPhos at 80 °C for 15 h. The title compound **12g** was isolated as pale yellow oil (109.6 mg, 89% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.28 (t, *J* = 7.1 Hz, 6H), 4.04-4.15 (m, 4H), 5.93 (d, *J* = 46.5 Hz, 1H), 6.44 (dd, *J* = 22.9 Hz, 1.1 Hz, 1H), 7.41-7.46 (m, 2H), 7.50-7.53 (m, 1H), 7.70 (d, *J* = 7.9 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 16.2 (d, *J* = 6.3 Hz), 62.4 (d, *J* = 6.2 Hz), 123.9 (d, *J* = 272.2 Hz), 126.4-126.5 (m), 127.8 (d, *J* = 1.6 Hz), 128.5-128.8 (m), 130.9 (d, *J* = 3.0 Hz), 131.0, 133.8 (m), 135.6 (d, *J* = 10.1 Hz), 137.6 (d, *J* = 181.0 Hz); ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 14.5; ^{19}F NMR (470 MHz, $CDCl_3$): δ -56.6; IR (KBr): ν (cm^{-1}) 3545, 2985, 1315, 1260, 1233, 1173, 1131, 1034, 969; GC-MS: *m*/*z* (rel intensity) 308 (M^+, 4), 268 (7), 240 (15), 200 (13), 183 (25), 151 (100), 129 (27), 101 (22), 81 (33); HRMS (ESI) calcd for $C_{13}H_{16}F_3NaO_3P$ $[M+Na]^+$ 331.0681, found 331.0698.

Diethyl (4-(4-fluorophenyl)but-1-en-3-yn-2-yl)phosphonate (**13**)



(**13**)

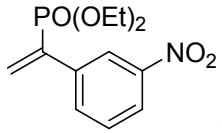
The compound **13** was synthesized from general procedure B in the presence of 5 mol % of $Pd_2(dba)_3$ and 20 mol % of SPhos at 50 °C for 10 h. The title compound **13** was isolated as pale yellow oil (90.2 mg, 80% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.37 (t, *J* = 7.1 Hz, 6H), 4.13-4.24 (m, 4H), 6.33 (dd, *J* = 43.7 Hz, 1.8 Hz, 1H), 6.48 (dd, *J* = 20.7 Hz, 1.8 Hz, 1H), 7.02 (t, *J* = 8.7 Hz, 2H), 7.42-7.45 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 16.3 (d, *J* = 6.3 Hz), 62.9 (d, *J* =

5.5 Hz), 84.2 (d, J = 12.2 Hz), 92.6 (d, J = 8.6 Hz), 115.7 (d, J = 22.3 Hz), 118.4-118.5 (m), 122.6 (d, J = 186.2 Hz), 133.5-133.6 (m), 138.3 (d, J = 6.3 Hz), 162.8 (d, J = 249.2 Hz); ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 12.6; ^{19}F NMR (470 MHz, $CDCl_3$): δ -109.8; IR (KBr): ν (cm^{-1}) 3482, 2984, 2202, 1601, 1507, 1392, 1251, 1157, 1023, 973; GC-MS: *m*/*z* (rel intensity) 282 (M^+, 5), 253 (3), 238 (17), 210 (12), 174 (10), 146 (100), 125 (44), 109 (7), 81 (18); HRMS (ESI) calcd for $C_{14}H_{16}FNaO_3P$ $[M+Na]^+$ 305.0713, found 305.0719.

General procedure C. Synthesis of 5s-t via Suzuki coupling of *α*-bromvinylphosphonates with potassium nitrophenyltrifluoroborates

A Schlenk flask was loaded with *α*-bromvinylphosphonate (145.8 mg, 0.6 mmol), $Pd_2(dba)_3$ (7.3 mg, 0.008 mmol), SPhos (13.1 mg, 0.032 mmol), potassium (3-nitrophenyl)trifluoroborate or potassium (4-nitrophenyl)trifluoroborate (91.6 mg, 0.4 mmol), Cs_2CO_3 (261 mg, 0.8 mmol) and held under vacuum for 10 min and filled with nitrogen. Then toluene/water (4/1, v/v) (2.5 mL) was introduced and the mixture was stirred at 90 °C for 15 h. Upon completion of the reaction, the resulting mixture was cooled down to rt and extracted with EtOAc (4 × 10 mL). The combined organic phase was dried over anhydrous $MgSO_4$. After the removal of solvent under reduced pressure, the crude product was purified by column chromatography on silica gel with petroleum ether/EtOAc (1/1, v/v) as the eluent to yield the product as a pale yellow oil.

Diethyl (1-(3-nitrophenyl)vinyl)phosphonate (5t)



(5t)

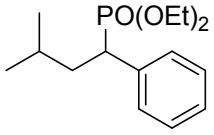
The compound **5t** was synthesized from general procedure C in the presence of 2 mol % of $Pd_2(dba)_3$ and 8 mol % of SPhos at 60 °C for 15 h. The title compound **5t** was isolated as pale yellow oil (107.2 mg, 94% yield). ^{1}H NMR (500 MHz, $CDCl_3$): δ 1.32 (t, J = 7.1 Hz, 6H), 4.12-4.19 (m, 4H), 6.25 (dd, J = 44.7 Hz, 1.0 Hz, 1H), 6.46 (dd, J = 21.8 Hz, 1.0 Hz, 1H), 7.54 (t, J = 8.0 Hz, 1H), 7.88-7.90 (m, 1H), 8.18-8.20 (m, 1H), 8.39 (dd, J = 3.2 Hz, 1.7 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 16.1 (d, J = 6.0 Hz), 62.4 (d, J = 5.7 Hz), 122.2 (d, J = 5.7 Hz), 122.8, 129.3, 133.3, 133.4 (d, J = 5.4 Hz), 138.1 (d, J = 177.5 Hz), 138.2 (d, J = 12.5 Hz), 148.1; ^{31}P NMR (202.5 MHz, $CDCl_3$): δ 15.3; IR (KBr): ν (cm^{-1}) 3481, 2984, 2908, 1532, 1351, 1238, 1049, 1023, 969; GC-MS: *m*/*z* (rel intensity) 285 (M^+, 5), 257 (15), 240 (13), 211 (11), 165 (10), 148 (43), 102 (100), 91 (50); HRMS (ESI) calcd for $C_{12}H_{16}NNaO_5P$ $[M+Na]^+$ 308.0658, found 308.0667.

General procedure D. Synthesis of 18 via photocatalytic Giese reaction of diethyl (1-phenylvinyl)phosphonate with potassium organotrifluoroborates

To an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, $Ir[dF(CF_3)ppy]_2(dtbbpy)PF_6$ (2.2 mg, 0.002 mmol, 0.02 equiv), potassium organotrifluoroborate (0.3 mmol, 3 equiv), diethyl (1-phenylvinyl)phosphonate (24.0 mg, 0.1 mmol, 1 equiv) were added. The tube was evacuated and filled with nitrogen for 3 times, and was charged with DMSO (3 mL, 0.03 M). The tube was irradiated with a 9 W blue LED light strip spiraled within a bowel for 24 h. After the reaction was complete, the mixture was diluted with EtOAc, washed with brine (3 × 10 mL), and the aqueous layer was extracted with EtOAc (4 × 10 mL).The organic layers

were combined and dried over MgSO4, concentrated in vacuo. Flash chromatography (silica gel, EtOAc/ petroleum ether = 1:1) afforded the product **18**.

Diethyl (3-methyl-1-phenylbutyl)phosphonate (18a)



(18a)

The compound **18a** was synthesized from general procedure D. The title compound **18a** was isolated as pale yellow oil (23.8 mg, 84% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 0.82-0.86 (m, 6H), 1.08 (t, *J* = 7.0 Hz, 3H), 1.28 (t, *J* = 7.1 Hz, 3H), 1.35-1.42 (m, 1H), 1.72-1.79 (m, 1H), 1.99-2.04 (m, 1H), 3.08-3.16 (m, 1H), 3.65-3.73 (m, 1H), 3.84-3.92 (m, 1H), 3.98-4.07 (m, 2H), 7.23-7.25 (m, 1H), 7.29-7.33 (m, 4H); ^{13}C NMR (126 MHz, $CDCl_3$) δ: 16.1 (d, *J* = 5.7 Hz), 16.3 (d, *J* = 5.9 Hz), 20.6, 23.4, 25.0 (d, *J* = 15.0 Hz), 38.1 (d, *J* = 3.6 Hz), 42.4 (d, *J* = 137.1 Hz), 61.7 (d, *J* = 7.3 Hz), 62.4 (d, *J* = 7.1 Hz), 126.9 (d, *J* = 3.4 Hz), 128.3 (d, *J* = 2.7 Hz), 129.2 (d, *J* = 6.8 Hz), 136.0 (d, *J* = 7.2 Hz); ^{31}P NMR (202 MHz, $CDCl_3$) δ: 29.6; IR (neat): ν (cm^{-1}) 2938, 2895, 2859, 2843, 1464, 1246, 1057, 1028, 962; LC-MS(ESI) $[M+H]^+$: 285.44; HRMS (ESI) $[M+H]^+$: calculated for $C_{15}H_{26}O_3P$: 285.1614, found 285.1635.

References of known compounds

5b, **5c**, **7b**, **7c**	S. Sobhani and M. Honarmand, *Synlett*, 2013, **24**, 236-240.
5a, **5n**, **5u**, **7a**, **12a**, **16**	D.-Y. Wang, X.-P. Hu, J. Deng, S.-B. Yu, Z.-C. Duan and Z. Zheng, *J. Org. Chem.*, 2009, **74**, 4408-4410.
5d, **5e**, **5g**, **5h**, **5i**, **5j**, **5k**, **5l**, **5p**, **5q**, **5r**, **5s**, **5w**, **5x**, **7d**, **7e**, **7f**, **7g**, **7h**, **15**	Y. Fang, L. Zhang, X. Jin, J. Li, M. Yuan, R. Li, T. Wang, T. Wang, H. Hu and J. Gu, *Eur. J. Org. Chem.*, 2016, 1577-1587.
5y	Y. Fang, M. Yuan, X. Jin, L. Zhang, R. Li, S. Yang and M. Fang, *Tetrahedron Lett.*, 2016, **57**, 1368-1371.
9a, **9d**	T. Okauchi, T. Yano, T. Fukamachi, J. Ichikawa and T. Minami, *Tetrahedron Lett*., 1999, **40**, 5337-5340.
18b	S. Beers, E. A. Malloy and C. Schwender, US 5508273 A 19960416, 1996.

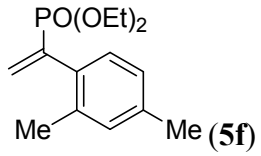
References

1 (*a*) G. A. Molander and B. Biolatto, *J. Org. Chem.*, 2003, **68**, 4302-4314; (*b*) G. A. Molander, B. W. Katona and F. Machrouhi, *J. Org. Chem.*, 2002, **67**, 8416-8423.

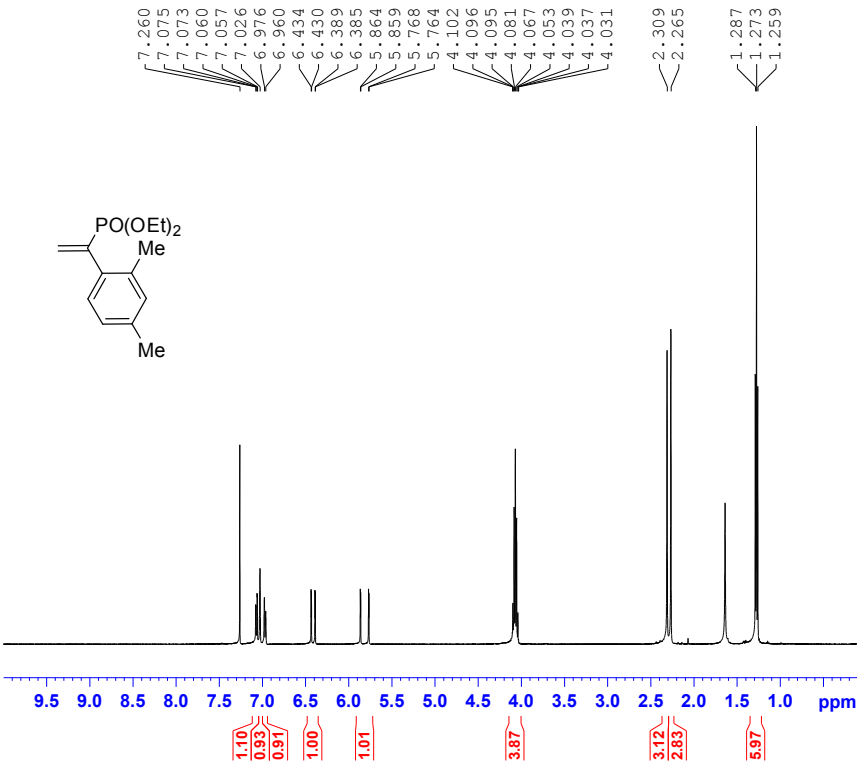
2 Ö. Dogan, M. Isci and M. Aygun, *Tetrahedron Asymmetry*, 2013, **24**, 562-567.

3 L. Rigger, R. L. Schmidt, K. M. Holman, M. Simonović and R. Micura, *Chem. Eur. J.*, 2013, **19**, 15872-15878.

^{1}H, ^{13}C, ^{31}P, ^{19}F NMR spectra of new compounds



(5f)

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TD0                        1

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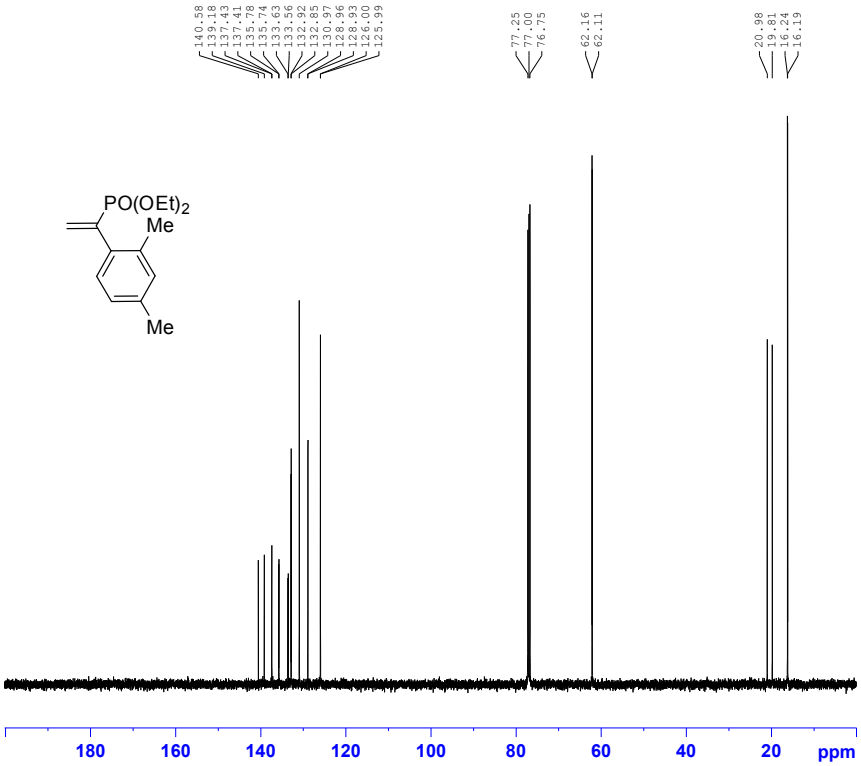


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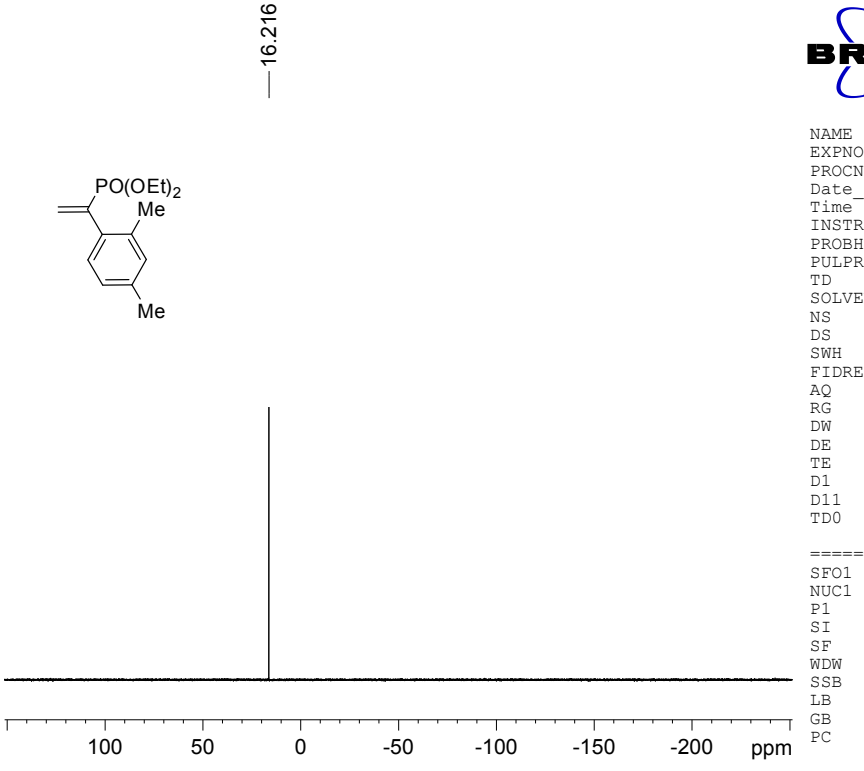


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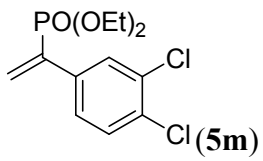
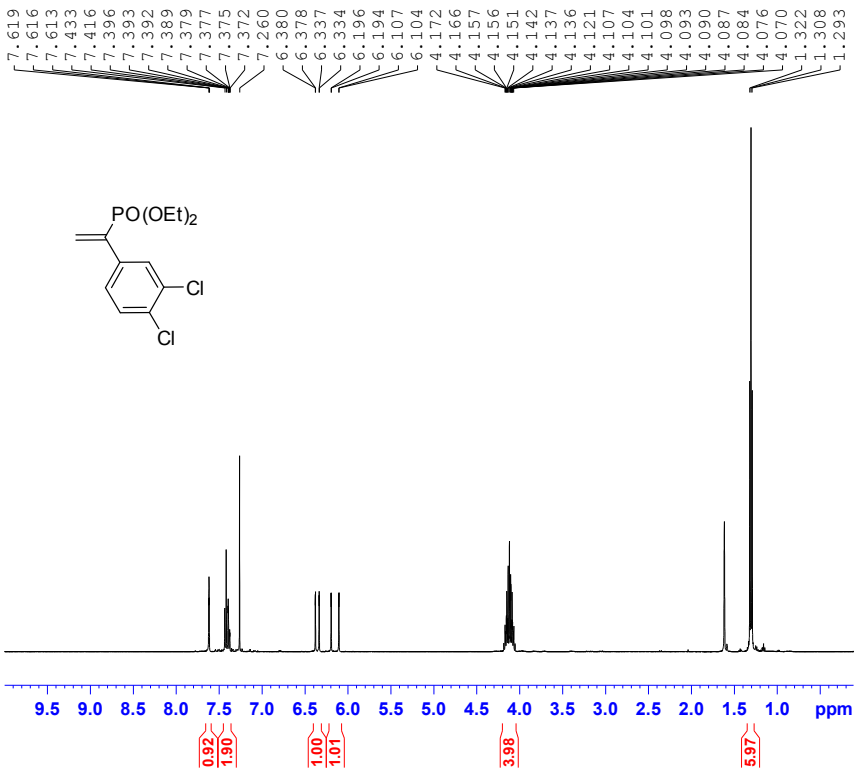


(5m)

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TE                  300.0 K
D1              1.00000000 sec
TD0                     1

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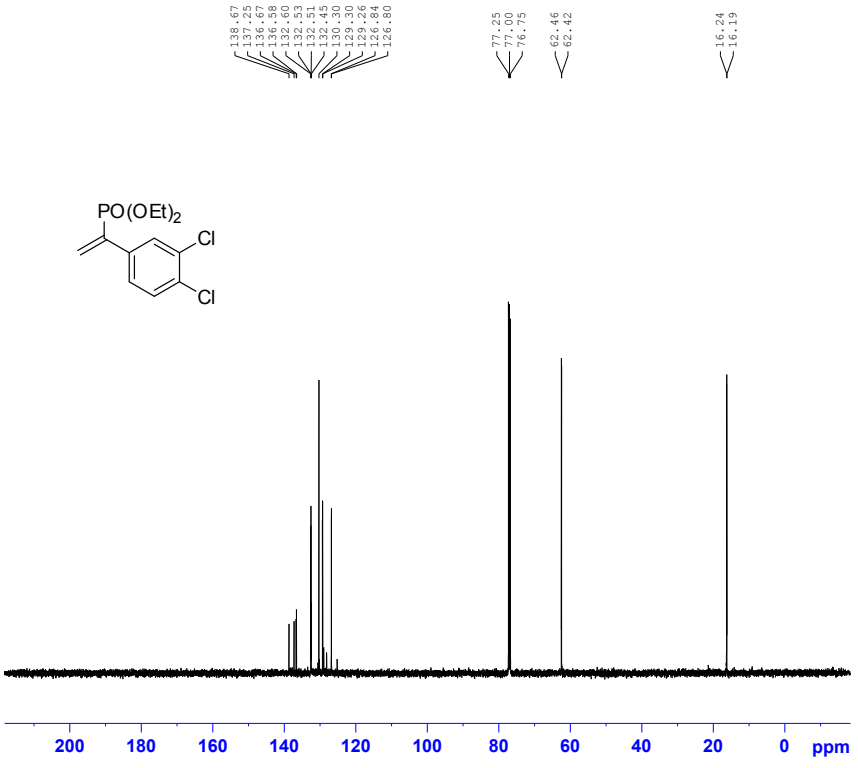


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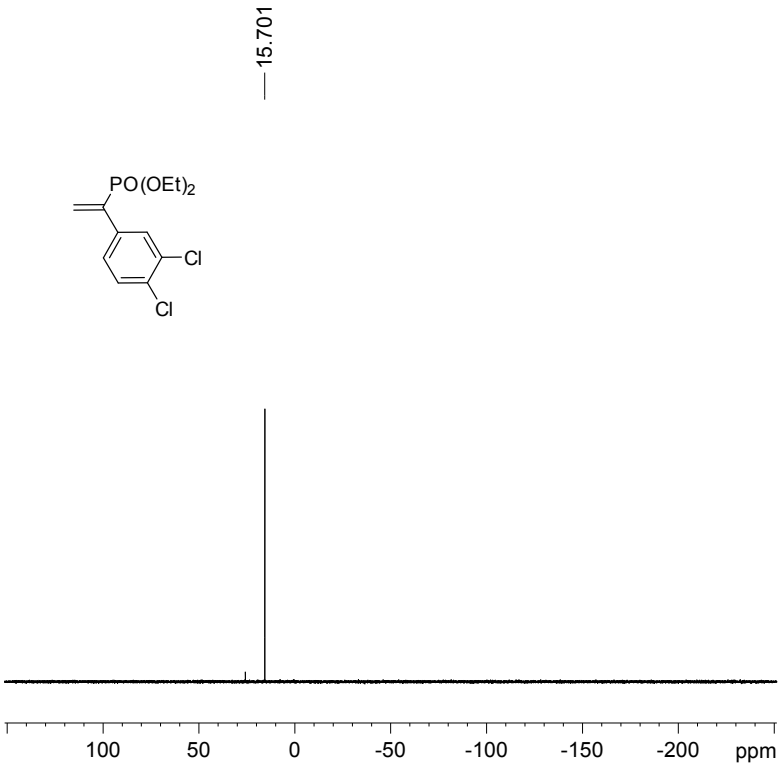


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D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

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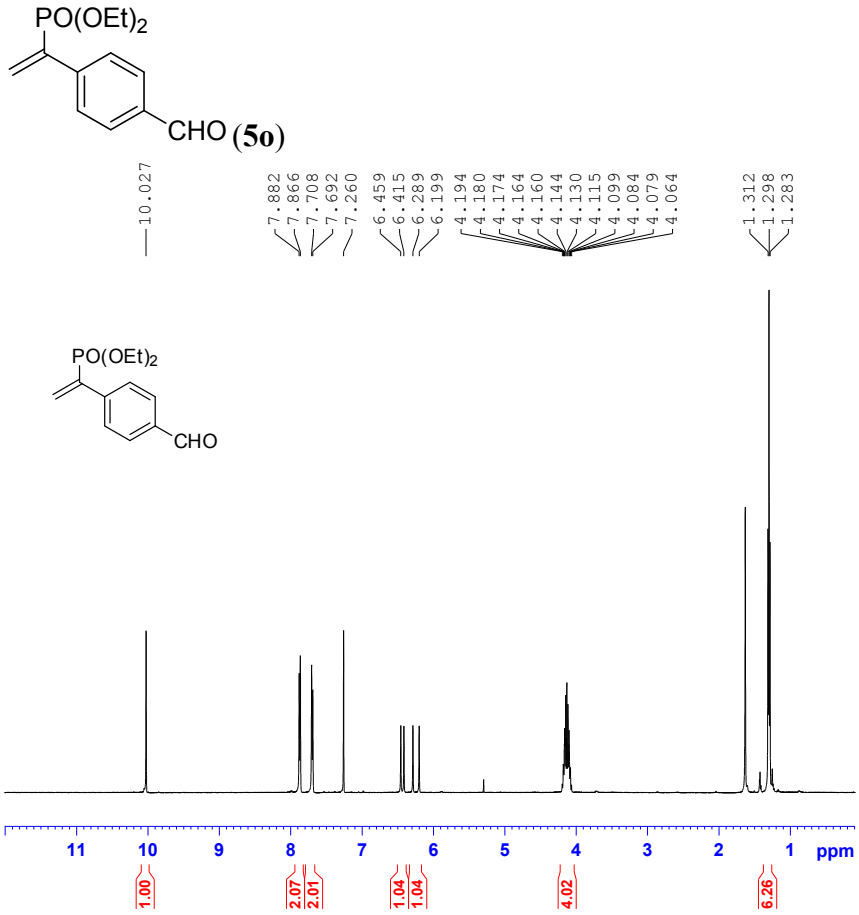


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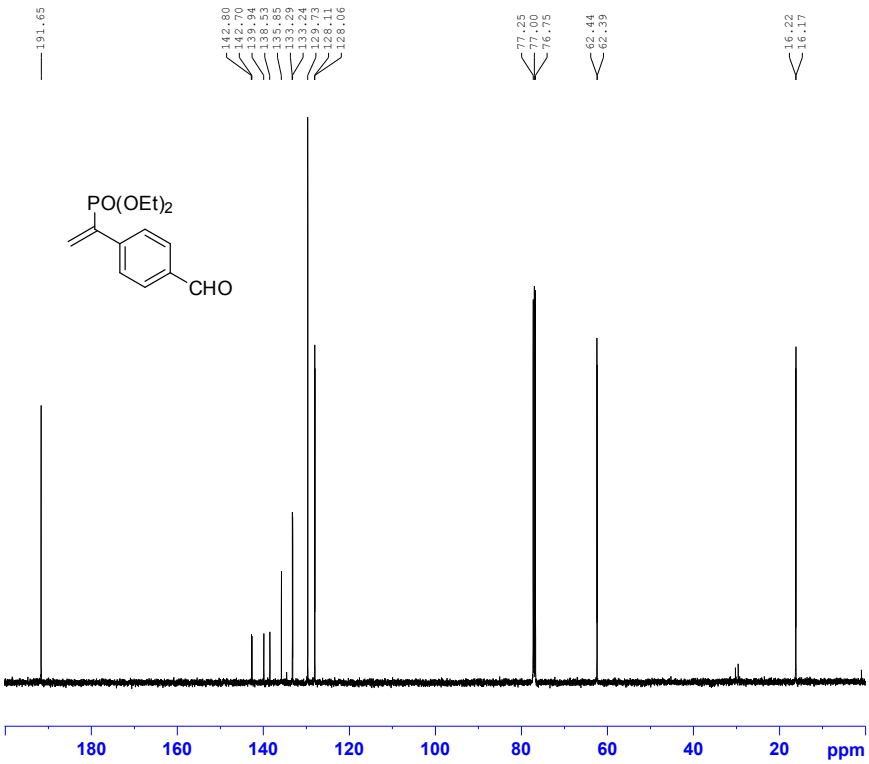
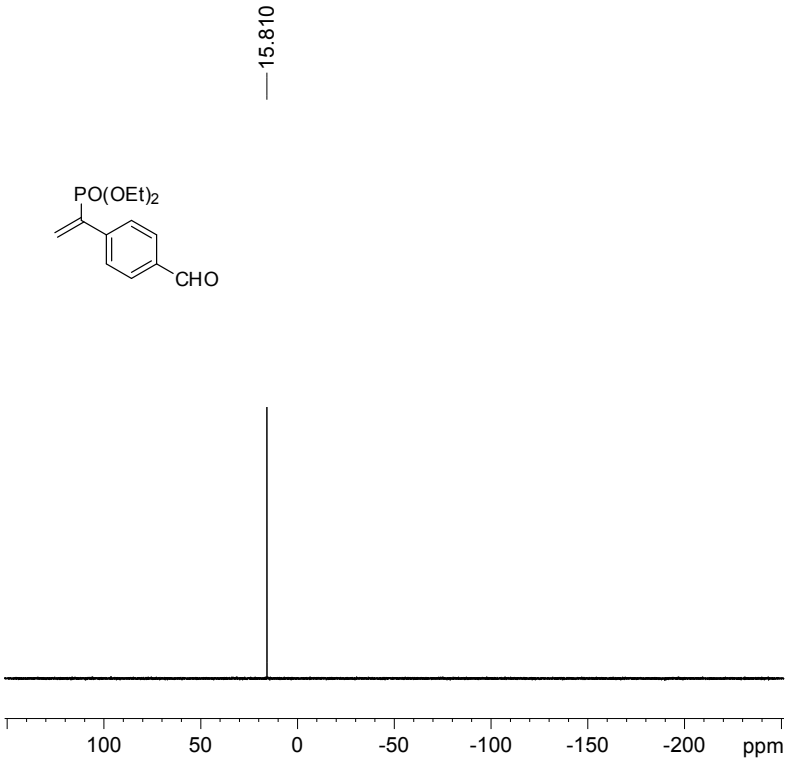
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ            3.2768500 sec
RG               137.21
DW                50.000 usec
DE                6.50 usec
TE                300.0 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                 1H
P1                11.11 usec
SI                65536
SF          500.1300137 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



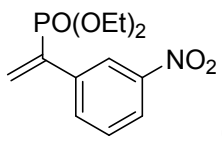

```
NAME      20151125fangyw
EXPNO                 42
PROCNO                 1
Date_           20151125
Time               12.07
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010548 sec
RG                201.95
DW                16.800 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                10.17 usec
SI                32768
SF          125.7577974 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

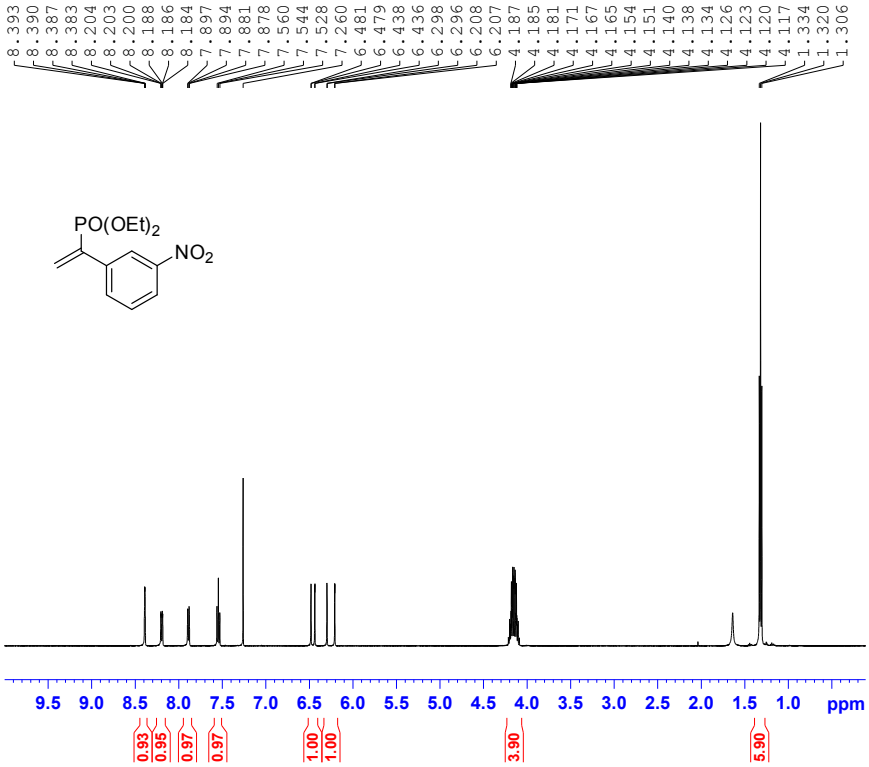



```
NAME      20151124fangyw
EXPNO                 73
PROCNO                 1
Date_           20151124
Time               14.50
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           81521.742 Hz
FIDRES         1.243923 Hz
AQ             0.4020041 sec
RG                201.95
DW                 6.133 usec
DE                 6.50 usec
TE                300.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        202.4462121 MHz
NUC1                31P
P1                13.00 usec
SI                32768
SF          202.4563350 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

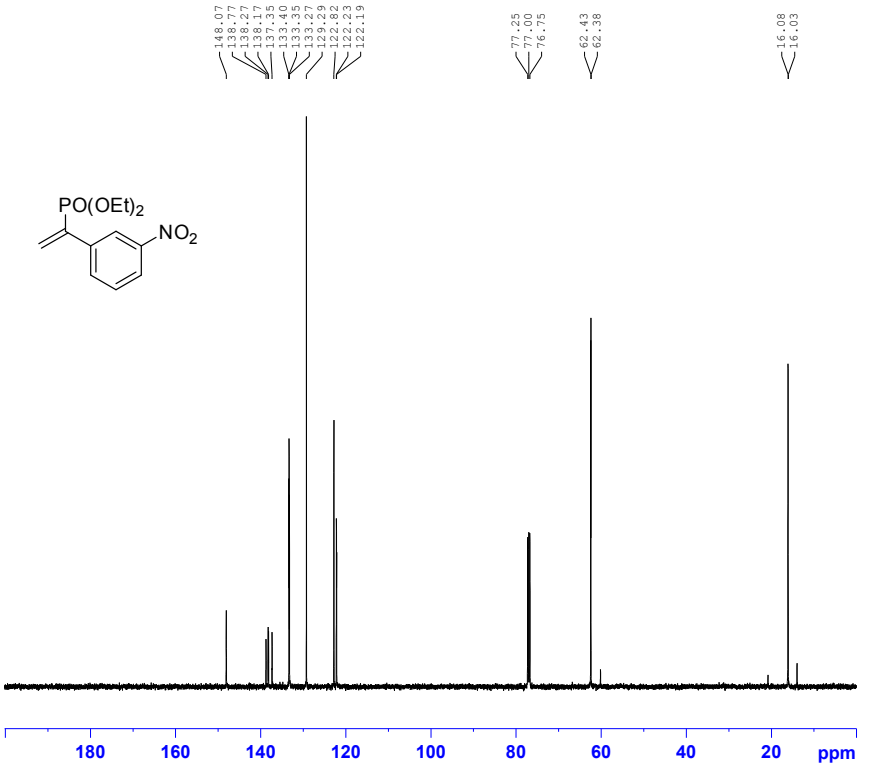


(5t)



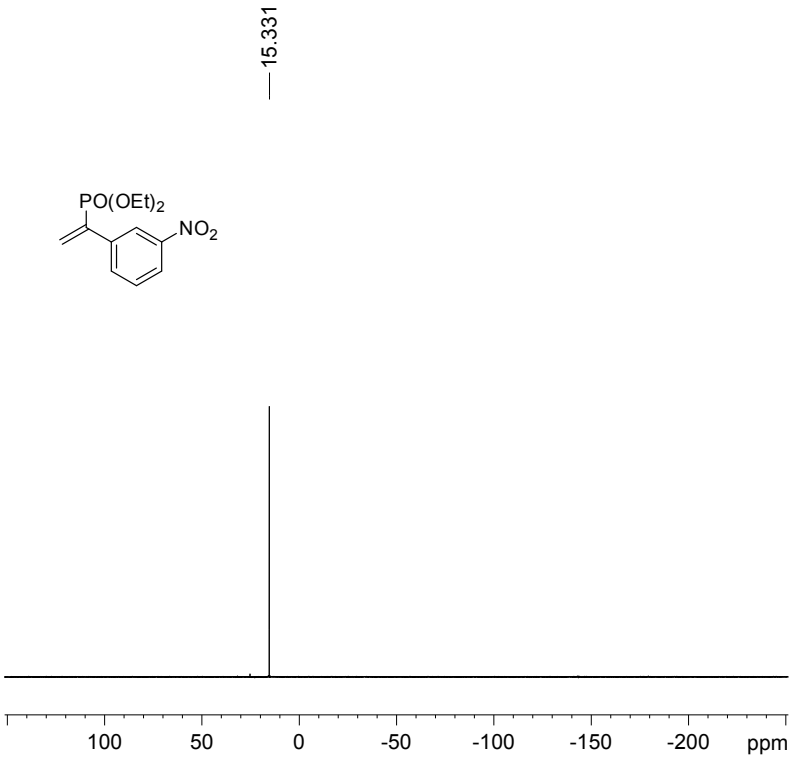

```
NAME          20151113fangyw
EXPNO                 51
PROCNO                 1
Date_           20151113
Time               14.45
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES        0.152588 Hz
AQ            3.2768500 sec
RG               137.21
DW               50.000 usec
DE                6.50 usec
TE               300.0 K
D1            1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         500.1330885 MHz
NUC1                1H
P1                11.11 usec
SI                65536
SF           500.1300134 MHz
WDW                EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```



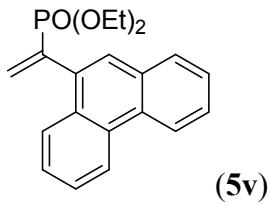

```
NAME          20151113fangyw
EXPNO                 52
PROCNO                 1
Date_           20151113
Time               16.09
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   174
DS                     4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG               201.95
DW               16.800 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                13C
P1                10.17 usec
SI                32768
SF           125.7578089 MHz
WDW                EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                1.40
```

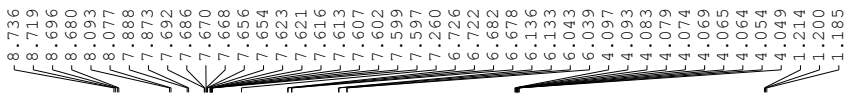


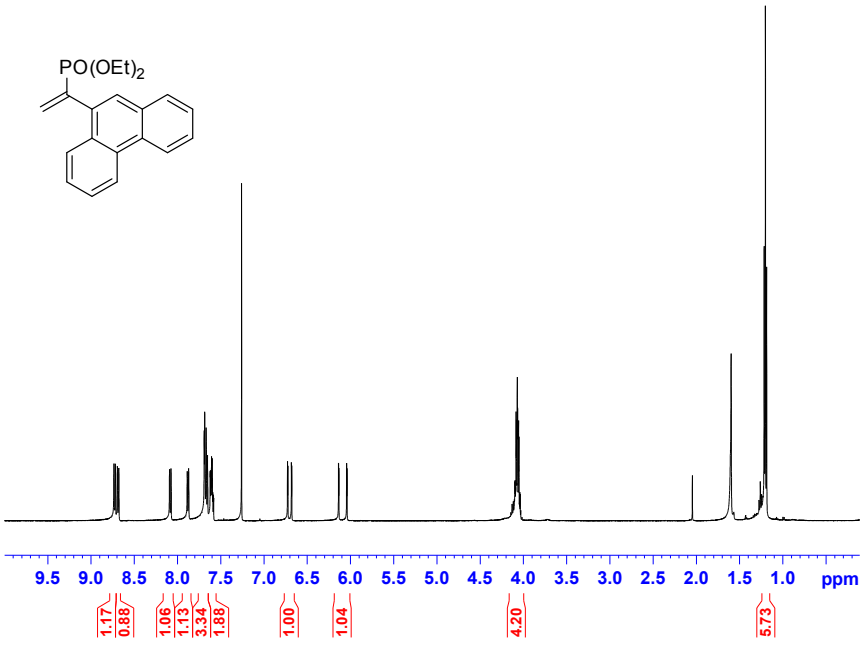

```
NAME      20151113fangyw
EXPNO                 53
PROCNO                 1
Date_           20151113
Time               14.47
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   32
DS                    4
SWH           81521.742 Hz
FIDRES         1.243923 Hz
AQ             0.4020041 sec
RG                201.95
DW                 6.133 usec
DE                 6.50 usec
TE                300.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        202.4462121 MHz
NUC1                31P
P1                13.00 usec
SI                32768
SF          202.4563350 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



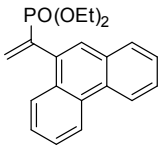
(5v)

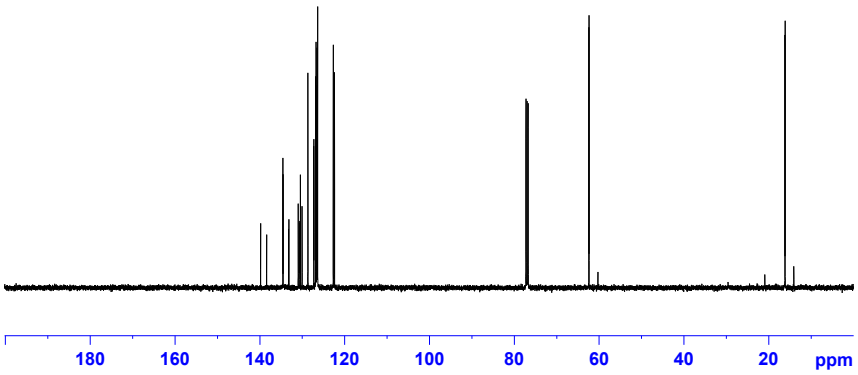





```
NAME      20151116fangyw
EXPNO                 20
PROCNO                 1
Date_           20151116
Time               15.32
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    2
SWH           10000.000 Hz
FIDRES         0.152588 Hz
AQ             3.2768500 sec
RG                137.21
DW                50.000 usec
DE                 6.50 usec
TE                300.0 K
D1           1.00000000 sec
TD0                   1

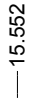
======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                 1H
P1                11.11 usec
SI                65536
SF          500.1300135 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

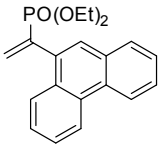


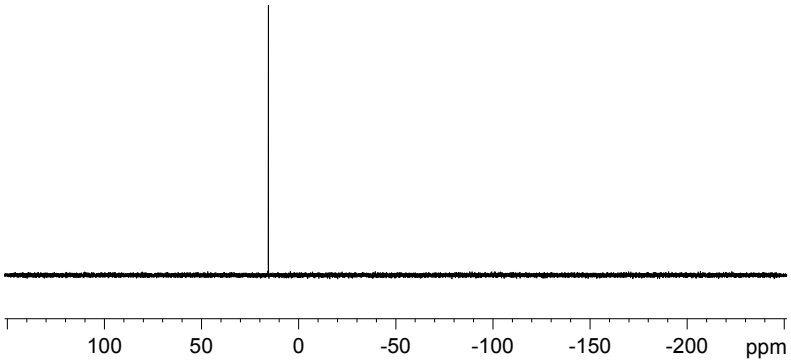



```
NAME      20151117fangyw
EXPNO                 20
PROCNO                 1
Date_           20151117
Time_             14.47
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                  128
DS                    4
SWH           29761.904 Hz
FIDRES         0.454131 Hz
AQ             1.1010548 sec
RG                201.95
DW                16.800 usec
DE                 6.50 usec
TE                300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                10.17 usec
SI                32768
SF          125.7578032 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

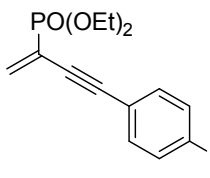




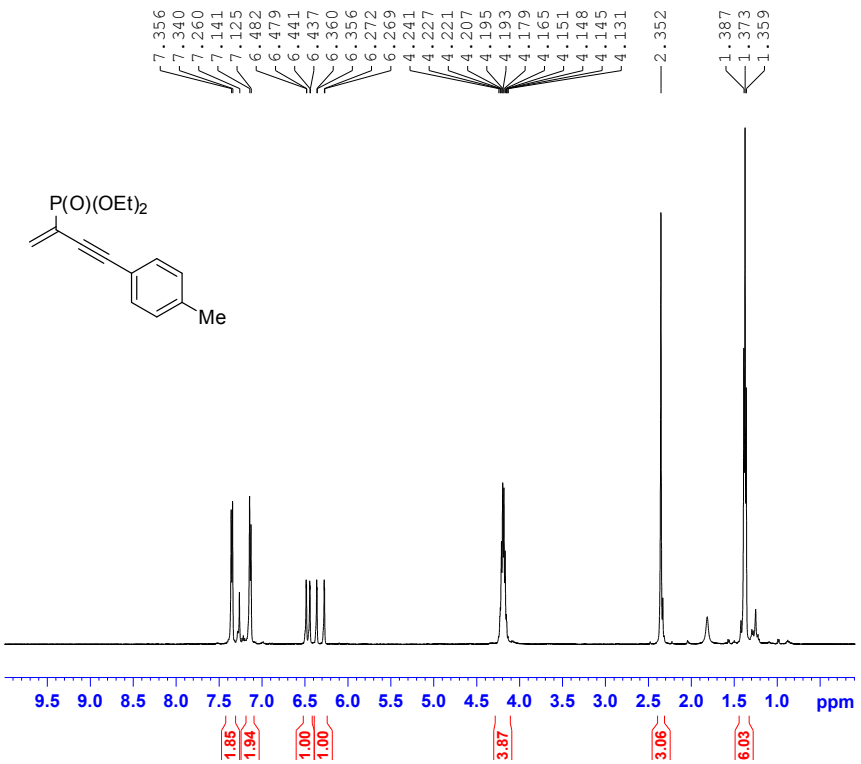



```
NAME      20151116fangyw
EXPNO                 23
PROCNO                 1
Date_           20151116
Time              15.33
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           81521.742 Hz
FIDRES         1.243923 Hz
AQ             0.4020041 sec
RG                201.95
DW                 6.133 usec
DE                 6.50 usec
TE                300.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1        202.4462121 MHz
NUC1                31P
P1                13.00 usec
SI                32768
SF          202.4563350 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

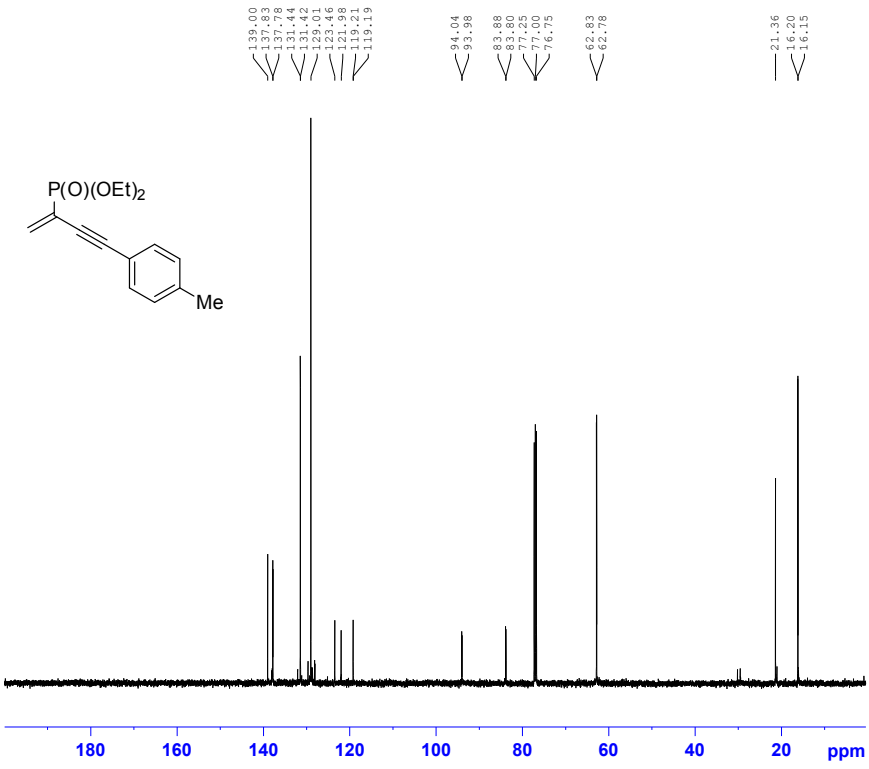


(9b)



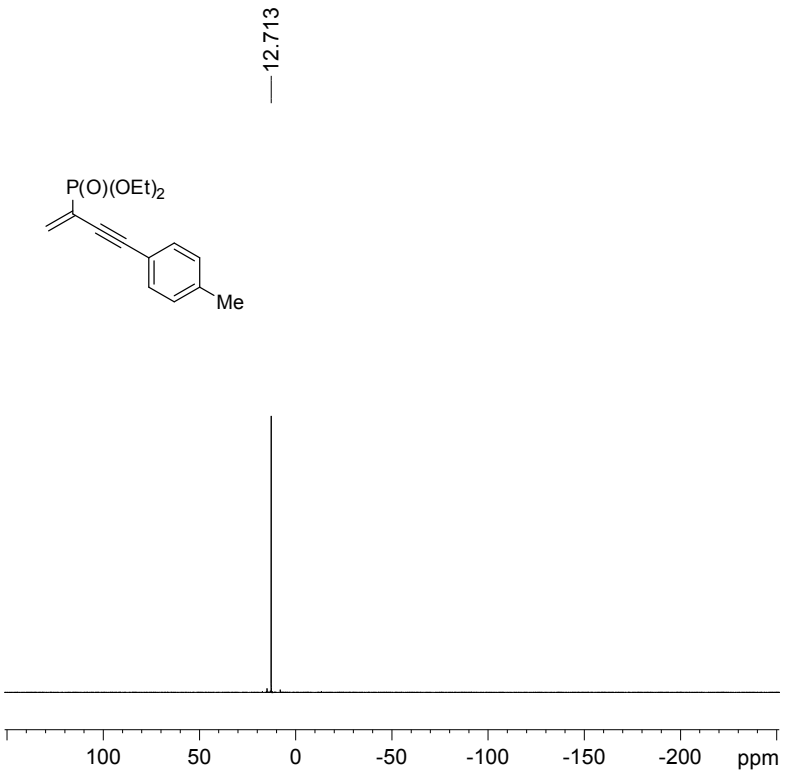

```
NAME      20151130fangyw
EXPNO                 80
PROCNO                 1
Date_           20151130
Time               15.04
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH           10000.000 Hz
FIDRES            0.152588 Hz
AQ             3.2768500 sec
RG                 70.97
DW                50.000 usec
DE                 6.50 usec
TE                300.0 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          500.1330885 MHz
NUC1                  1H
P1                 11.11 usec
SI                 65536
SF            500.1300136 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



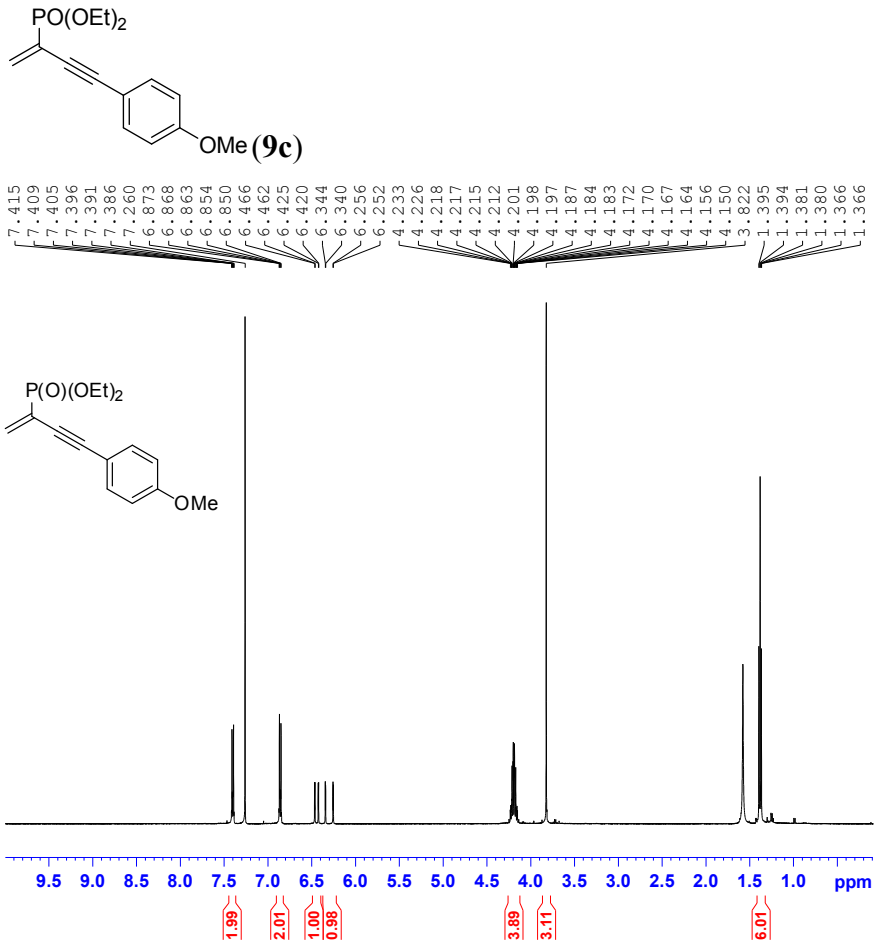

```
NAME      20151130fangyw
EXPNO                 82
PROCNO                 1
Date_           20151130
Time               15.56
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    66
DS                     4
SWH           29761.904 Hz
FIDRES            0.454131 Hz
AQ             1.1010548 sec
RG                201.95
DW                16.800 usec
DE                 6.50 usec
TE                300.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          125.7703637 MHz
NUC1                  13C
P1                 10.17 usec
SI                 32768
SF            125.7578053 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



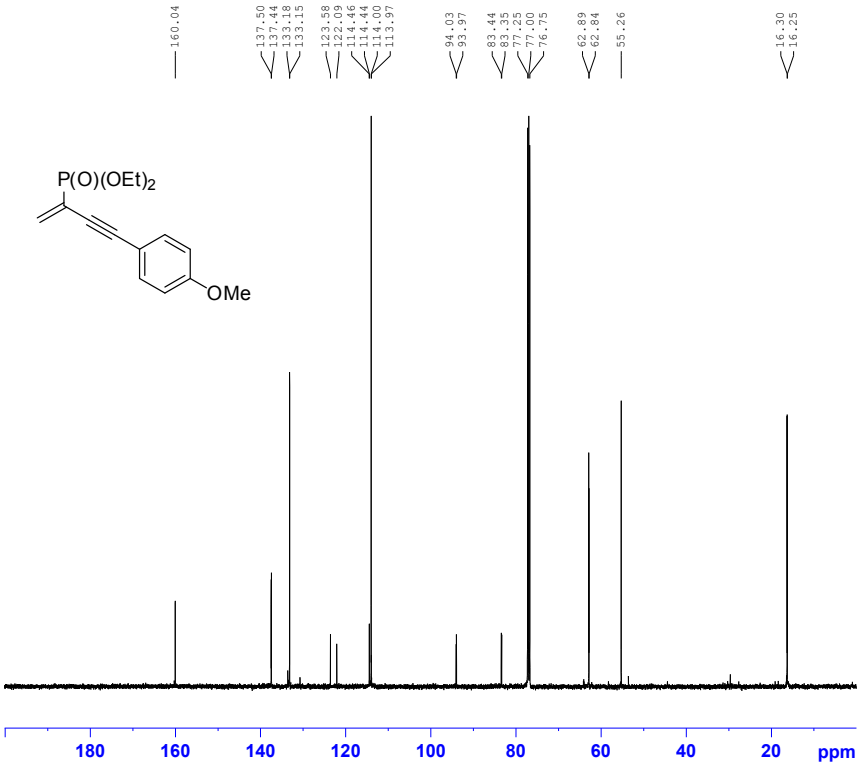

```
NAME      20151130fangyw
EXPNO                 83
PROCNO                 1
Date_           20151130
Time              15.51
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD              65536
SOLVENT          CDCl3
NS                  16
DS                   4
SWH          81521.742 Hz
FIDRES        1.243923 Hz
AQ           0.4020041 sec
RG              201.95
DW               6.133 usec
DE                6.50 usec
TE               300.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1       202.4462121 MHz
NUC1              31P
P1              13.00 usec
SI              32768
SF         202.4563350 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```



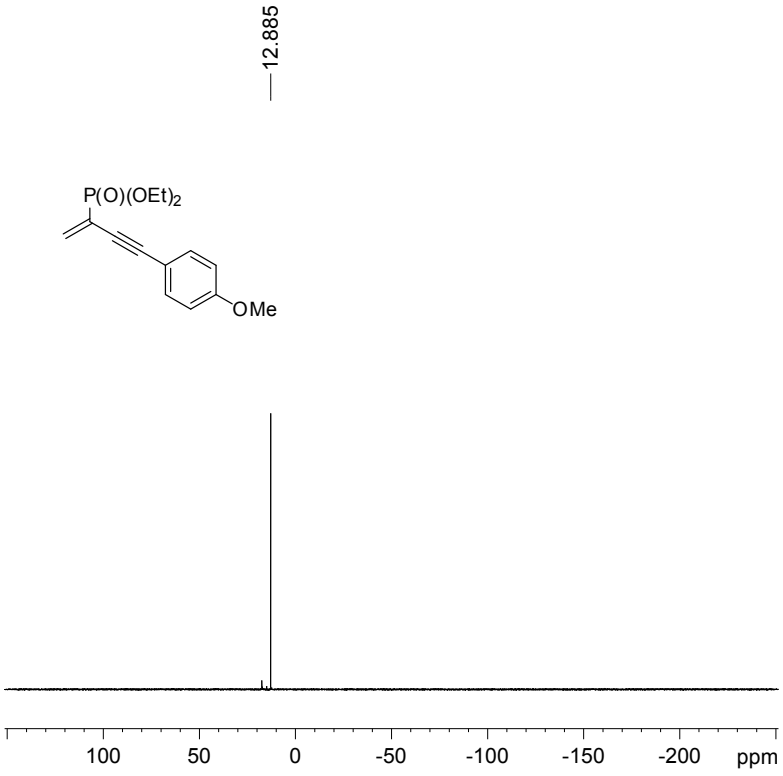

```
NAME      20151117fangyw
EXPNO                 40
PROCNO                 1
Date_           20151117
Time              15.06
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG           zg30
TD              65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH          10000.000 Hz
FIDRES        0.152588 Hz
AQ           3.2768500 sec
RG              160.87
DW              50.000 usec
DE                6.50 usec
TE               300.0 K
D1          1.00000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1              1H
P1              11.11 usec
SI              65536
SF          500.1300135 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00
```



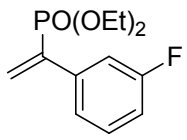

```
NAME      20151118fangyw
EXPNO                 42
PROCNO                 1
Date_           20151118
Time               12.11
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    4
SWH           29761.904 Hz
FIDRES        0.454131 Hz
AQ            1.1010548 sec
RG               201.95
DW               16.800 usec
DE                6.50 usec
TE               300.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         125.7703637 MHz
NUC1                 13C
P1                 10.17 usec
SI                 32768
SF           125.7577952 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

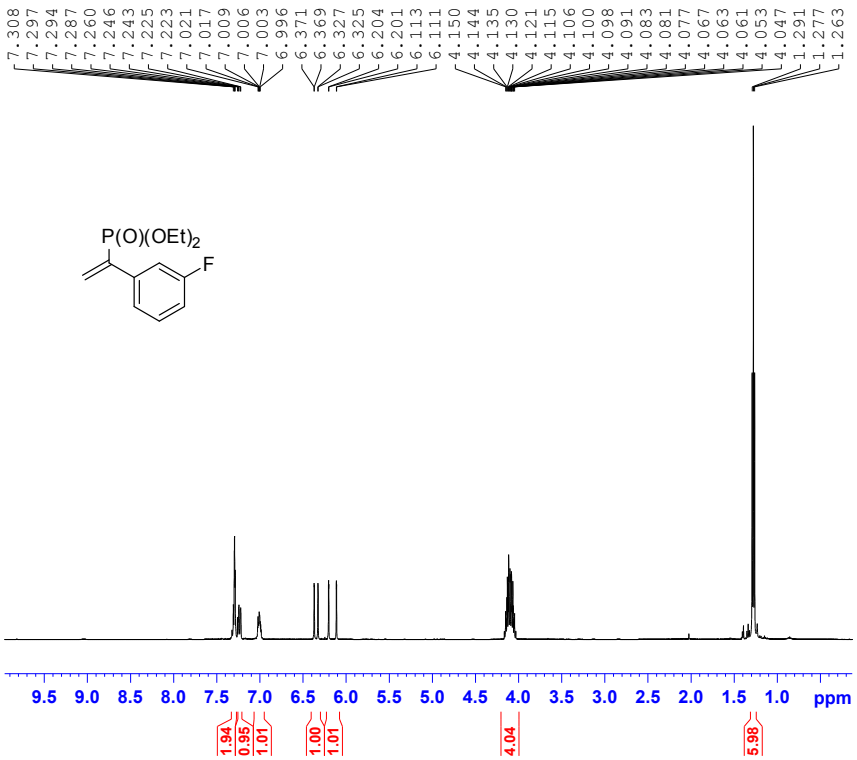



```
NAME      20160114fangyw
EXPNO                 10
PROCNO                 1
Date_           20160114
Time               10.03
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           81521.742 Hz
FIDRES        1.243923 Hz
AQ            0.4020041 sec
RG               201.95
DW                6.133 usec
DE                6.50 usec
TE               300.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1         202.4462121 MHz
NUC1                 31P
P1                13.00 usec
SI                 32768
SF           202.4563350 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



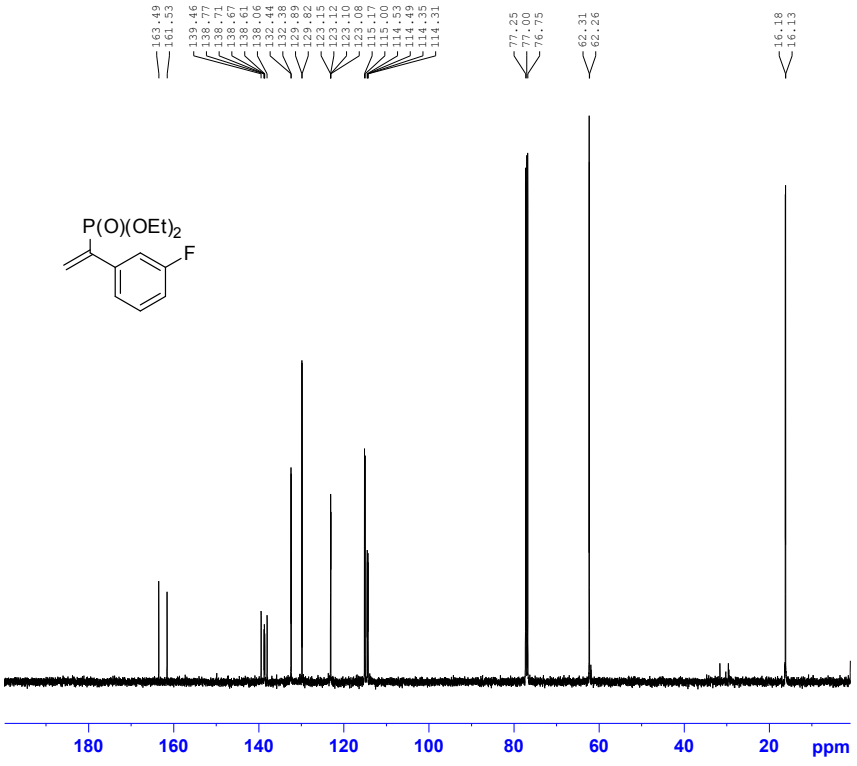
(12b)



BRUKER

```
NAME            20150122
EXPNO                2
PROCNO               1
Date_         20150122
Time             14.54
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG          zg30
TD              65536
SOLVENT          CDCl3
NS                  16
DS                   2
SWH          10000.000 Hz
FIDRES           0.152588 Hz
AQ           3.2768500 sec
RG                55.23
DW               50.000 usec
DE                6.50 usec
TE               295.7 K
D1           1.00000000 sec
TD0                  1

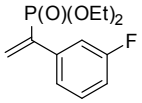
======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1                1H
P1                11.50 usec
SI                65536
SF          500.1300134 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```

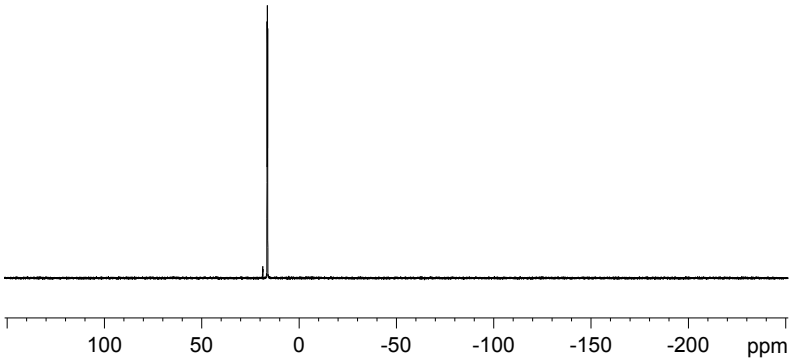


BRUKER

```
NAME        20150123fang
EXPNO                 22
PROCNO                 1
Date_           20150123
Time               13.56
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  256
DS                    4
SWH           29761.904 Hz
FIDRES            0.454131 Hz
AQ            1.1010548 sec
RG                201.95
DW               16.800 usec
DE                6.50 usec
TE               296.0 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1

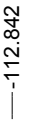
======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1                13C
P1                9.75 usec
SI                32768
SF          125.7578000 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

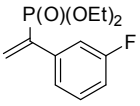


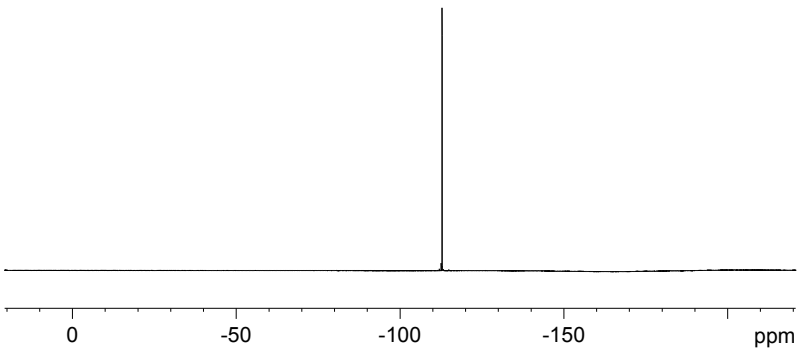



```
NAME          20150123fang
EXPNO                   23
PROCNO                   1
Date_             20150123
Time                 13.41
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      32
DS                       4
SWH              81521.742 Hz
FIDRES            1.243923 Hz
AQ                0.4020041 sec
RG                   201.95
DW                    6.133 usec
DE                     6.50 usec
TE                    295.0 K
D1             2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          202.4462121 MHz
NUC1                  31P
P1                  13.00 usec
SI                  32768
SF            202.4563350 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

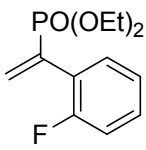




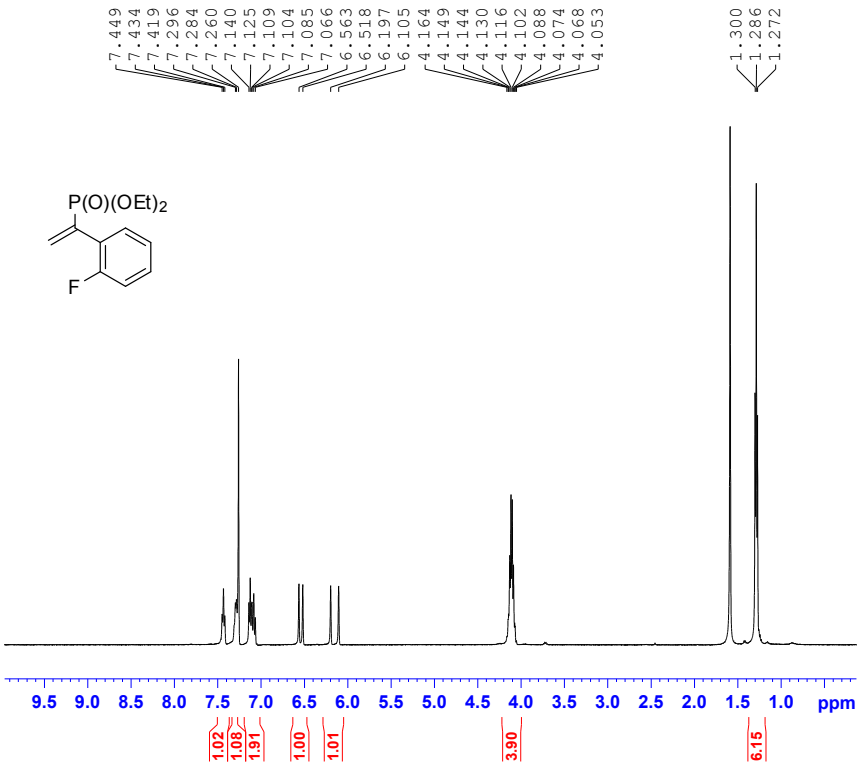



```
NAME          20150123fang
EXPNO                   24
PROCNO                   1
Date_             20150123
Time                 13.59
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG              zgflqn
TD                  131072
SOLVENT              CDCl3
NS                      32
DS                       4
SWH             113636.367 Hz
FIDRES            0.866977 Hz
AQ                0.5767668 sec
RG                   201.95
DW                    4.400 usec
DE                     6.50 usec
TE                    295.4 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          470.5453180 MHz
NUC1                  19F
P1                  14.75 usec
SI                  65536
SF            470.5923770 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



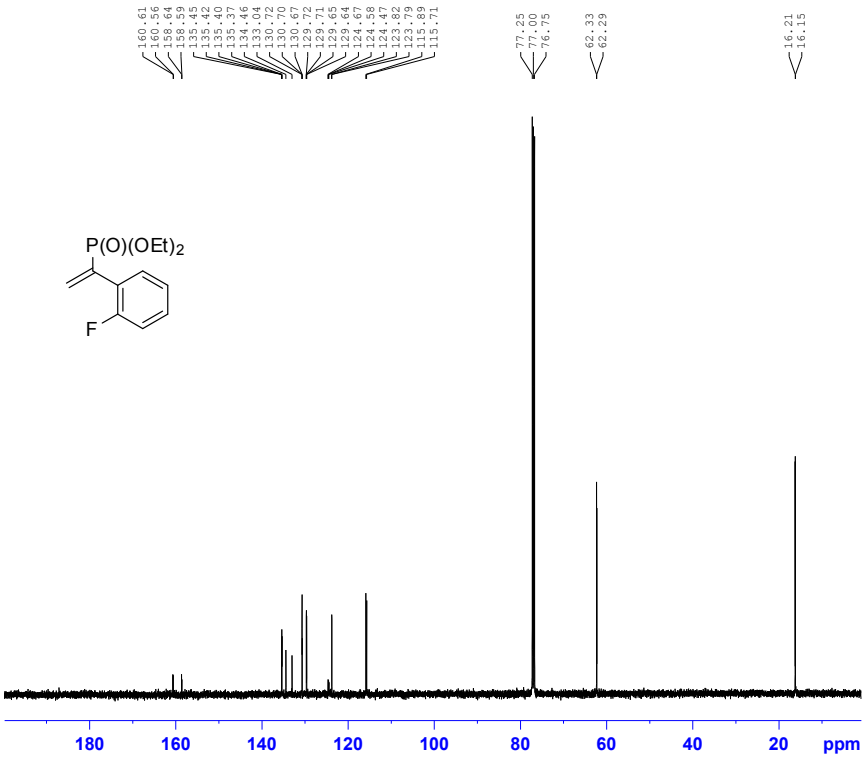
(12c)



BRUKER

```
NAME          20151124fangyw
EXPNO                     10
PROCNO                     1
Date_               20151124
Time                   13.40
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        16
DS                         2
SWH               10000.000 Hz
FIDRES                0.152588 Hz
AQ                3.2768500 sec
RG                    160.87
DW                    50.000 usec
DE                     6.50 usec
TE                    300.0 K
D1                1.00000000 sec
TD0                        1

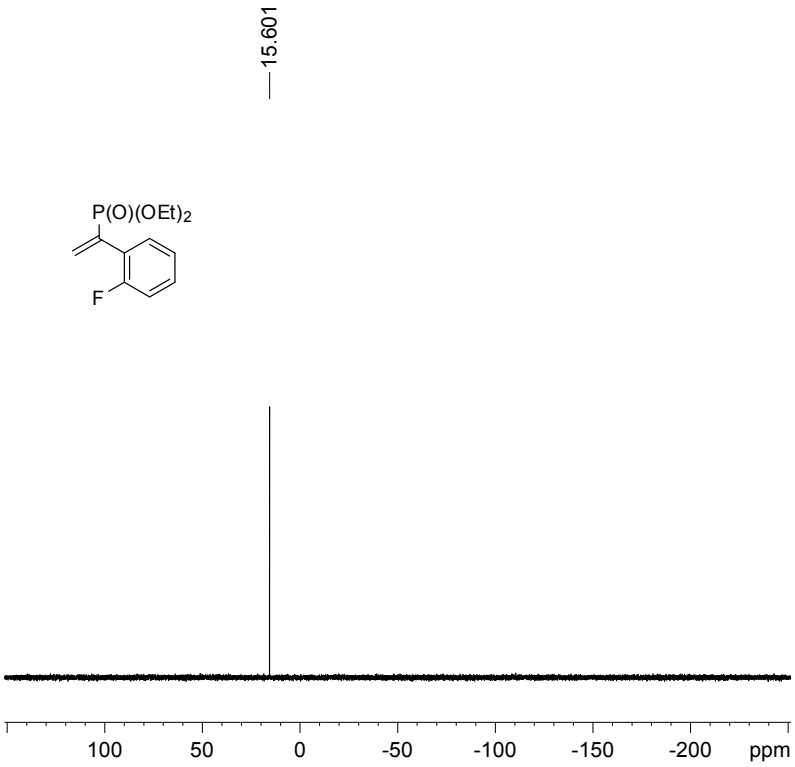
======== CHANNEL f1 ========
SFO1           500.1330885 MHz
NUC1                      1H
P1                     11.11 usec
SI                     65536
SF             500.1300142 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



BRUKER

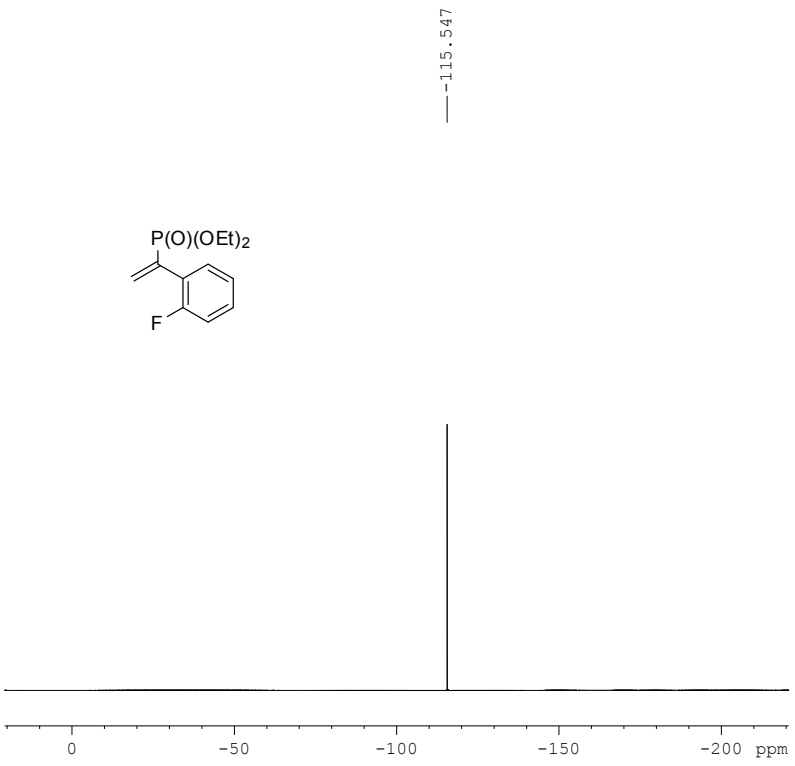
```
NAME          20151125fangyw
EXPNO                     12
PROCNO                     1
Date_               20151125
Time                   11.18
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       256
DS                         4
SWH               29761.904 Hz
FIDRES                0.454131 Hz
AQ                1.1010548 sec
RG                    201.95
DW                    16.800 usec
DE                     6.50 usec
TE                    300.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1           125.7703637 MHz
NUC1                      13C
P1                     10.17 usec
SI                     32768
SF             125.7577931 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```



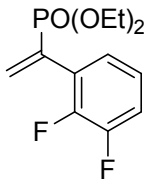

```
NAME      20151124fangyw
EXPNO                13
PROCNO                1
Date_          20151124
Time              13.42
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           81521.742 Hz
FIDRES         1.243923 Hz
AQ            0.4020041 sec
RG               201.95
DW                6.133 usec
DE                6.50 usec
TE               300.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        202.4462121 MHz
NUC1                31P
P1                13.00 usec
SI                32768
SF          202.4563350 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

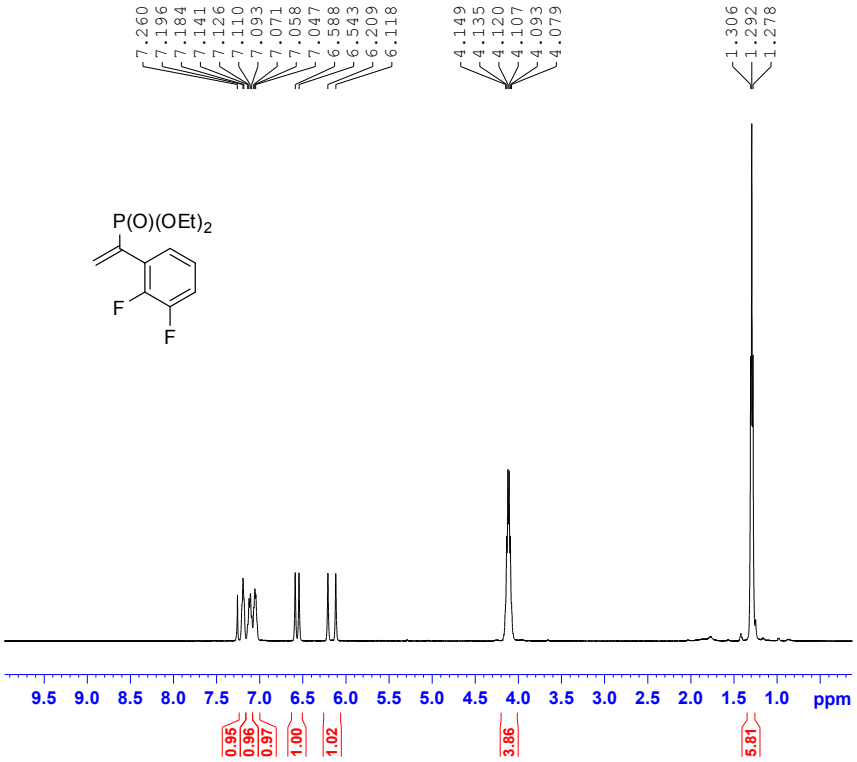



```
NAME      20151130fangyw
EXPNO                10
PROCNO                1
Date_          20151130
Time              10.32
INSTRUM           spect
PROBHD   5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD               131072
SOLVENT           CDCl3
NS                   16
DS                    4
SWH          113636.367 Hz
FIDRES         0.866977 Hz
AQ            0.5767668 sec
RG               201.95
DW                4.400 usec
DE                6.50 usec
TE               300.0 K
D1           1.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        470.5453180 MHz
NUC1                19F
P1                14.75 usec
SI                65536
SF          470.5923770 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

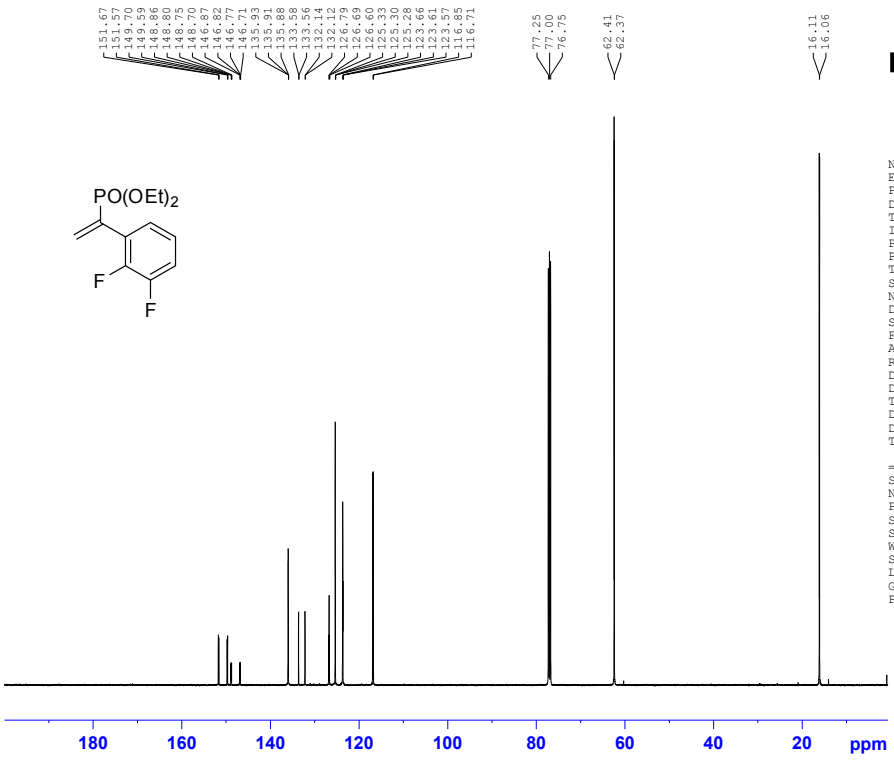


(12d)



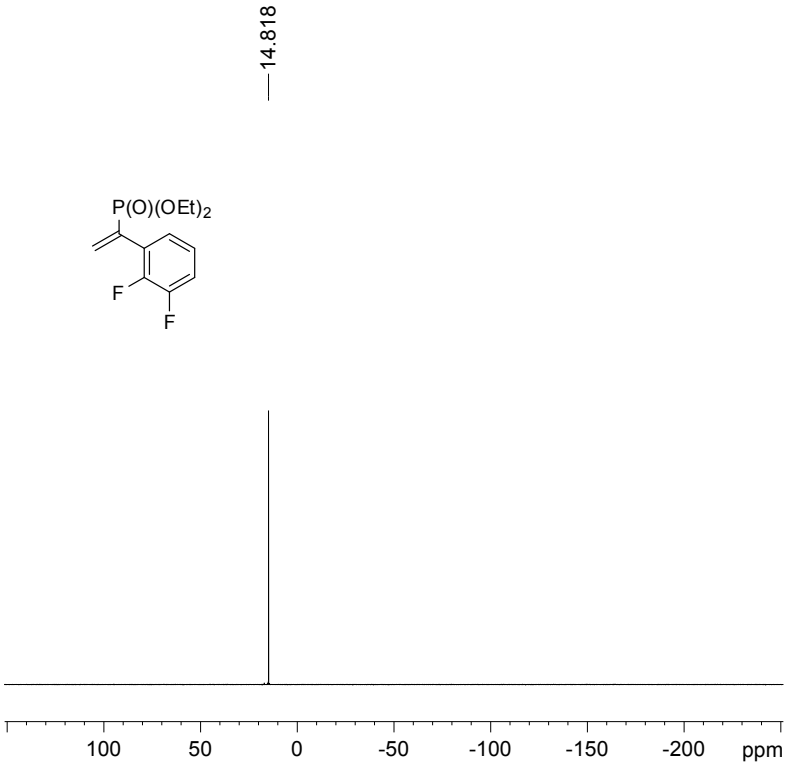

```
NAME      20151216fangyw-1
EXPNO                   30
PROCNO                   1
Date_             20151216
Time                 10.51
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      16
DS                       2
SWH              10000.000 Hz
FIDRES               0.152588 Hz
AQ                3.2768500 sec
RG                     82.11
DW                  50.000 usec
DE                   6.50 usec
TE                   300.0 K
D1              1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          500.1330885 MHz
NUC1                    1H
P1                   11.11 usec
SI                   65536
SF            500.1300136 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



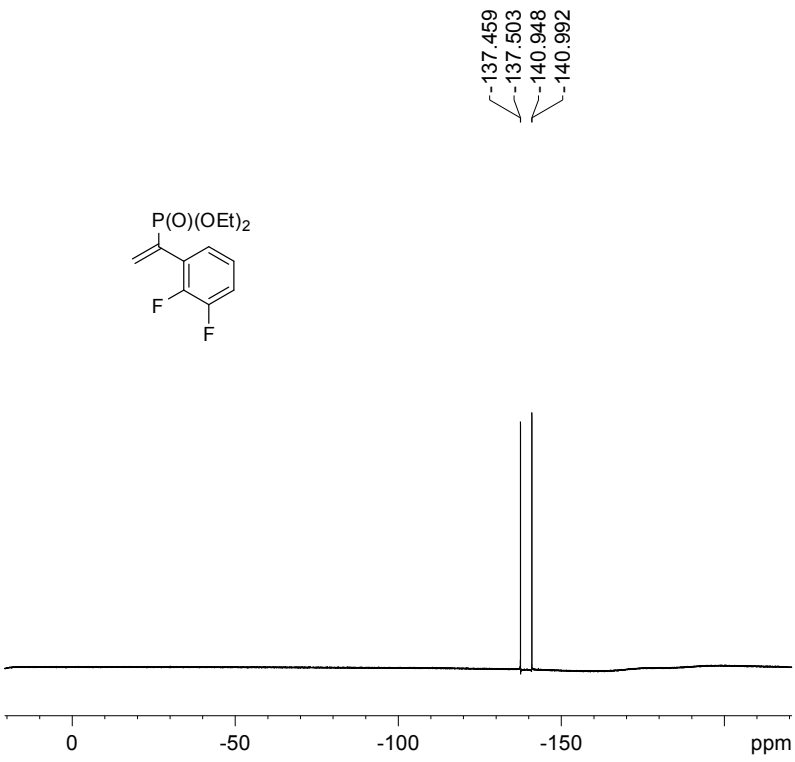

```
NAME             20170902
EXPNO                  10
PROCNO                  1
Date_            20170902
Time                3.42
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                  65536
SOLVENT             CDCl3
NS                   4096
DS                      4
SWH              29761.904 Hz
FIDRES               0.454131 Hz
AQ                1.1010548 sec
RG                   201.95
DW                  16.800 usec
DE                   6.50 usec
TE                   296.8 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          125.7703637 MHz
NUC1                   13C
P1                  10.17 usec
SI                   32768
SF            125.7577998 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



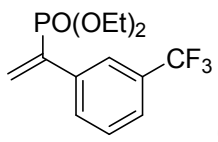

```
NAME      20151216fangyw-1
EXPNO                   33
PROCNO                   1
Date_           20151216
Time                14.21
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT             CDCl3
NS                     16
DS                      4
SWH            81521.742 Hz
FIDRES          1.243923 Hz
AQ             0.4020041 sec
RG                201.95
DW                 6.133 usec
DE                 6.50 usec
TE                300.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         202.4462121 MHz
NUC1                31P
P1                 13.00 usec
SI                 32768
SF           202.4563350 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

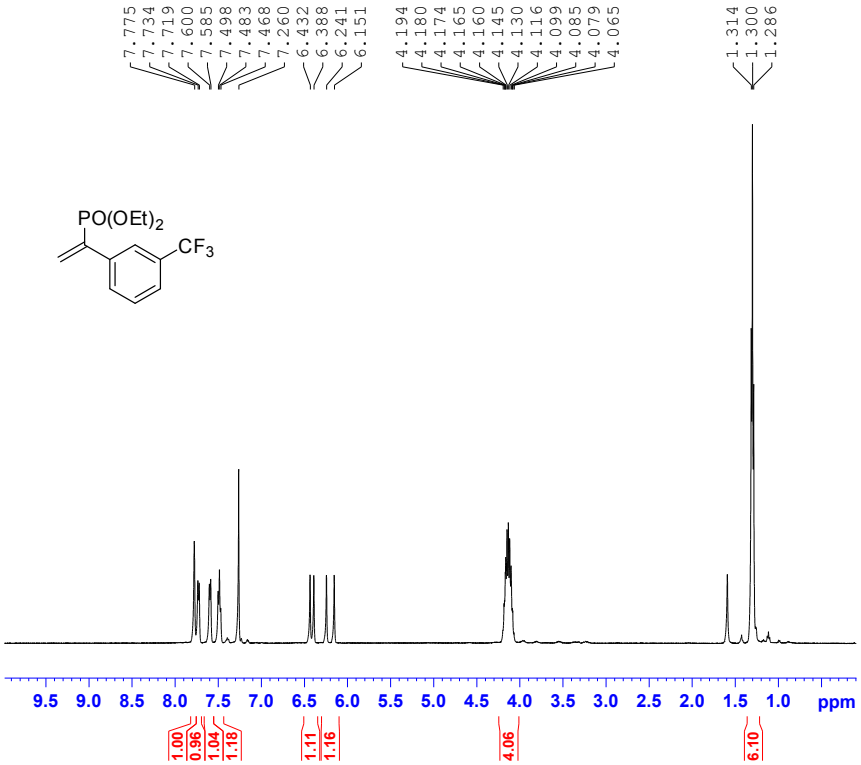



```
NAME      20151230fangyw
EXPNO                   50
PROCNO                   1
Date_           20151230
Time                10.30
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                131072
SOLVENT             CDCl3
NS                     16
DS                      4
SWH           113636.367 Hz
FIDRES          0.866977 Hz
AQ             0.5767668 sec
RG                201.95
DW                 4.400 usec
DE                 6.50 usec
TE                300.0 K
D1           1.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         470.5453180 MHz
NUC1                19F
P1                 14.75 usec
SI                 65536
SF           470.5923770 MHz
WDW                   no
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```

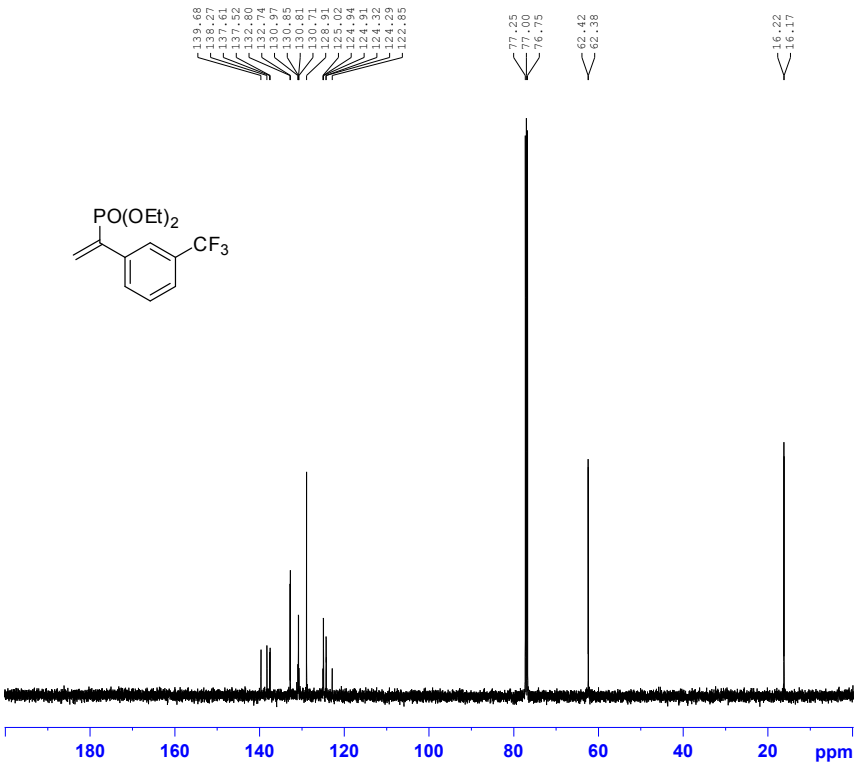


(12e)



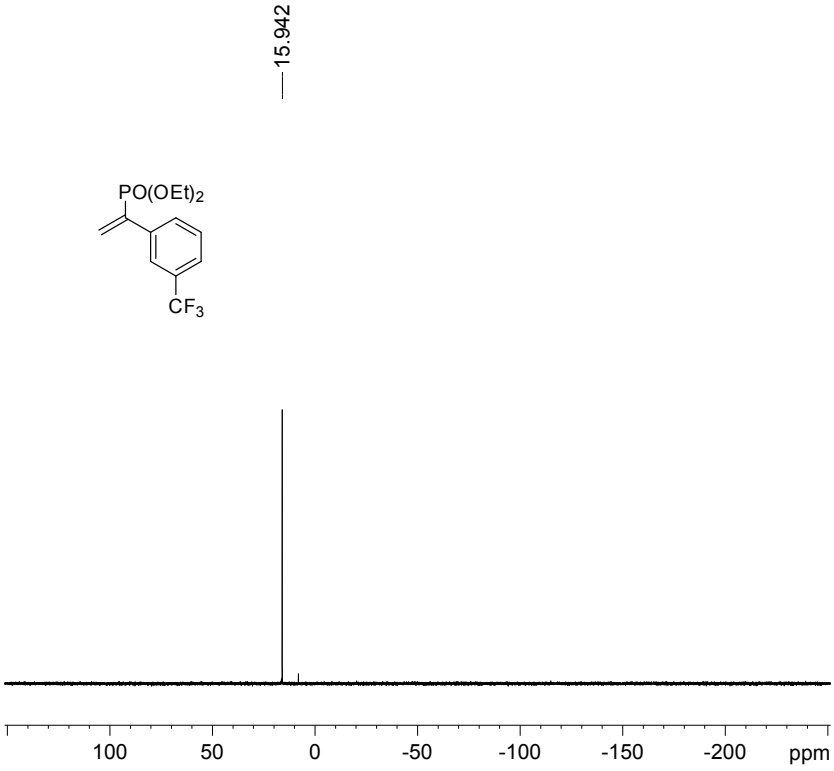

```
NAME        20151216fangyw-1
EXPNO                 20
PROCNO                 1
Date_           20151216
Time                10.45
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH            10000.000 Hz
FIDRES             0.152588 Hz
AQ              3.2768500 sec
RG                137.21
DW                 50.000 usec
DE                  6.50 usec
TE                 300.0 K
D1              1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          500.1330885 MHz
NUC1                  1H
P1                 11.11 usec
SI                 65536
SF            500.1300135 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



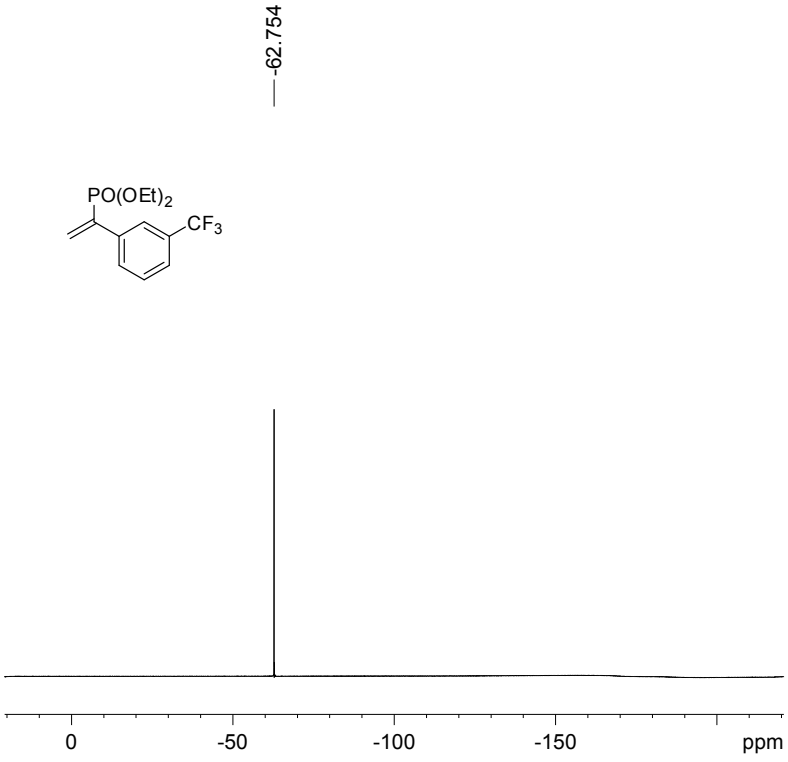

```
NAME        20151216fangyw-1
EXPNO                 22
PROCNO                 1
Date_           20151216
Time                14.15
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    99
DS                     4
SWH            29761.904 Hz
FIDRES             0.454131 Hz
AQ              1.1010548 sec
RG                201.95
DW                 16.800 usec
DE                  6.50 usec
TE                 300.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          125.7703637 MHz
NUC1                  13C
P1                 10.17 usec
SI                 32768
SF            125.7577925 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



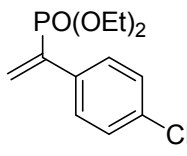

```
NAME          20170902
EXPNO               20
PROCNO               1
Date_         20170902
Time             11.10
INSTRUM          spect
PROBHD   5 mm PABBO B
PULPROG       zgpg30
TD             65536
SOLVENT         CDCl3
NS                 16
DS                  4
SWH         81521.742 Hz
FIDRES       1.243923 Hz
AQ          0.4020041 sec
RG              201.95
DW           6.133 usec
DE              6.50 usec
TE              296.1 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                 1

======== CHANNEL f1 ==
SFO1        202.4462121 MH
NUC1               31P
P1              13.00 usec
SI                 32768
SF          202.4563350 MHz
WDW                EM
```

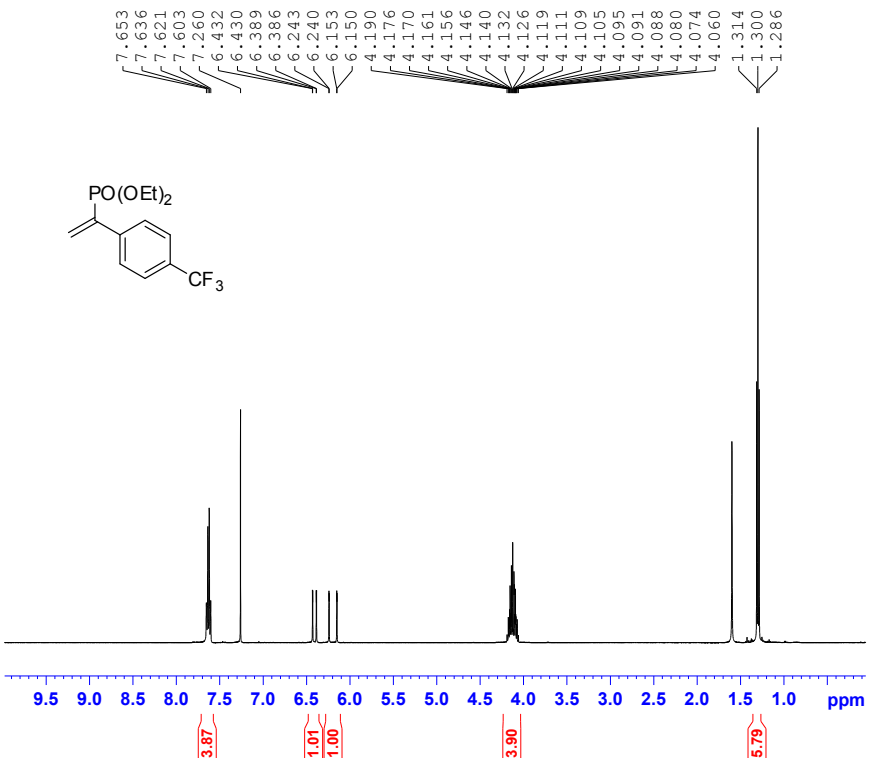



```
NAME      20151230fangyw
EXPNO                 30
PROCNO                 1
Date_           20151230
Time               10.18
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG      zgfhigqn.2
TD                131072
SOLVENT            CDCl3
NS                    16
DS                     4
SWH           113636.367 Hz
FIDRES          0.866977 Hz
AQ              0.5767668 sec
RG                 201.95
DW                  4.400 usec
DE                  6.50 usec
TE                300.0 K
D1            1.00000000 sec
D11           0.03000000 sec
D12           0.00002000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1         470.5453180 MHz
NUC1                  19F
P1                 14.75 usec
SI                 65536
SF           470.5923770 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



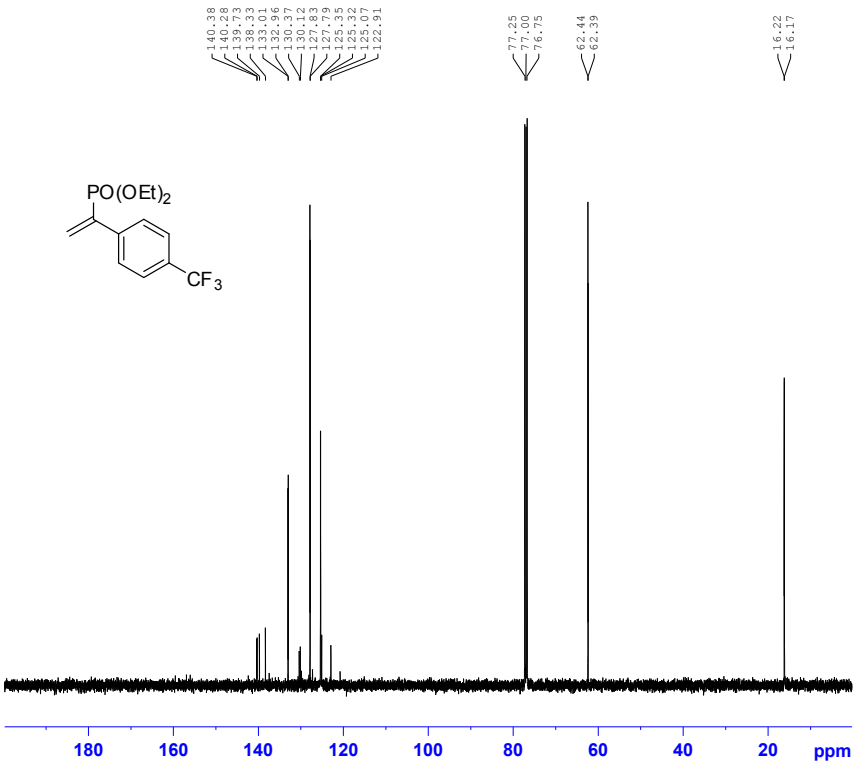
(12f)



BRUKER

```
NAME        20151118fangyw
EXPNO                   30
PROCNO                   1
Date_             20151118
Time                 11.28
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      16
DS                       2
SWH              10000.000 Hz
FIDRES               0.152588 Hz
AQ                3.2768500 sec
RG                   137.21
DW                   50.000 usec
DE                    6.50 usec
TE                   300.0 K
D1               1.00000000 sec
TD0                      1

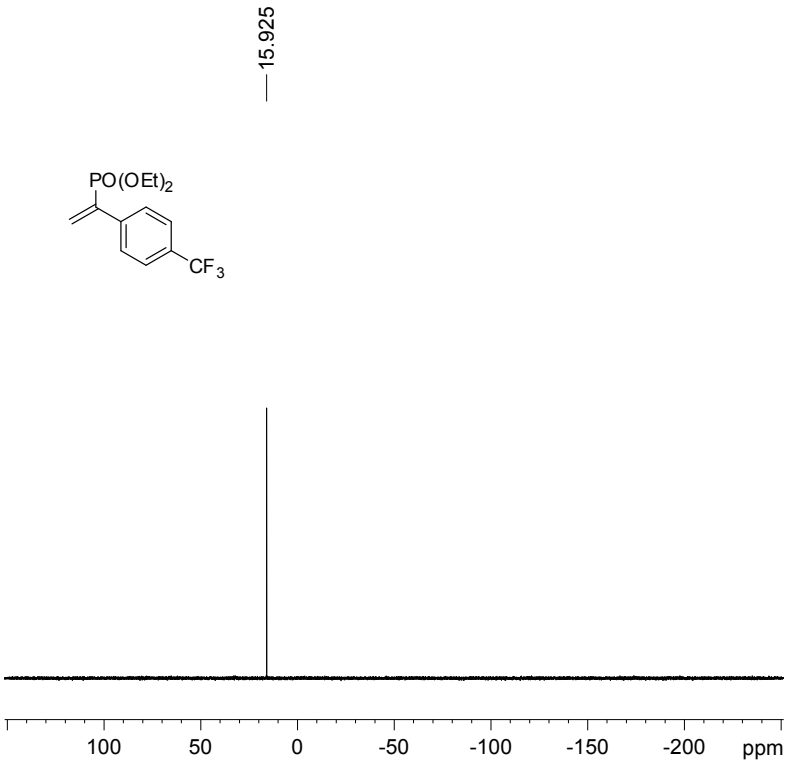
======== CHANNEL f1 ========
SFO1          500.1330885 MHz
NUC1                    1H
P1                   11.11 usec
SI                   65536
SF            500.1300135 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



BRUKER

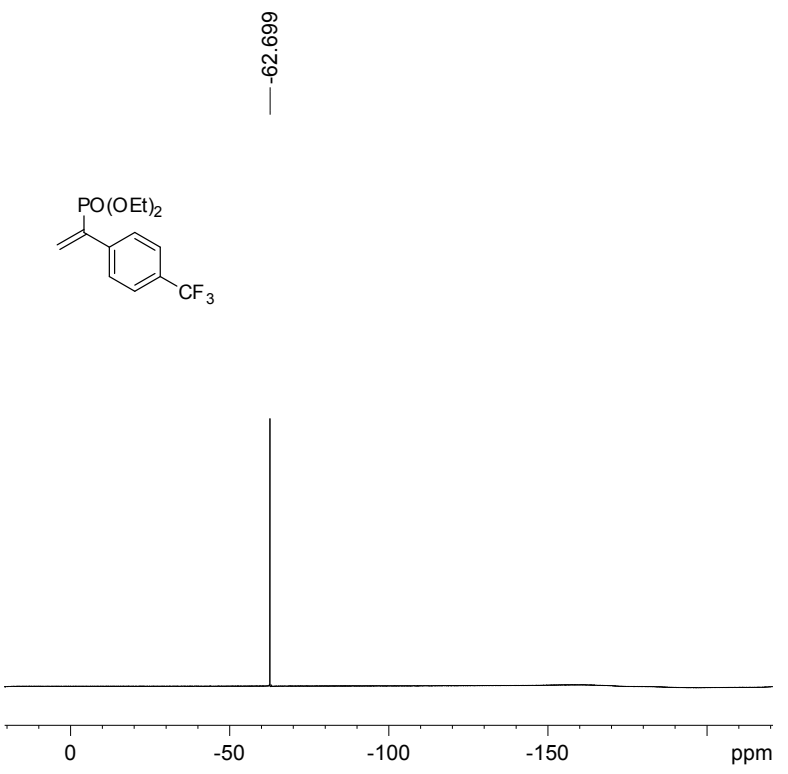
```
NAME        20151119fangyw
EXPNO                   32
PROCNO                   1
Date_             20151119
Time                 10.17
INSTRUM              spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     128
DS                       4
SWH              29761.904 Hz
FIDRES               0.454131 Hz
AQ                1.1010548 sec
RG                   201.95
DW                   16.800 usec
DE                    6.50 usec
TE                   300.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          125.7703637 MHz
NUC1                    13C
P1                   10.17 usec
SI                   32768
SF            125.7577939 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



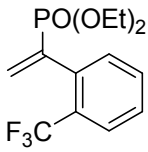

```
NAME      20151118fangyw
EXPNO               33
PROCNO               1
Date_         20151118
Time             11.30
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  32
DS                   4
SWH          81521.742 Hz
FIDRES        1.243923 Hz
AQ           0.4020041 sec
RG              201.95
DW               6.133 usec
DE                6.50 usec
TE               300.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1       202.4462121 MHz
NUC1               31P
P1               13.00 usec
SI               32768
SF         202.4563350 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

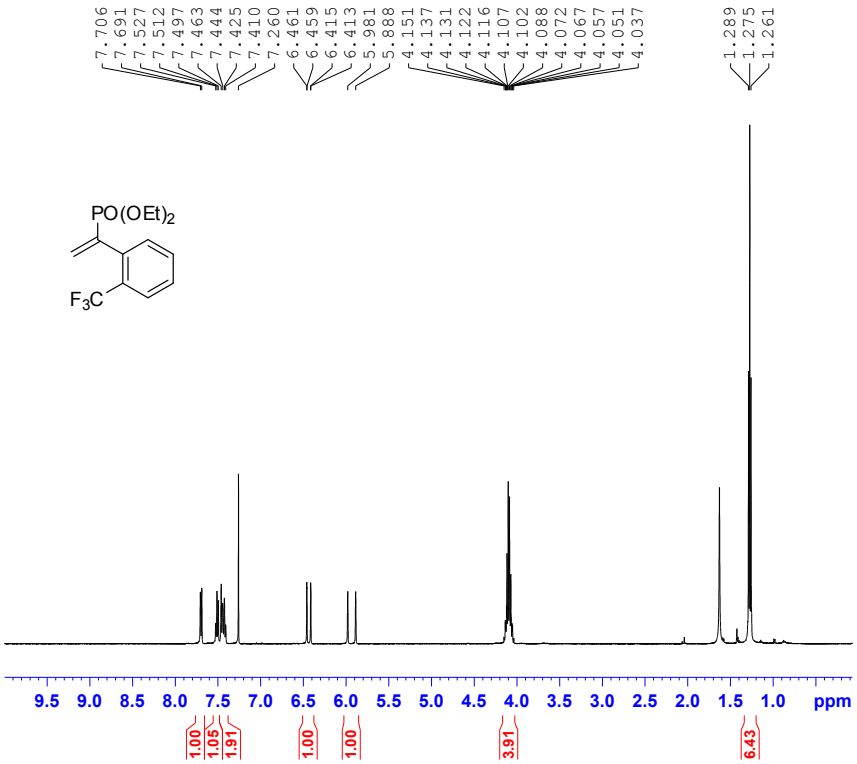



```
NAME      20151230fangyw
EXPNO               40
PROCNO               1
Date_         20151230
Time             10.24
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG      zgfhigqn.2
TD              131072
SOLVENT          CDCl3
NS                  16
DS                   4
SWH          113636.367 Hz
FIDRES         0.866977 Hz
AQ           0.5767668 sec
RG              201.95
DW               4.400 usec
DE                6.50 usec
TE               300.0 K
D1          1.00000000 sec
D11         0.03000000 sec
D12         0.00002000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1       470.5453180 MHz
NUC1               19F
P1               14.75 usec
SI               65536
SF         470.5923770 MHz
WDW                EM
SSB                 0
LB               0.30 Hz
GB                  0
PC               1.00
```



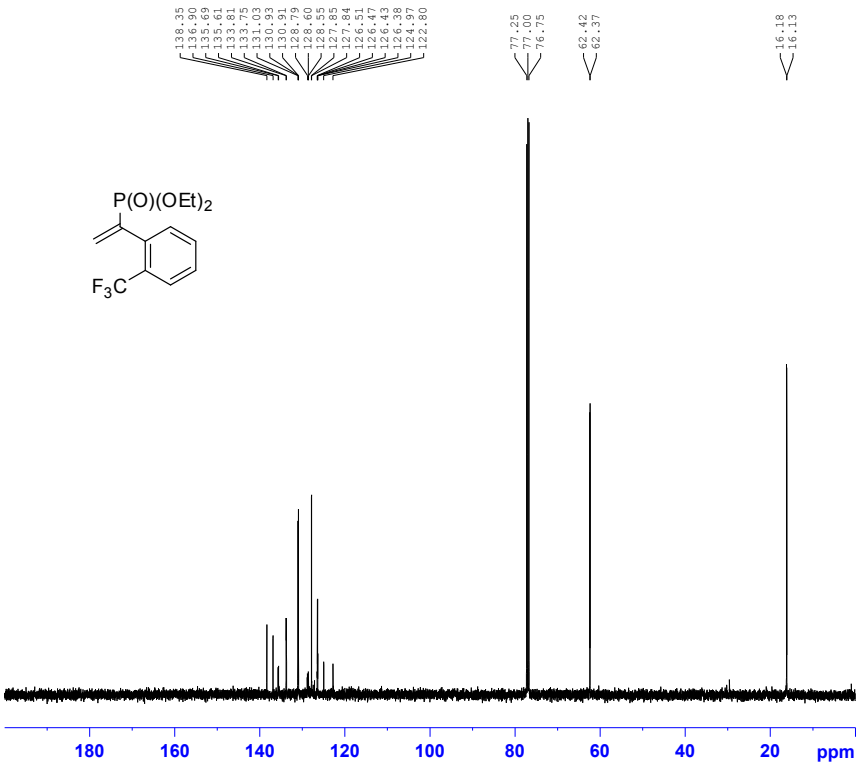
(12g)



BRUKER

```
NAME        20151202fangyw
EXPNO               20
PROCNO               1
Date_         20151202
Time              9.20
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                 16
DS                  2
SWH         10000.000 Hz
FIDRES          0.152588 Hz
AQ              3.2768500 sec
RG                121.57
DW                50.000 usec
DE                 6.50 usec
TE                300.0 K
D1          1.00000000 sec
TD0                 1

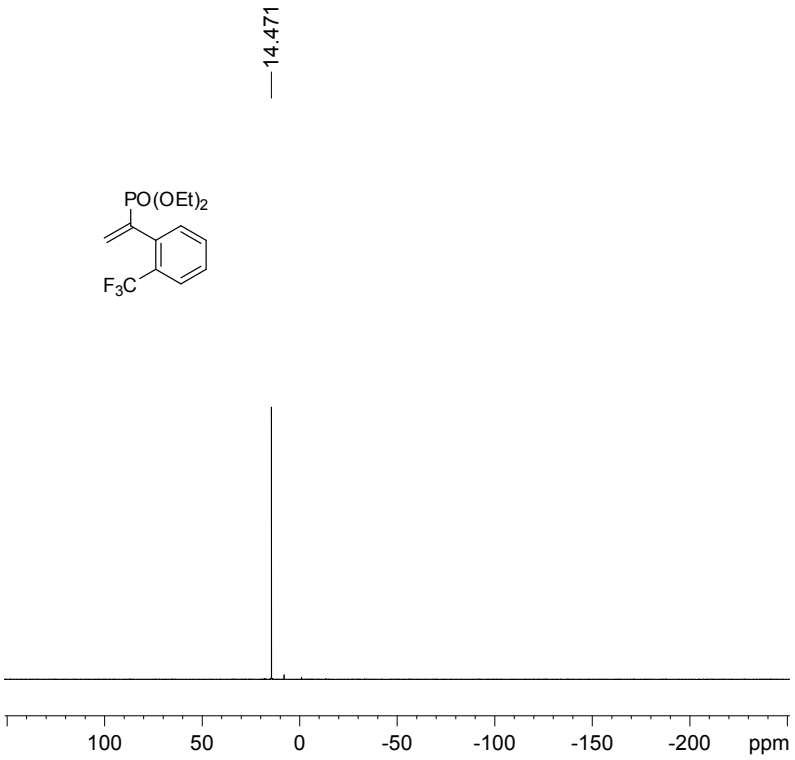
======== CHANNEL f1 ========
SFO1        500.1330885 MHz
NUC1               1H
P1                11.11 usec
SI               65536
SF          500.1300133 MHz
WDW                EM
SSB                 0
LB                0.30 Hz
GB                  0
PC                1.00
```



BRUKER

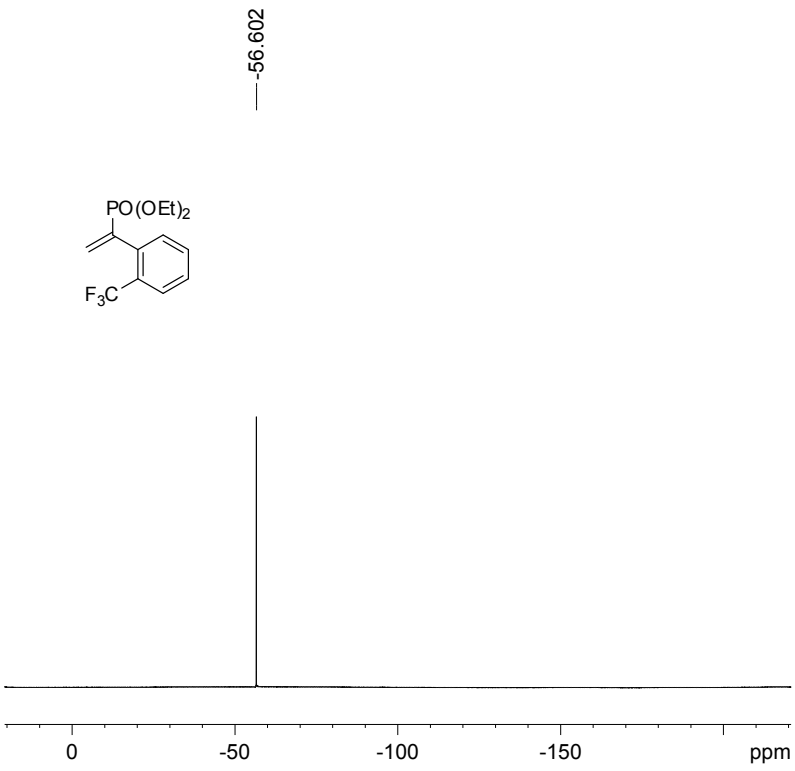
```
NAME        20151202fangyw
EXPNO               22
PROCNO               1
Date_         20151202
Time              14.24
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG        zgpg30
TD              65536
SOLVENT         CDCl3
NS                 84
DS                  4
SWH         29761.904 Hz
FIDRES          0.454131 Hz
AQ              1.1010548 sec
RG                201.95
DW                16.800 usec
DE                 6.50 usec
TE                300.0 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                 1

======== CHANNEL f1 ========
SFO1        125.7703637 MHz
NUC1               13C
P1                10.17 usec
SI               32768
SF          125.7577945 MHz
WDW                EM
SSB                 0
LB                1.00 Hz
GB                  0
PC                1.40
```



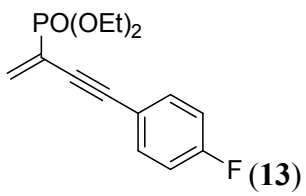

```
NAME      20151202fangyw
EXPNO               23
PROCNO               1
Date_         20151202
Time             14.18
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  16
DS                   4
SWH          81521.742 Hz
FIDRES        1.243923 Hz
AQ           0.4020041 sec
RG              201.95
DW               6.133 usec
DE                6.50 usec
TE               300.1 K
D1          2.00000000 sec
D11         0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        202.4462121 MHz
NUC1                31P
P1                13.00 usec
SI                32768
SF          202.4563350 MHz
WDW                 EM
SSB                  0
LB                1.00 Hz
GB                   0
PC                1.40
```

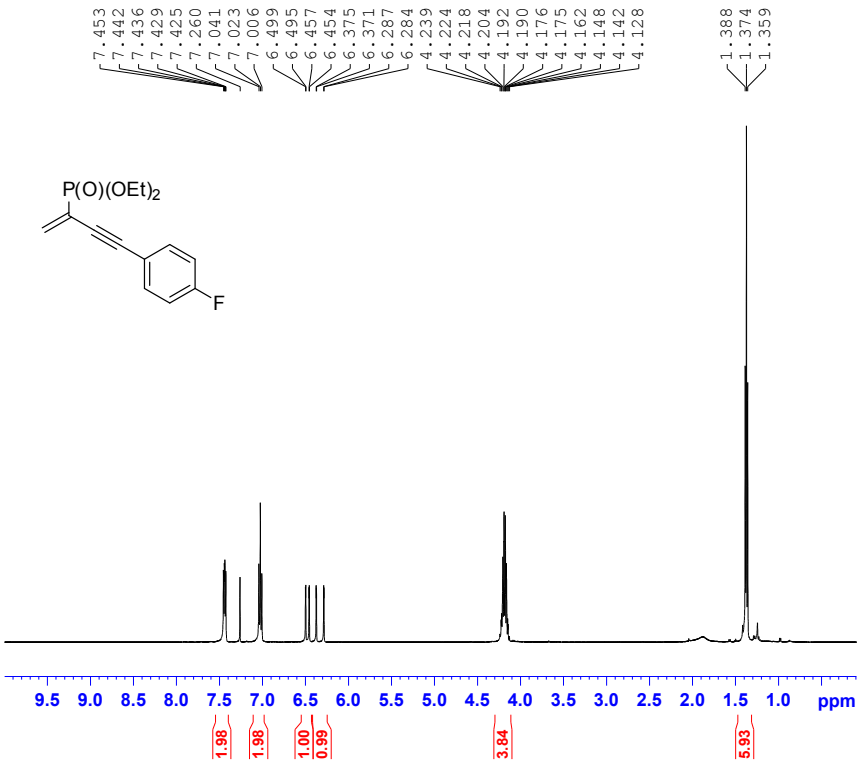



```
NAME      20151230fangyw
EXPNO               20
PROCNO               1
Date_         20151230
Time             10.13
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD              131072
SOLVENT          CDCl3
NS                  16
DS                   4
SWH         113636.367 Hz
FIDRES        0.866977 Hz
AQ           0.5767668 sec
RG              201.95
DW               4.400 usec
DE                6.50 usec
TE               300.0 K
D1          1.00000000 sec
D11         0.03000000 sec
D12         0.00002000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        470.5453180 MHz
NUC1                19F
P1                14.75 usec
SI                65536
SF          470.5923770 MHz
WDW                 EM
SSB                  0
LB                0.30 Hz
GB                   0
PC                1.00
```



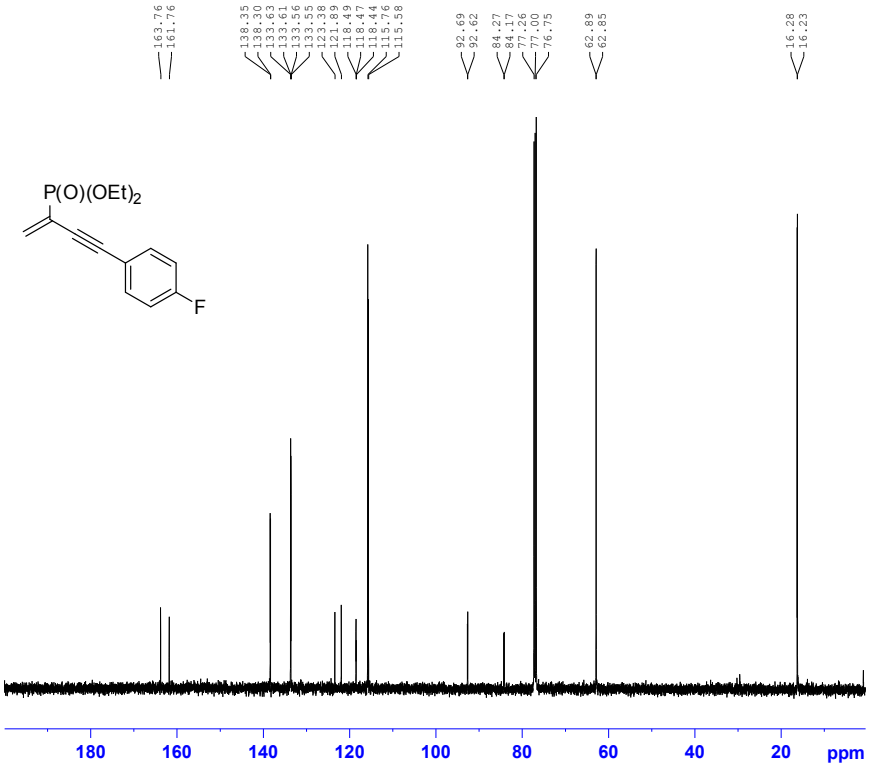
(13)



BRUKER

```
NAME          20151201fangyw
EXPNO                     10
PROCNO                     1
Date_              20151201
Time                  13.21
INSTRUM               spect
PROBHD     5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               10000.000 Hz
FIDRES            0.152588 Hz
AQ                3.2768500 sec
RG                    93.83
DW                   50.000 usec
DE                    6.50 usec
TE                   300.0 K
D1               1.00000000 sec
TD0                       1

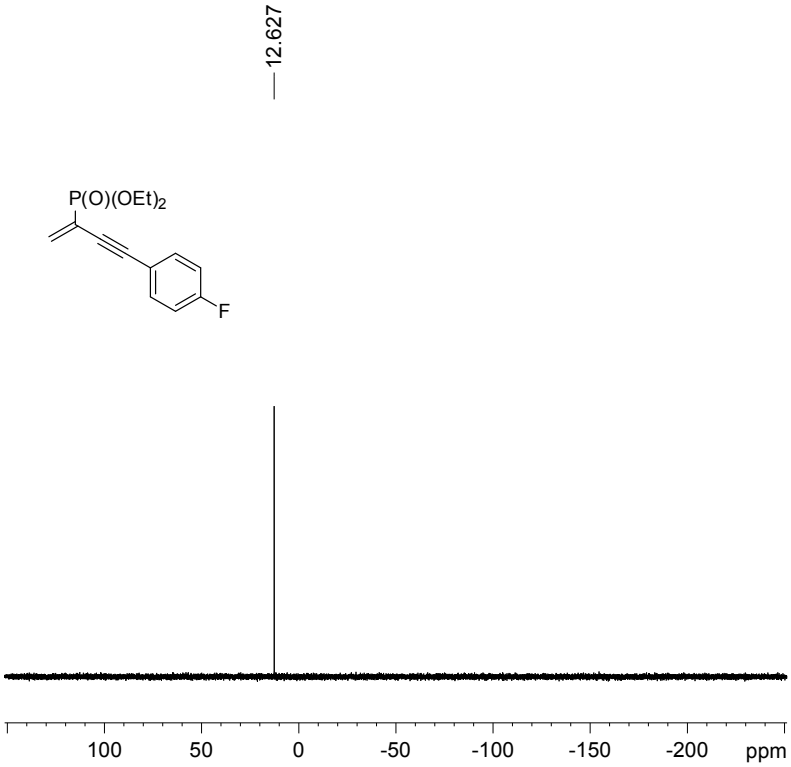
======== CHANNEL f1 ========
SFO1            500.1330885 MHz
NUC1                      1H
P1                    11.11 usec
SI                     65536
SF              500.1300132 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```



BRUKER

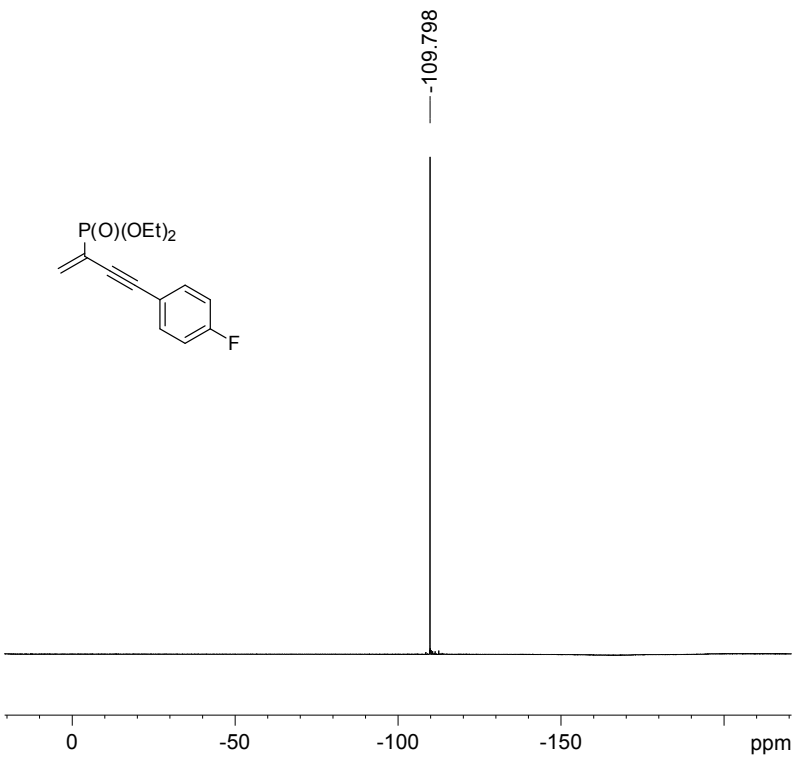
```
NAME          20151201fangyw
EXPNO                     12
PROCNO                     1
Date_              20151201
Time                  15.03
INSTRUM               spect
PROBHD     5 mm PABBO BB/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      128
DS                        4
SWH               29761.904 Hz
FIDRES            0.454131 Hz
AQ                1.1010548 sec
RG                   201.95
DW                   16.800 usec
DE                    6.50 usec
TE                   300.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            125.7703637 MHz
NUC1                      13C
P1                    10.17 usec
SI                     32768
SF              125.7577965 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```



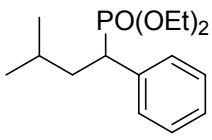

```
NAME      20151230fangyw
EXPNO               10
PROCNO               1
Date_         20151230
Time             10.07
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           81521.742 Hz
FIDRES         1.243923 Hz
AQ            0.4020041 sec
RG               201.95
DW                6.133 usec
DE                6.50 usec
TE               300.1 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        202.4462121 MHz
NUC1               31P
P1               13.00 usec
SI               32768
SF          202.4563350 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40
```

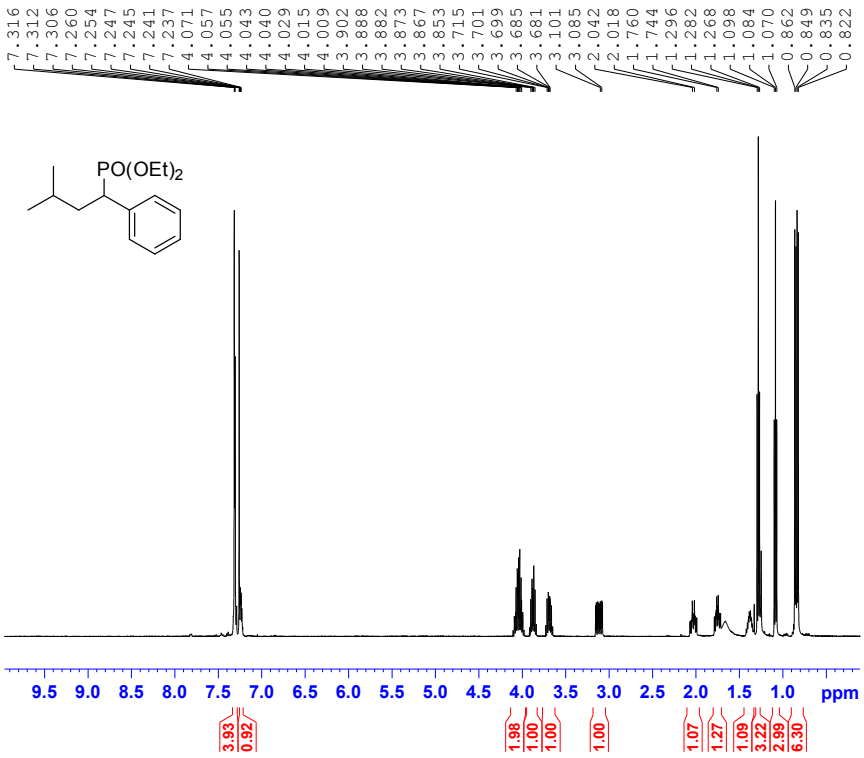



```
NAME      20151201fangyw
EXPNO              14
PROCNO              1
Date_         20151201
Time             14.55
INSTRUM          spect
PROBHD   5 mm PABBO BB/
PULPROG      zgfhigqn.2
TD               131072
SOLVENT           CDCl3
NS                   16
DS                    4
SWH          113636.367 Hz
FIDRES         0.866977 Hz
AQ            0.5767668 sec
RG               201.95
DW                4.400 usec
DE                6.50 usec
TE               300.0 K
D1           1.00000000 sec
D11          0.03000000 sec
D12          0.00002000 sec
TD0                  1

======== CHANNEL f1 ========
SFO1        470.5453180 MHz
NUC1               19F
P1               14.75 usec
SI               65536
SF          470.5923770 MHz
WDW                no
SSB                 0
LB               0.00 Hz
GB                  0
PC               1.00
```

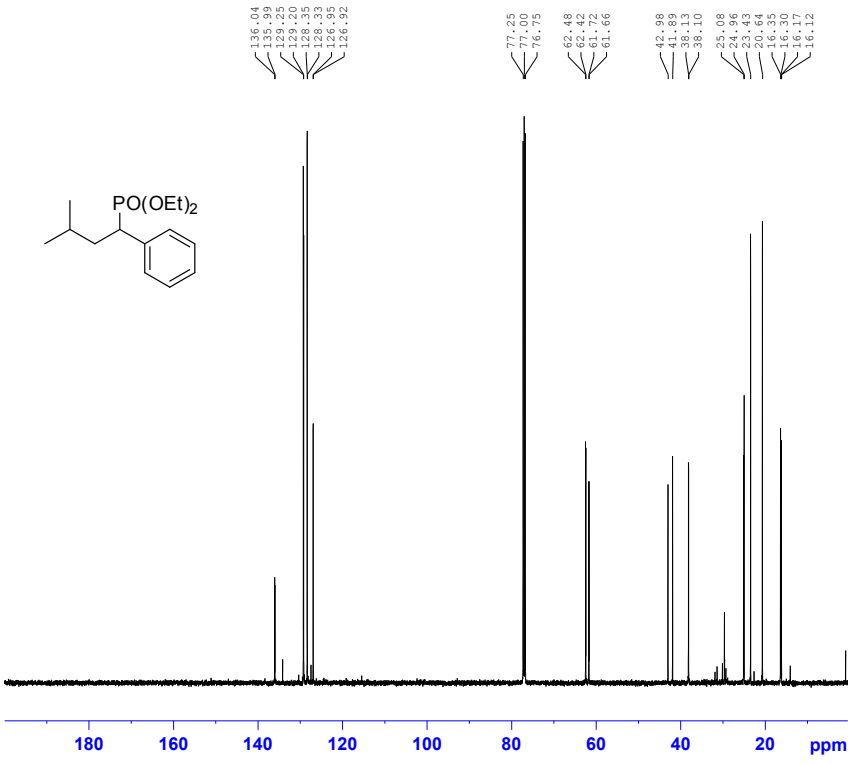


(18a)



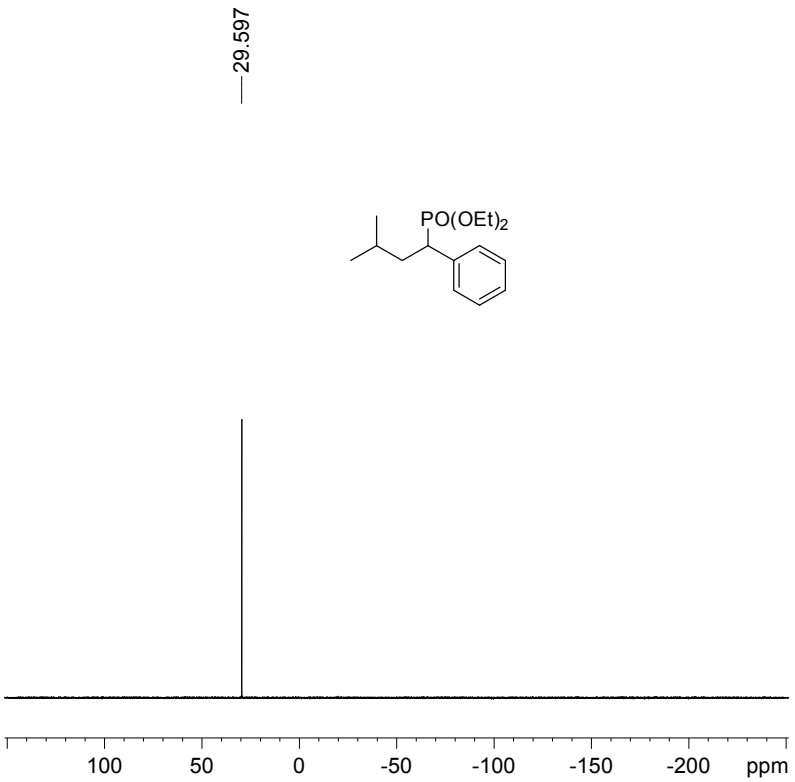

```
NAME            20170619 F
EXPNO                   40
PROCNO                   1
Date_             20170619
Time                 13.55
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDC13
NS                      16
DS                       2
SWH           10000.000 Hz
FIDRES            0.152588 Hz
AQ             3.2768500 sec
RG                  121.57
DW                  50.000 usec
DE                    6.50 usec
TE                  293.2 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          500.1330885 MHz
NUC1                    1H
P1                   11.11 usec
SI                   65536
SF            500.1300137 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```




```
NAME            20170620
EXPNO                   10
PROCNO                   1
Date_             20170620
Time                 10.36
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDC13
NS                     512
DS                       4
SWH           29761.904 Hz
FIDRES            0.454131 Hz
AQ             1.1010548 sec
RG                  201.95
DW                  16.800 usec
DE                    6.50 usec
TE                  294.3 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1          125.7703637 MHz
NUC1                    13C
P1                   10.17 usec
SI                   32768
SF            125.7577990 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



NAME 20170619 F
EXPNO 41
PROCNO 1
Date_ 20170619
Time 13.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 32
DS 4
SWH 81521.742 Hz
FIDRES 1.243923 Hz
AQ 0.4020041 sec
RG 201.95
DW 6.133 usec
DE 6.50 usec
TE 293.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ====
SFO1 202.4462121 MHz
NUC1 31P
P1 13.00 usec
SI 32768
SF 202.4563350 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40