Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.


# Supporting information

## A mild electroassisted synthesis of (hetero)arylphosphonates

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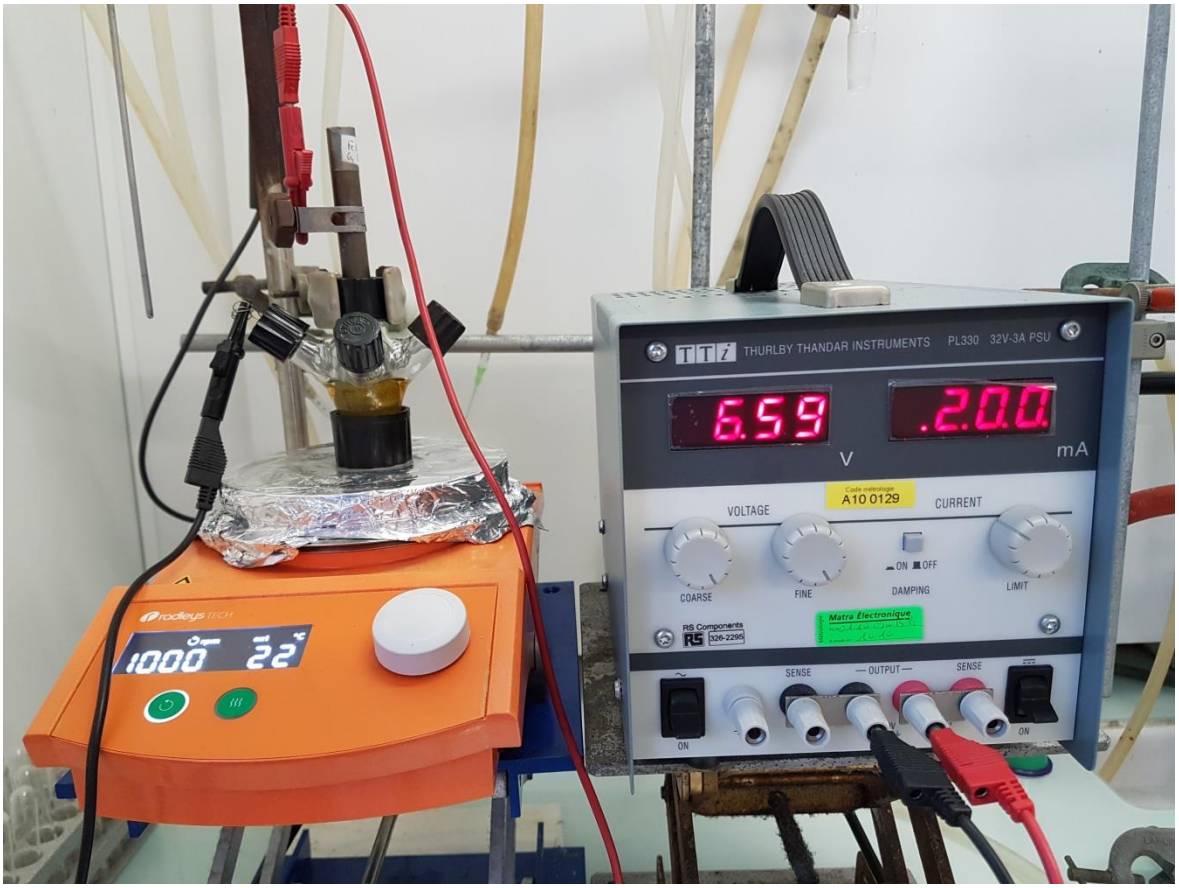
## General

Solvents and reagents were purchased from commercial suppliers and were used without further purification. (2,2'-Bipyridine)nickel bromide ($NiBr_2$bpy) was prepared from $NiBr_2 \cdot xH_2O$ and 2,2'-bipyridyl.[1] Reactions were monitored by gas chromatography (GC) using a chromatograph fitted with a capillary column (*l* = 5.5 m, *i.d.* = 0.25 mm, depth of film (*d.f.*) = 0.25 µm). Melting points (mp) were measured in unsealed capillary tubes. Infrared spectra (FTIR) were recorded in ATR mode. NMR spectra were recorded in $CDCl_3$ at 400 MHz ($^1$H), 100 MHz ($^{13}$C), 162 MHz ($^{31}$P) and 376 MHz ($^{19}$F). NMR spectra were calibrated using the residual solvent signal. Mass spectra [MS in electron-impact ($EI_+$) ionization mode] were measured with a GC-MS spectrometer fitted with a capillary column (*l* = 25 m, *i.d.* = 0.25 mm, *d.f.* = 0.25 µm). Purification was carried out manually by flash chromatography on silica gel (70–200 µm). All literature previously described and characterized compounds are linked with the corresponding bibliographic references.
Electrosyntheses were carried out in a 25 mL undivided cell fitted with an iron/nickel (64/36) rod anode (diameter: 12 mm, 30 mm of the surface is submerged in the reaction solution, purchased from Goodfellow) surrounded by a nickel foam cathode (dimension: 80 mm x 40 mm, purchased from Goodfellow). These electrode materials have proved to be the best in most previously described electrochemical couplings involving the generation of Ni(0) by electroreduction of nickel(II) salts. A simple digital electricity generator was used for the electrolyses under galvanostatic mode (Figure 1).

**Figure 1** The electrochemical cell with the generator

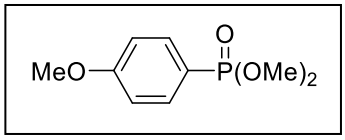


## General procedure for electrochemical (hetero)aryl and vinyl C-P couplings

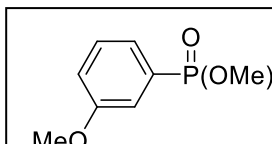
In an 25 mL electrochemical cell equipped with an iron/nickel (64/36) rod anode surrounded by a nickel foam cathode were successfully added acetonitrile (20 mL), tetrabutylammonium bromide (200 mg, 0.15 mmol), used as supporting electrolyte, and 1,2-dibromoethane (100 µL, 0.3 mmol). A constant current of 0.2 A was applied at room temperature for 15 min under argon bubbling, time during which electroreduction of 1,2-dibromoethane furnishes organometallic species that acts as water scavengers and additionally furnishes salts that increase medium conductibility. The electric current and argon bubbling were then stopped. $NiBr_2$bpy complex (10 mol%), (hetero)aryl or vinyl bromide (4 mmol), dimethyl phosphite (733 µL, 8 mmol) and trimethylamine (1.1 mL, 8 mmol) were added before a constant current of 0.2 A was applied at room temperature. The reaction was monitored by GC and stopped once aryl bromide was completely consumed (2-4 h). The reaction mixture was then poured in a saturated EDTA-$Na_2$ aqueous solution (50 mL) and the resulting solution was extracted with EtOAc (3×50 mL). The combined organic layers were washed with brine (50 mL), dried over $Na_2SO_4$, filtered, and concentrated under vacuum. The crude product was purified by flash chromatography (silica gel, 70–200 µm) to give the pure product.

## Characterization data for compounds (3a-x), (3'a), (3"a) and (5a-b)

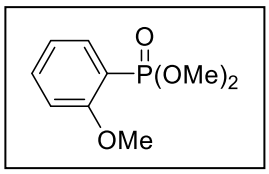
**Dimethyl (4-methoxyphenyl)phosphonate** (**3a**).[1] Pale yellow oil. Yield: 86% (740 mg). FC: elution with a gradient of petroleum ether/acetone (80/20), then (70/30) and (60/40); GC (60 °C, 8 °C/min): $t_R$ 14.31 min; ATR-FTIR (neat, $cm^{-1}$) *v* 2953, 2848, 1599, 1505, 1461, 1241, 1131, 1017, 810, 768, 535; $^1$H NMR (400 MHz) *δ* 7.73 (dd, *J* = 12.8, 8.8 Hz, 2H), 6.97 (dd, *J* = 8.8, 3.4 Hz, 2H), 3.85 (s, 3H), 3.73 (d, *J* = 11.1 Hz, 6H); $^{13}$C NMR (100 MHz) *δ* 163.1 (d, *J* = 3.4 Hz), 133.9 (d, *J* = 11.3 Hz), 118.1 (d, *J* = 195.9 Hz), 114.1 (d, *J* = 16.1 Hz), 55.3, 52.5 (d, *J* = 5.5 Hz); $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 22.56; MS, *m/z* (relative intensity) 217 (13), 216 ($[M]^{\cdot+}$, 92), 215 (100), 201 (9), 186 (11), 185 (17), 183 (19), 171 (56), 170 (6), 135 (6), 123 (8), 122 (12), 121 (67), 108 (26), 93 (6), 91 (10), 79 (6), 78 (12), 77 (16), 63 (12).



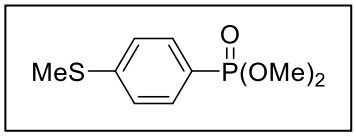

**Dimethyl (3-methoxyphenyl)phosphonate** (**3b**).[2] Pale yellow oil. Yield: 75% (650 mg). FC: elution with a gradient of petroleum ether/acetone (80/20), then (70/30) and (60/40); GC (60 °C, 8 °C/min): $t_R$ 13.78 min; ATR-FTIR (neat, $cm^{-1}$) *v* 2954, 2850, 1578, 1464, 1420, 1237, 1017, 827, 766, 557; $^1$H NMR (400 MHz) *δ* 7.41-7.27 (m, 3H), 7.11-7.06 (m, 1H), 3.83 (s, 3H), 3.75 (d, *J* = 11.1 Hz, 6H); $^{13}$C NMR (100 MHz) *δ* 159.6 (d, *J* = 19.0 Hz), 129.8 (d, *J* = 17.7 Hz), 128.2 (d, *J* = 187.7 Hz), 124.1 (d, *J* = 9.2 Hz), 119.1 (d, *J* = 3.2 Hz), 116.5 (d, *J* = 11.3 Hz), 55.4 , 52.70 (d, *J* = 5.5 Hz); $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 21.44; MS, *m/z* (relative intensity) 217 (9), 216 ($[M]^{\cdot+}$, 75), 215 (100), 201 (11), 186 (19), 185 (13), 183 (18), 171 (14), 154 (7), 121 (19), 108 (12), 92 (5), 91 (7), 79 (7), 78 (12), 77 (12), 63 (9).




**Dimethyl (2-methoxyphenyl)phosphonate** (**3c**).[2] Pale yellow oil. Yield: 35% (300 mg). FC: elution with a gradient of petroleum ether/acetone (80/20), then (70/30), (60/40) and (50/50); GC (60 °C, 8 °C/min): $t_R$ 13.82 min; ATR-FTIR (neat, $cm^{-1}$) *v* 2953, 2849, 1592, 1480, 1433, 1248, 1018, 807, 760, 584; $^1$H NMR (400 MHz) *δ* 7.79 (ddd, *J* = 14.9, 7.6, 1.7 Hz, 1H), 7.50 (ddd, *J* = 7.5, 4.5, 0.8 Hz, 1H), 7.00 (tdd, *J* = 7.5, 3.5, 0.7 Hz, 1H), 6.97-6.90 (m, 1H), 3.89 (s, 3H), 3.78 (d, *J* = 11.3 Hz, 6H); $^{13}$C NMR (100 MHz) *δ* 161.3 (d, *J* = 2.6 Hz), 135.2 (d, *J* = 7.0 Hz), 134.5 (d, *J* = 2.1 Hz), 120.5 (d, *J* = 14.6 Hz), 115.5 (d, *J* = 188.7 Hz), 111.2 (d, *J* = 9.5 Hz), 55.9, 52.81 (d, *J* = 5.7 Hz); $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 23.33; MS, *m/z* (relative intensity) 217 (11), 216 ($[M]^{\cdot+}$, 100), 215 (46), 187 (92), 185 (52), 184 (40), 183 (91), 169 (52), 155 (59), 153 (62), 141 (48), 110 (35), 104 (17), 91 (41), 80 (13), 79 (24), 78 (17), 77 (35), 63 (16), 51 (19).



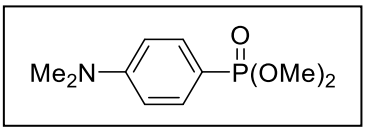

**Dimethyl (4-(methylthio)phenyl)phosphonate** (**3d**). Pale brown oil. Yield: 82% (760 mg). FC: elution with a gradient of petroleum ether/acetone (90/10); GC (60 °C, 8 °C/min): $t_R$ 16.83 min; ATR-FTIR (neat, $cm^{-1}$) *v* 2952, 2850, 1583, 1247, 1014, 786, 592; $^1$H NMR (400 MHz) *δ* 7.70 (dd, *J* = 12.9, 8.3 Hz, 2H), 7.31 (dd, *J* = 8.3, 3.6 Hz, 2H), 3.76 (d, *J* = 11.1 Hz, 6H), 2.53 (s, 3H); $^{13}$C NMR (100 MHz) *δ* 145.3 (d, *J* = 3.5 Hz), 132.2 (d, *J* = 10.6 Hz), 125.2 (d, *J* = 15.5 Hz), 122.3 (d, *J* = 193.1 Hz), 52.7 (d, *J* = 5.5 Hz), 14.8; $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 21.96; MS, *m/z* (relative intensity) 233 (12), 232 ($[M]^{\cdot+}$, 100), 231 (32), 217 (14), 201 (8), 199 (10), 187 (27), 186 (14), 185 (5), 139 (7),



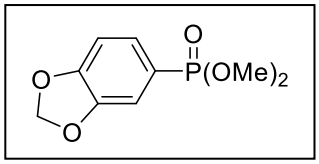

[1] S. Wang, D. Qiu, F. Mo, Y. Zhang, J. Wang, *J. Org. Chem.*, 2016, **81**, 11603.
[2] A. J. Kendall, C. A. Salazar, P. F. Martino, D. R. Tyler, *Organometallics*, 2014, **33**, 6171.

138 (6), 137 (60), 124 (15), 122 (5), 91 (6). HRMS (ESI$^+$) *m/z* calcd for $C_9H_{14}O_3PS$ [M + H], 233.039578; found, 233.039276.

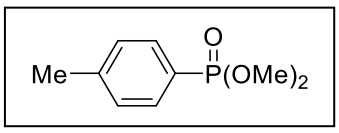
**Dimethyl (4-(dimethylamino)phenyl)phosphonate** (**3e**). Pale yellow oil. Yield: 75% (690 mg). FC: elution with a gradient of petroleum ether/acetone (80/20), then (70/30); GC (60 °C, 8 °C/min): $t_R$ 17.72 min; ATR-FTIR (neat, cm$^{-1}$) *v* 2950, 2849, 1599, 1521, 1237, 1127, 1017, 793, 532; $^1$H NMR (400 MHz) *δ* 7.61 (dd, *J* = 12.5, 9.0 Hz, 2H), 6.69 (dd, *J* = 9.0, 3.5 Hz, 2H), 3.69 (d, *J* = 11.1 Hz, 6H), 3.00 (s, 6H); $^{13}$C NMR (100 MHz) *δ* 153.0 (d, *J* = 2.8 Hz), 133.5 (d, *J* = 11.4 Hz), 111.2 (d, *J* = 15.7 Hz), 111.0 (d, *J* = 199.9 Hz),.52.3 (d, *J* = 5.3 Hz), 39.9; $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 24.73; MS, *m/z* (relative intensity) 230 (11), 229 ([M]$^{\cdot+}$, 100), 228 (65), 214 (7), 198 (5), 196 (12), 184 (11), 183 (7), 182 (11), 135 (5), 134 (33), 121 (9), 120 (8), 118 (10), 91 (5); HRMS (ESI$^+$) *m/z* calcd for $C_{10}H_{17}NO_3P$ [M + H], 230.094056; found, 230.093991.



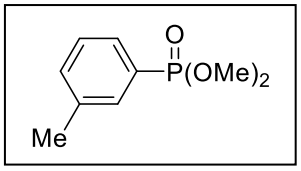

**Dimethyl benzo[d][1,3]dioxol-5-ylphosphonate** (**3f**). Pale brown oil. Yield: 84% (770 mg). FC: elution with a gradient of petroleum ether/acetone (80/20), then (70/30); GC (60 °C, 8 °C/min): $t_R$ 15.45 min; ATR-FTIR (neat, cm$^{-1}$) *v* 2954, 2851, 1481, 1426, 1241, 1017, 814, 777, 539; $^1$H NMR (400 MHz) *δ* 7.35 (ddd, *J* = 14.0, 7.9, 1.4 Hz, 1H), 7.16 (dd, *J* = 12.9, 1.0 Hz, 1H), 6.87 (dd, *J* = 7.9, 3.7 Hz, 1H), 6.01 (s, 2H), 3.72 (d, *J* = 11.1 Hz, 6H); $^{13}$C NMR (100 MHz) *δ* 151.4 (d, *J* = 3.5 Hz), 148.0 (d, *J* = 22.8 Hz), 127.7 (d, *J* = 11.1 Hz), 119.7 (d, *J* = 194.5 Hz), 111.3 (d, *J* = 12.3 Hz), 108.7 (d, *J* = 18.8 Hz), 101.7, 52.7 (d, *J* = 5.4 Hz); $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 21.86; MS, *m/z* (relative intensity) 231 (11), ([M]$^{\cdot+}$, 100), 229 (74), 215 (15), 200 (8), 199 (19), 197 (19), 185 (36), 184 (10), 183 (8), 136 (10), 135 (92), 122 (33), 121 (15), 79 (9), 77 (10), 63 (7), 62 (7); HRMS (ESI$^+$) *m/z* calcd for $C_9H_{12}O_5P$ [M + H], 231.041687; found, 231.041399.




**Dimethyl (4-methylphenyl)phosphonate** (**3g**).[3] Pale yellow oil. Yield: 74% (590 mg). FC: elution with a gradient of petroleum ether/acetone (90/10), then (80/20); GC (60 °C, 8 °C/min): $t_R$ 12.04 min; ATR-FTIR (neat, cm$^{-1}$) *v* 2953, 2851, 1459, 1249, 1129, 1018, 807, 767, 517; $^1$H NMR (400 MHz) *δ* 7.67 (dd, *J* = 13.1, 7.9 Hz, 2H), 7.27 (dd, *J* = 6.7, 3.1 Hz, 2H), 3.73 (d, *J* = 11.1 Hz, 6H), 2.39 (s, 3H); $^{13}$C NMR (100 MHz) *δ* 143.3 (d, *J* = 3.1 Hz), 132.0 (d, *J* = 10.3 Hz), 129.3 (d, *J* = 15.5 Hz), 123.5 (d, *J* = 190.9 Hz), 52.6 (d, *J* = 5.5 Hz), 21.7; $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 22.40; MS, *m/z* (relative intensity) 200 ([M]$^{\cdot+}$, 34), 199 (100), 170 (5), 169 (13), 167 (13), 155 (37), 137 (6), 106 (5), 105 (48), 103 (7), 92 (5), 91 (33), 79 (8), 77 (7), 65 (10), 63 (6).



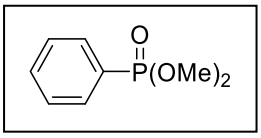

**Dimethyl (3-methylphenyl)phosphonate** (**3h**).[4] Pale yellow oil. Yield: 81% (650 mg). FC: elution with a gradient of petroleum ether/acetone (90/10), then (80/20); GC (60 °C, 8 °C/min): $t_R$ 11.87 min; ATR-FTIR (neat, cm$^{-1}$) *v* 2953, 2851, 1458, 1412, 1248, 1018, 826, 768, 563; $^1$H NMR (400 MHz) *δ* 7.64-7.53 (m, 2H), 7.38-7.31 (m, 2H), 3.74 (d, *J* = 11.1 Hz, 6H), 2.38 (s, 3H); $^{13}$C NMR (100 MHz) *δ* 138.4 (d, *J* = 15.0 Hz), 133.5 (d, *J* = 3.2 Hz), 132.4 (d, *J* = 10.0 Hz), 128.9 (d,



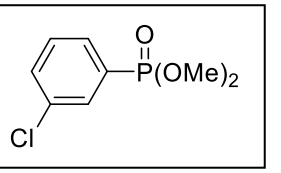

[3] J. Li, X. Bi, H. Wang and J. Xiao, *RSC Adv.*, 2014, **4**, 19214.
[4] T.-H. Chen, D. M. Reddy, C.-F. Lee, *RSC Adv*., 2017, **7**, 30214.

*J* = 9.7 Hz), 128.5 (d, *J* = 15.9 Hz), 126.7 (d, *J* = 187.8 Hz), 52.7 (d, *J* = 5.5 Hz), 21.3; $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 22.09; MS, *m/z* (relative intensity) 201 (6), 200 ($[M]^{\cdot+}$, 34), 199 (100), 185 (5), 170 (6), 169 (14), 167 (10), 155 (27), 105 (26), 92 (6), 91 (32), 89 (8), 79 (7), 65 (10), 63 (5).

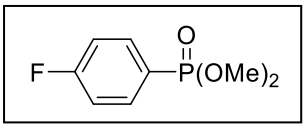
**Dimethyl phenylphosphonate** (**3j**).[5] Pale yellow oil. Yield: 81% (600 mg). FC: elution with a gradient of petroleum ether/acetone (90/10), then (80/20) and (70/30); GC (60 °C, 8 °C/min): $t_R$ 10.63 min; ATR-FTIR (neat, $cm^{-1}$) *v* 2954, 2851, 1440, 1242, 1131, 1017, 786, 750, 557; $^1$H NMR (400 MHz) *δ* 7.83-7.73 (m, 2H), 7.58 -7.51 (m, 2H), 7.49-7.40 (m, 2H), 3.73 (d, *J* = 11.1 Hz, 6H); $^{13}$C NMR (100 MHz) *δ* 132.6 (d, *J* = 3.0 Hz), 131.9 (d, *J* = 9.9 Hz), 128.5 (d, *J* = 15.1 Hz), 127.0 (d, *J* = 188.7 Hz), 52.7 (d, *J* = 5.6 Hz); $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 21.56; MS, *m/z* (relative intensity) 187 ($[M+H]^{\cdot+}$, 6), 186 ($[M]^{\cdot+}$, 22), 185 (100), 156 (7), 155 (16), 153 (8), 141 (34), 105 (6), 104 (7), 92 (5), 91 (39), 79 (7), 78 (14), 77 (22), 51 (14).



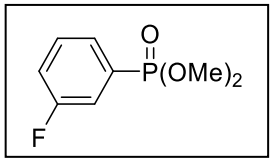

**Dimethyl (3-chlorophenyl)phosphonate** (**3k**).[6] Pale yellow oil. Yield: 59% (520 mg). FC: elution with a gradient of petroleum ether/acetone (90/10), then (80/20); GC (60 °C, 8 °C/min): $t_R$ 12.54 min; ATR-FTIR (neat, $cm^{-1}$) *v* 2954, 2851, 1566, 1468, 1403, 1249, 1143, 1018, 817, 747, 560; $^1$H NMR (400 MHz) *δ* 7.78-7.72 (m, 1H), 7.66 (ddd, *J* = 13.0, 7.5, 1.1 Hz, 1H), 7.54-7.48 (m, 1H), 7.40 (td, *J* = 7.8, 4.9 Hz, 1H), 3.75 (d, *J* = 11.1 Hz, 6H); $^{13}$C NMR (100 MHz) *δ* 134.9 (d, *J* = 20.4 Hz), 132.7 (d, *J* = 3.1 Hz), 131.8 (d, *J* = 10.7 Hz), 130.0 (d, *J* = 16.5 Hz), 129.9 (d, *J* = 9.2 Hz), 129.4 (d, *J* = 188.8 Hz), 52.9 (d, *J* = 5.6 Hz); $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 19.30; MS, *m/z* (relative intensity) 222 (14), 221 (36), 220 ($[M]^{\cdot+}$, 36), 219 (100), 190 (11), 189 (14), 187 (6), 185 (10), 175 (20), 139 (8), 138 (7), 127 (6), 125 (22), 112 (11), 111 (8), 91 (7), 79 (8), 77 (8), 75 (15), 74 (8).




**Dimethyl (4-fluorophenyl)phosphonate** (**3l**).[6] Pale yellow oil. Yield: 78% (640 mg). FC: elution with a gradient of petroleum ether/acetone (90/10), then (80/20); GC (60 °C, 8 °C/min): $t_R$ 9.68 min; ATR-FTIR (neat, $cm^{-1}$) *v* 3478, 2956, 2853, 1592, 1502, 1462, 1228, 1129, 1015, 820, 774, 505; $^1$H NMR (400 MHz) *δ* 7.86-7.71 (m, 2H), 7.14 (td, *J* = 8.4, 2.9 Hz, 2H), 3.73 (d, *J* = 11.1 Hz, 6H); $^{13}$C NMR (100 MHz) *δ* 165.5 (dd, *J* = 253.9, 3.9 Hz), 134.5 (dd, *J* = 11.3, 8.9 Hz), 123.1 (dd, *J* = 193.6, 3.4 Hz), 115.9 (dd, *J* = 21.5, 16.4 Hz), 52.7 (d, *J* = 5.5 Hz); $^{19}$F NMR ($CDCl_3$, 376 MHz): δ -105.51; $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 20.58; MS, *m/z* (relative intensity) 205 (5), 204 ($[M]^{\cdot+}$, 21), 203 (100), 174 (13), 173 (18), 171 (7), 159 (45), 123 (5), 122 (8), 110 (6), 109 (49), 96 (11), 95 (7), 83 (5), 79 (7), 75 (13), 74 (8).



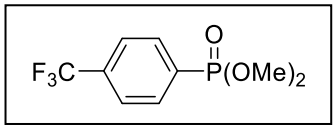

**Dimethyl (3-fluorophenyl)phosphonate** (**3m**).[2] Pale yellow oil. Yield: 69% (560 mg). FC: elution with a gradient of petroleum ether/acetone (90/10), then (80/20); GC (60 °C, 8 °C/min): $t_R$ 9.82 min; ATR-FTIR (neat, $cm^{-1}$) *v* 2956, 2853, 1421, 1253, 1225, 1020, 829, 770, 685, 559; $^1$H NMR (400 MHz) *δ* 7.58 (dd, *J* = 12.8, 7.5 Hz, 1H), 7.53-7.41 (m, 2H), 7.30-7.22 (m, 1H), 3.77 (d, *J* = 11.1 Hz, 6H); $^{13}$C NMR (100 MHz) *δ* 162.5 (dd, *J* = 249.7, 21.6 Hz), 130.6 (dd, *J* = 17.7, 7.5 Hz), 128.6 (d, *J* = 6.2 Hz), 127.6 (dd, *J* = 9.2, 3.3 Hz), 119.9 (dd, *J* = 21.1, 3.1 Hz), 118.8 (dd, *J* = 22.4, 10.5 Hz), 52.9 (d, *J* = 5.6 Hz); $^{19}$F NMR ($CDCl_3$, 376 MHz): δ -111.29 (d, *J* = 8.7 Hz); $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 19.48 (d, *J* = 8.7 Hz); MS, *m/z*




[5] M. Kalek, A. Ziadi, J. Stawinski, *Org. Lett.*, 2008, **10**, 4637.
[6] S.-Y. Chen, R.-S. Zeng, J.-P. Zou, O. T. Asekun, *J. Org. Chem.*, 2014, **79**, 1449.

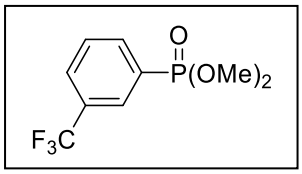
(relative intensity) 204 ([M]$^{+}$, 28), 203 (100), 174 (10), 173 (22), 171 (6), 159 (30), 123 (9), 122 (15), 110 (12), 109 (40), 96 (21), 95 (8), 83 (5), 79 (9), 77 (6), 75 (21), 74 (12).

**Dimethyl (4-(trifluoromethyl)phenyl)phosphonate** (**3n**).[7] Pale yellow oil. Yield: 59% (600 mg). FC:



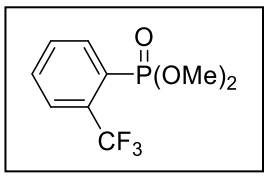
elution with a gradient of petroleum ether/acetone (90/10), then (80/20) and (70/30); GC (60 °C, 8 °C/min): $t_R$ 9.51 min; ATR-FTIR (neat, cm$^{-1}$) *v* 2958, 2855, 1462, 1400, 1322, 1288, 1254, 1127, 1014, 827, 804, 601; $^{1}$H NMR (400 MHz) *δ* 7.90 (dd, *J* = 13.0, 7.9 Hz, 2H), 7.70 (dd, *J* = 8.0, 3.5 Hz, 2H), 3.76 (d, *J* = 11.1 Hz, 6H); $^{13}$C NMR (100 MHz) *δ* 134.3 (qd, *J* = 32.7, 3.2 Hz), 132.4 (d, *J* = 10.1 Hz), 130.5, 125.4 (dq, *J* = 15.2, 3.7 Hz), 123.5 (q, *J* = 273.1 Hz), 52.9 (d, *J* = 5.7 Hz); $^{19}$F NMR ($CDCl_3$, 376 MHz): δ -63.42 (d, *J* = 1.0 Hz); $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 19.00; MS, *m/z* (relative intensity) 254 ([M]$^{+}$, 22), 253 (100), 235 (13), 234 (15), 224 (12), 223 (22), 221 (7), 209 (22), 172 (6), 160 (7), 159 (27), 146 (9), 145 (17), 140 (26), 127 (8), 125 (6), 109 (5), 95 (6), 93 5), 79 (12).

**Dimethyl (3-(trifluoromethyl)phenyl)phosphonate** (**3o**).[8] Pale yellow oil. Yield: 65% (660 mg). FC:



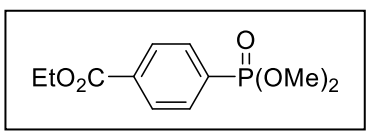
elution with a gradient of petroleum ether/acetone (90/10), then (80/20) and (70/30); GC (60 °C, 8 °C/min): $t_R$ 9.53 min; ATR-FTIR (neat, cm$^{-1}$) *v* 2958, 2855, 1609, 1461, 1427, 1328, 1251, 1125, 1021, 831, 559; $^{1}$H NMR (400 MHz) *δ* 8.04 (d, *J* = 13.7 Hz, 1H), 7.97 (dd, *J* = 13.0, 7.7 Hz, 1H), 7.80 (d, *J* = 7.8 Hz, 1H), 7.60 (td, *J* = 7.7, 3.9 Hz, 1H), 3.77 (d, *J* = 11.1 Hz, 6H); $^{13}$C NMR (100 MHz) *δ* 135.1 (d, *J* = 9.6 Hz), 131.2 (qd, *J* = 33.0, 15.8 Hz), 129.6, 129.3 (q, *J* = 3.6 Hz), 129.2 (d, *J* = 15.0 Hz), 128.7 (dq, *J* = 11.3, 3.8 Hz), 127.7, 123.6 (qd, *J* = 272.7, 2.7 Hz), 52.9 (d, *J* = 5.7 Hz); $^{19}$F NMR ($CDCl_3$, 376 MHz): δ -63.93; $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 19.04; MS, *m/z* (relative intensity) 255 (5), 254 ([M]$^{+}$, 20), 253 (100), 235 (16), 234 (9), 224 (11), 223 (16), 221 (6), 214 (6), 209 (21), 172 (5), 160 (6), 159 (23), 146 (9), 145 (12), 140 (15); 127 (7), 125 (6), 95 (5), 79 (11).

**Dimethyl (2-(trifluoromethyl)phenyl)phosphonate** (**3p**). Pale yellow oil. Yield: 15% (150 mg). FC:



elution with a gradient of petroleum ether/acetone (90/10), then (80/20) and (70/30); GC (60 °C, 8 °C/min): $t_R$ 10.16 min; ATR-FTIR (neat, cm$^{-1}$) *v* 2958, 2855, 1443, 1311, 1251, 1126, 1021, 771, 561; $^{1}$H NMR (400 MHz) *δ* 8.24-8.16 (m, 1H), 7.84-7.77 (m, 1H), 7.71-7.59 (m, 2H), 3.79 (d, *J* = 11.4 Hz, 6H); $^{13}$C NMR (100 MHz) *δ* 136.3 (d, *J* = 7.4 Hz), 132.6 (d, *J* = 2.8 Hz), 131.5 (d, *J* = 13.0 Hz), 127.4 (dq, *J* = 11.6, 5.7 Hz), 126.7, 124.8, 123.2 (qd, *J* = 273.9, 4.8 Hz), 53.1 (d, *J* = 5.9 Hz); $^{19}$F NMR ($CDCl_3$, 376 MHz): δ -59.43 (d, *J* = 1.4 Hz).;$^{31}$P NMR ($CDCl_3$, 162 MHz): δ 17.55; MS, *m/z* (relative intensity) 254 ([M]$^{+}$, 14), 253 (100), 235 (7), 233 (25), 204 (7), 203 (36), 189 (21), 185 (62), 175 (9), 161 (11), 159 (13), 155 (7), 153 (8), 146 (12), 145 (9), 126 (7), 125 (10), 92 (9). HRMS (ESI$^{+}$) *m/z* calcd for $C_9H_{11}F_3O_3P$ [M + H], 255.039242; found, 255.039130.

**Ethyl 4-(dimethoxyphosphoryl)benzoate** (**3q**).[1] Pale yellow oil. Yield: 49% (510 mg). FC: elution with

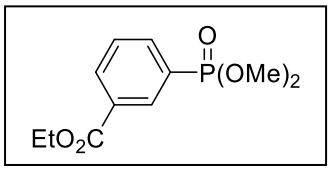


a gradient of petroleum ether/acetone (90/10), then (80/20) and (70/30); GC (60 °C, 8 °C/min): $t_R$ 16.99 min; ATR-FTIR (neat, cm$^{-1}$) *v* 2956, 2852, 1718, 1462, 1397, 1369, 1270, 1102, 1016, 762, 584; $^{1}$H NMR (400 MHz) *δ* 8.11 (dd, *J* = 8.5, 3.9 Hz, 2H), 7.85 (dd, *J* = 13.0, 8.5 Hz, 2H), 4.38 (q, *J* = 7.1 Hz, 2H), 3.75 (d, *J* = 11.1 Hz, 6H), 1.38 (t, *J* = 7.1 Hz, 3H); $^{13}$C NMR (100 MHz) *δ*

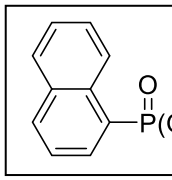
[7] X.-Y. Jiao, W. G. Bentrude, *J. Org. Chem.* 2003, **68**, 3303.
[8] W. G. Bentrude, J.-J. L. Fu, P. E. Rogers, *J. Am. Chem. Soc.*, 1972, **95**, 3625.

165.6, 134.2 (d, *J* = 3.2 Hz), 131.9 (d, *J* = 10.1 Hz), 131.6 (d, *J* = 187.4 Hz), 129.4 (d, *J* = 15.1 Hz), 61.5, 52.8 (d, *J* = 5.6 Hz), 14.2; $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 19.89; MS, *m/z* (relative intensity) 258 ($[M]^{\cdot+}$, 13), 257 (26), 230 (10), 229 (36), 214 (24), 213 (100), 199 (6), 186 (7), 185 (21), 164 (5), 163 (6), 155 (6), 136 (26), 135 (8), 91 (12), 79 (6), 77 (7).

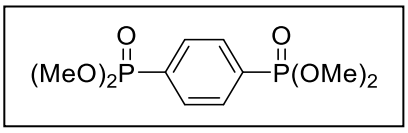
**Ethyl 3-(dimethoxyphosphoryl)benzoate** (**3r**). Pale brown oil. Yield: 91% (940 mg). FC: elution with a gradient of petroleum ether/acetone (90/10), then (80/20) and (70/30); GC (60 °C, 8 °C/min): $t_R$ 16.97 min; ATR-FTIR (neat, $cm^{-1}$) *v* 2957, 2853, 1719, 1250, 1138, 1018, 828, 750, 560; $^1$H NMR (400 MHz) *δ* 8.45 (d, *J* = 13.9 Hz, 1H), 8.24 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.98 (dd, *J* = 12.9, 7.6 Hz, 1H), 7.59-7.54 (m, 1H), 4.40 (q, *J* = 7.1 Hz, 2H), 3.78 (d, *J* = 11.1 Hz, 7H), 1.40 (t, *J* = 7.1 Hz, 5H; $^{13}$C NMR (100 MHz) *δ* 165.6 (d, *J* = 2.3 Hz), 136.0 (d, *J* = 10.1 Hz), 133.6 (d, *J* = 2.9 Hz), 132.9 (d, *J* = 10.9 Hz), 131.0 (d, *J* = 15.1 Hz), 128.8 (d, *J* = 14.9 Hz), 127.8 (d, *J* = 190.1 Hz), 61.5, 52.9 (d, *J* = 5.6 Hz), 14.3; $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 20.11; MS, *m/z* (relative intensity) 258 ($[M]^{\cdot+}$, 17), 257 (38), 240 (13), 231 (18), 230 (9), 229 (72), 214 (18), 213 (100), 199 (13), 186 (19), 185 (54), 154 (5), 148 (7), 136 (6), 135 (5), 131 (6), 91 (7), 79 (9), 77 (11). HRMS ($ESI^+$) *m/z* calcd for $C_{11}H_{16}O_5P$ [M + H], 259.072987; found, 259.072859.



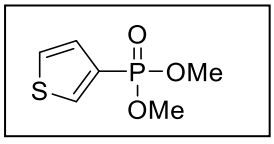

**Dimethyl naphthalen-1-ylphosphonate** (**3u**).[9] Pale yellow oil. Yield: 48% (450 mg). FC: elution with a gradient of petroleum ether/acetone (90/10), then (80/20); GC (60 °C, 8 °C/min): $t_R$ 17.27 min; ATR-FTIR (neat, $cm^{-1}$) *v* 2952, 2849, 1508, 1459, 1244, 1016, 824, 804, 774, 566; $^1$H NMR (400 MHz) *δ* 8.46 (d, *J* = 8.5 Hz, 1H), 8.23 (ddd, *J* = 16.4, 7.1, 1.3 Hz, 1H), 8.04 (d, *J* = 8.2 Hz, 1H), 7.89 (d, *J* = 8.1 Hz, 1H), 7.61 (ddd, *J* = 8.5, 6.9, 1.4 Hz, 1H), 7.57-7.49 (m, 2H), 3.78 (d, *J* = 11.3 Hz, 6H); $^{13}$C NMR (100 MHz) *δ* 134.9 (d, *J* = 9.1 Hz), 133.9 (d, *J* = 3.4 Hz), 133.6 (d, *J* = 12.8 Hz), 132.7 (d, *J* = 11.0 Hz), 128.8 (d, *J* = 1.9 Hz), 127.7, 126.5, 126.5, 124.5 (d, *J* = 16.7 Hz), 123.4 (d, *J* = 183.7 Hz), 52.7 (d, *J* = 5.4 Hz); $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 22.25; MS, *m/z* (relative intensity) 237 (13), 236 ($[M]^{\cdot+}$, 100), 235 (56), 221 (23), 218 (13), 205 (7), 204 (14), 203 (34), 189 (5), 186 (5), 173 (9), 155 (14), 142 (11), 141 (67), 140 (7), 128 (26), 127 (16), 126 (10), 115 (17).




**Tetramethyl 1,4-phenylenebis(phosphonate)** (**3v**).[10] White solid. mp 102-103 °C; Yield: 40% (470 mg). FC: elution with a gradient of petroleum dichloromethane/acetone (70/30); ATR-FTIR (neat, $cm^{-1}$) *v* 2999, 2955, 2853, 1457, 1244, 1138, 10010, 819, 773, 599, 547; $^1$H NMR (400 MHz) *δ* 7.92-7.88 (m , 4H), 3.78 (d, *J* = 11.1 Hz, 12H); $^{13}$C NMR (100 MHz) *δ* 132.09–131.64 (m), 131.78 (dd, *J* = 187.5, 3.1 Hz), 53.0–52.9 (m); $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 19.48.




**Dimethyl thiophen-3-ylphosphonate** (**3w**). Pale yellow oil. Yield: 68% (520 mg). FC: elution with a gradient of petroleum ether/acetone (90/10), then (80/20) and (70/30); GC (60 °C, 8 °C/min): $t_R$ 10.24 min; ATR-FTIR (neat, $cm^{-1}$) *v* 2953, 2851, 1459, 1394, 1244, 1016, 821, 759, 621, 565; $^1$H NMR (400 MHz) *δ* 8.96 (br s, 1H), 8.78 (br s, 1H), 8.08 (dd, *J* = 13.4, 7.8 Hz, 1H), 7.57-7.35 (m, 1H), 3.79 (d, *J* =

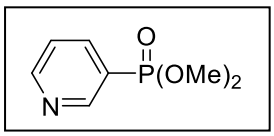



[9] Y.-L. Zhao, G.-J. Wu, Y. Li, L.-X. Gao, F.-S. Han, *Chem. Eur. J.*, 2012, **18**, 9622.

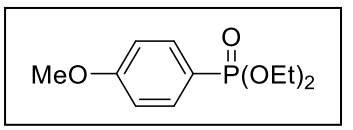
[10] S. S. Iremonger, J. Liang, R. Vaidhyanathan, I. Martens, G. K. H. Shimizu, T. D. Daff, M. Z. Aghaji, S. Yeganegi, T. K. Woo, *J. Am. Chem. Soc.*, 2011**, 133**, 20048.

11.2 Hz, 6H); $^{13}$C NMR (100 MHz) *δ* 135.8 (d, *J* = 18.0 Hz), 129.0 (d, *J* = 16.8 Hz), 128.0 (d, *J* = 198.0 Hz),.127.4 (d, *J* = 19.7 Hz), 52.7 (d, *J* = 5.5 Hz); $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 16.02; MS, *m/z* (relative intensity) 193 (11), 192 ($[M]^{\cdot+}$, 92), 191 12), 177 (72), 163 (8), 162 (57), 161 (36), 159 (30), 147 (79), 119 (20), 111 (13), 110 (23), 99 (13), 98 (22), 97 (100), 84 (33), 81 (9), 79 (11), 63 (12, 58 (17); HRMS ($ESI^+$) *m/z* calcd for $C_6H_{10}O_3PS$ [M + H], 193.008278; found, 193.008128.

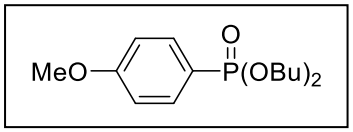
**Dimethyl pyridin-3-ylphosphonate** (**3x**). Pale yellow oil. Yield: 20% (150 mg). FC: elution with a gradient of petroleum ether/acetone (70/30), then (60/40) and (50/50); GC (60 °C, 8 °C/min): $t_R$ 10.74 min; ATR-FTIR (neat, $cm^{-1}$) *v* 2955, 2851, 1629, 1582, 1462, 1408, 1237, 1021, 765, 560, 531; $^1$H NMR (400 MHz) *δ* 8.96 (br s, 1H), 8.78 (br s, 1H), 8.08 (dd, *J* = 13.4, 7.8 Hz, 1H), 7.57-7.35 (m, 1H), 3.79 (d, *J* = 11.2 Hz, 6H); $^{13}$C NMR (100 MHz) *δ* 153.2, 152.3 (d, *J* = 12.1 Hz), 139.6 (d, *J* = 8.3 Hz), 123.7 (d, *J* = 192.1 Hz), 123.5 (d, *J* = 13.1 Hz), 52.9 (d, *J* = 5.7 Hz); $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 18.61; MS, *m/z* (relative intensity) 187 ($[M]^{\cdot+}$, 18), 186 (100), 157 (6), 156 (7), 154 (7), 142 (14), 106 (5), 93 (34), 92 (9), 79 (31), 78 (13), 65 (6), 52 (9), 51 (16); HRMS ($ESI^+$) *m/z* calcd for $C_7H_{11}NO_3P$ [M + H], 188.047106; found, 188.046901.



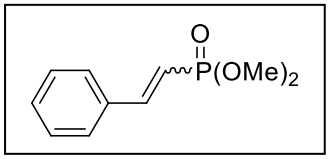

**Diethyl (4-methoxyphenyl)phosphonate** (**3'a**).[3] Pale brown oil. Yield: 79% (770 mg). FC: elution with a gradient of petroleum ether/acetone (90/10), then (80/20), and (70/30); GC (60 °C, 8 °C/min): $t_R$ 15.73 min; ATR-FTIR (neat, $cm^{-1}$) *v* 2981, 2853, 1599, 1506, 1239, 1131, 1018, 956, 806, 537; $^1$H NMR (400 MHz) *δ* 7.73 (dd, *J* = 12.7, 8.8 Hz, 2H), 6.95 (dd, *J* = 8.8, 3.3 Hz, 2H), 4.26-3.96 (m, 4H), 3.84 (s, 3H), 1.30 (t, *J* = 7.1 Hz, 6H); $^{13}$C NMR (100 MHz) *δ* 162.9 (d, *J* = 3.4 Hz), 133.8 (d, *J* = 11.3 Hz), 119.5 (d, *J* = 194.9 Hz), 114.0 (d, *J* = 16.0 Hz), 61.9 (d, *J* = 5.3 Hz), 55.3 (s), 16.3 (d, *J* = 6.6 Hz); $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 19.76; MS, *m/z* (relative intensity) 244 ($[M]^{\cdot+}$, 18), 217 (5), 215 (49), 201 (5), 189 (9), 188 (100), 172 (17), 171 (23), 170 (16), 135 (11), 124 (8), 123 (6), 109 (5), 108 (22), 94 (11), 78 (6), 78 (12), 77 (6).




**Dibutyl (4-methoxyphenyl)phosphonate** (**3"a**).[4] Pale brown oil. Yield: 78% (930 mg). FC: elution with a gradient of petroleum ether/acetone (90/10), then (80/20); GC (60 °C, 8 °C/min): $t_R$ 20.12 min; ATR-FTIR (neat, $cm^{-1}$) *v* 2956, 2874, 1600, 1244, 1131, 1020, 973, 830, 810, 807, 541; $^1$H NMR (400 MHz) *δ* 7.73 (dd, *J* = 12.6, 8.7 Hz, 2H), 6.95 (dd, *J* = 8.5, 3.2 Hz, 2H), 4.13-3.90 (m, 4H), 3.84 (s, 3H), 1.73-1.56 (m, 4H), 1.42-1.33 (m, 4H), 0.89 (t, *J* = 7.4 Hz, 6H); $^{13}$C NMR (100 MHz) *δ* 162.8 (d, *J* = 3.3 Hz), 133.8 (d, *J* = 11.2 Hz), 119.5 (d, *J* = 195.5 Hz), 114.0 (d, *J* = 16.0 Hz), 65.6 (d, *J* = 5.5 Hz), 55.3 (s), 32.5 (d, *J* = 6.6 Hz), 18.8 (s), 13.61; $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 19.73; MS, *m/z* (relative intensity) 300 ($[M]^{\cdot+}$, 3), 245 (24), 244 (11), 190 (8), 189 (100), 188 (48), 171 (28), 170 (10), 162 (14), 147 (5), 109 (10), 108 (9).



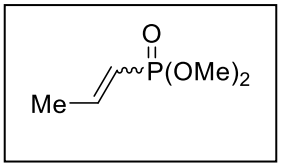

**Dimethyl (2-phenylethenyl)phosphonate** (**5a**).[11] *E*/*Z*: 92/8 diastereoisomers mixture. Pale yellow oil. Yield: 77% (650 mg). FC: elution with a gradient of petroleum ether/acetone (90/10), then (80/20); GC (60 °C, 8 °C/min): $t_R$ 14.91 min; ATR-FTIR (neat, $cm^{-1}$) *v* 2952, 2850, 1616, 1449, 1246, 1020, 831, 724; $^1$H NMR (400 MHz) *δ* 7.66-7.46 (m, 3 H, *E* isomer), 7.44-7.33 (m, 3, *E*




[11] J.-W. Yuan, L.-R. Yang, P. Mao and L.-B. Qu, *RSC Adv.*, 2016, **6**, 87058.

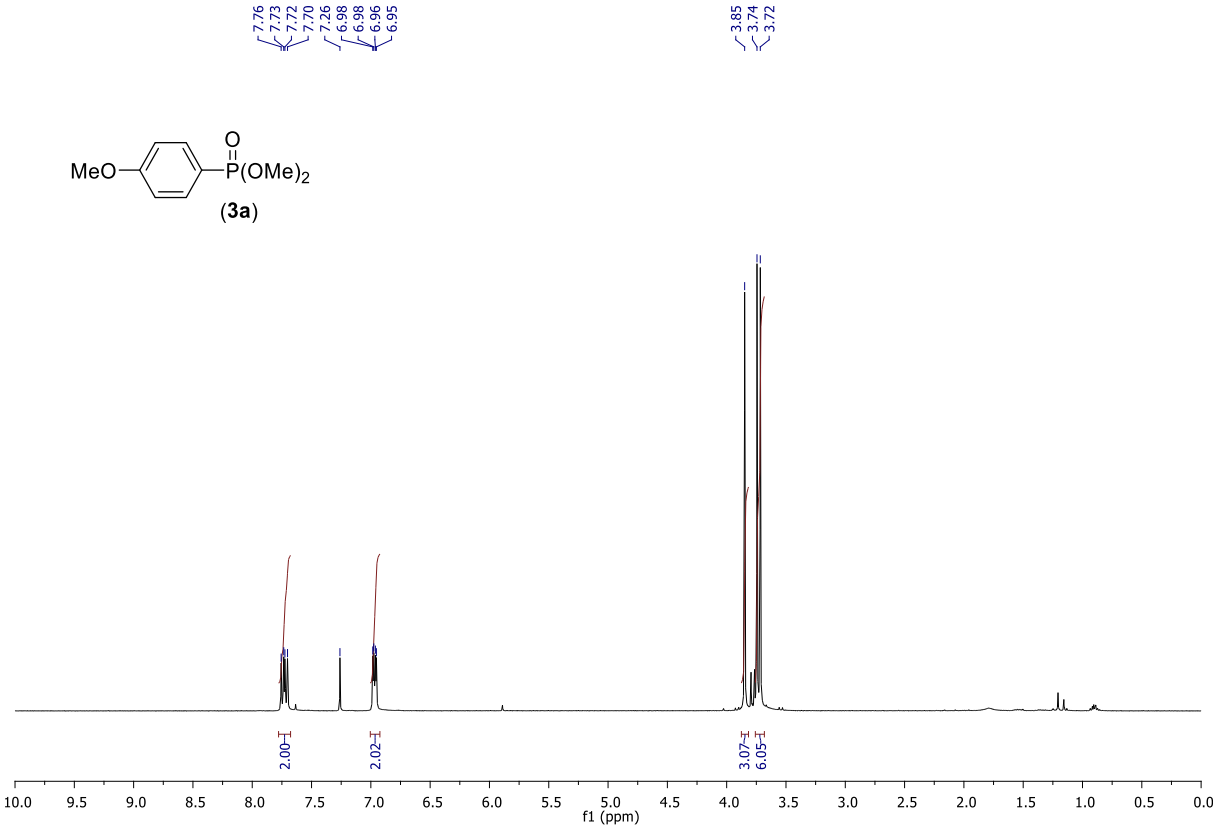
isomer), 7.31-7.27 (m, 4H, *Z* isomer), 7.22-7.18 (m, 3H, *Z* isomer), 6.21 (t, *J* = 17.7 Hz, 1H, *E* isomer), 3.77 (d, *J* = 11.1 Hz, 6H, *E* isomer), 3.72 (d, *J* = 10.8 Hz, 3H, *Z* isomer); $^{13}$C NMR (100 MHz), *E* isomer: *δ* 149.7 (d, *J* = 6.7 Hz), 134.7 (d, *J* = 23.4 Hz), 130.4, 128.9, 127.8, 112.4 (d, *J* = 192.2 Hz), 52.5 (d, *J* = 5.5 Hz); $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 33.45 *Z* isomer), 22.44 (*E* isomer); MS, *m/z* (relative intensity) 213 (6), 212 ($[M]^{\cdot+}$, 42), 211 (17), 181 (9), 179 (5), 149 (23), 121 (5), 118 (11), 117 (100), 116 (26), 115 (43), 110 (5), 103 (6), 102 (11), 91 (9), 80 (10), 79 (13), 77 (11).

**Dimethyl (1-propenyl)phosphonate** (**5b**).[12] *E*/*Z*: 90/10 diastereoisomers mixture. Colourless oil. Yield: 58% (350 mg). FC: elution with a gradient of petroleum ether/acetone (90/10), then (80/20) and (70/30); GC (60 °C, 8 °C/min): $t_R$ 5.14 min; ATR-FTIR (neat, $cm^{-1}$) *ν* 2954, 2852, 1634, 1444, 1235, 1021, 822; $^1$H NMR (400 MHz) *δ* 6.87-6.72 (m, 1H, *E* isomer), 5.82-5.73 (m, 1H, *Z* isomer), 5.67-5.57 (m, 1H, *E* isomer), 5.24-5.19 (m, 1H, *Z* isomer), 3.73 (d, *J* = 10.8 Hz, 6H, *Z* isomer), 3.69 (d, *J* = 11.1 Hz, 6H, *E* isomer), 1.92-1.90 (m, 6H, *E* + *Z* isomers); $^{13}$C NMR (100 MHz), *E* isomer: *δ* 150.2 (d, *J* = 5.0 Hz), 117.0 (d, *J* = 189.1 Hz), 52.3 (d, *J* = 5.6 Hz), 20.2 (d, *J* = 24.1 Hz); $^{31}$P NMR ($CDCl_3$, 162 MHz): δ 29.65 (*Z* isomer), 21.23 (*E* isomer); MS, *m/z* (relative intensity) 150 ($[M]^{\cdot+}$, 5), 149 (12), 135 (100), 123 (7), 119 (9), 110 (12), 109 (14), 105 (13), 103 (21), 96 (62), 95 (7), 93 (14), 87 (22), 80 (21), 79 (54), 78 (5), 66 (19), 65 (16), 63 (9), 55 (5).




[12] W. Rauf and J. M. Brown, *Angew. Chem. Int. Ed.*, 2008, **47**, 4228.

## NMR data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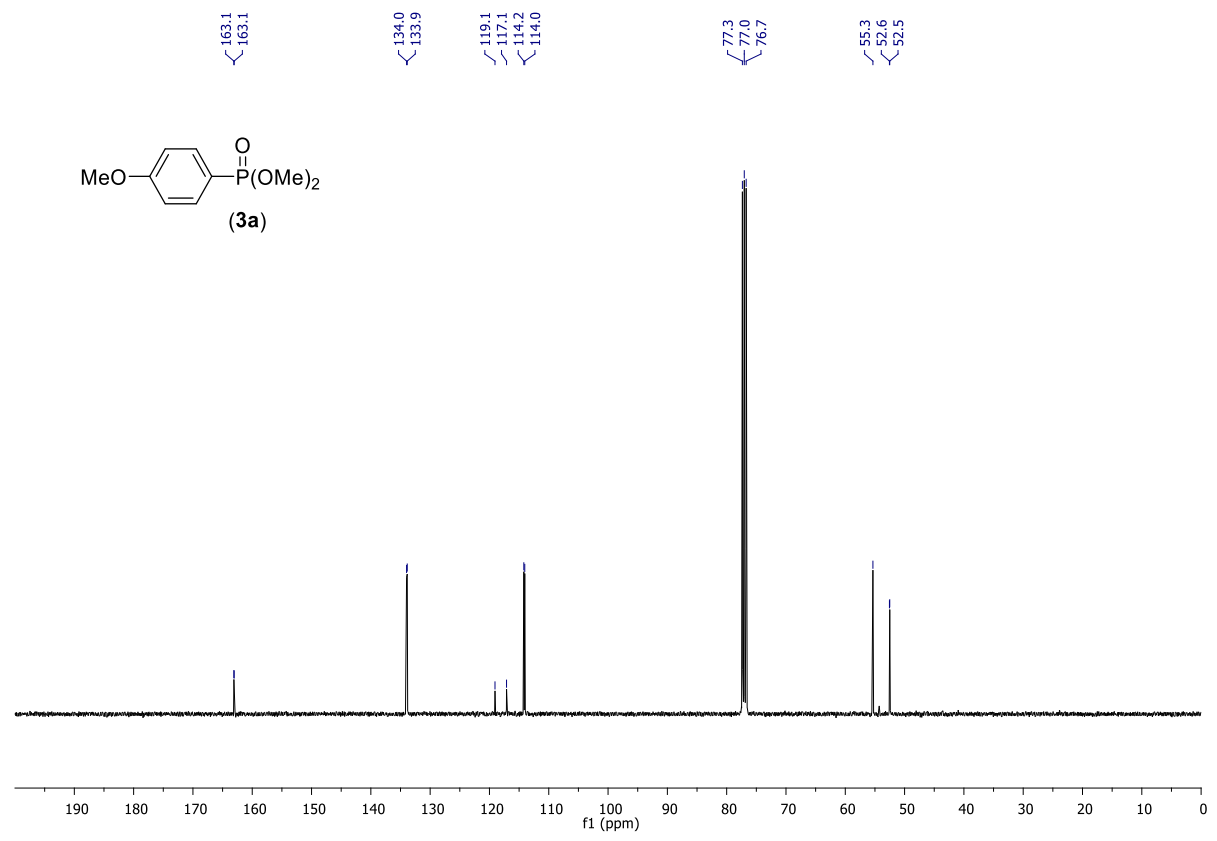
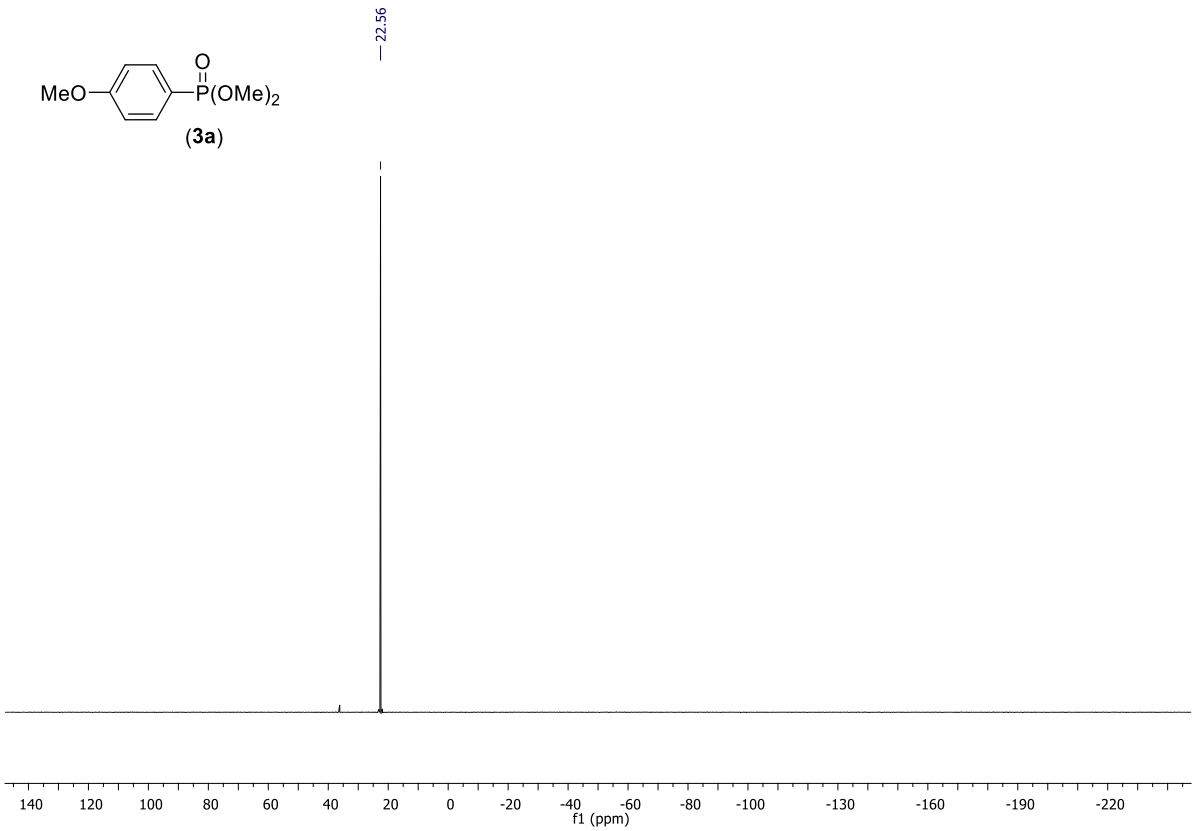
7.73
7.72
7.70
7.26
6.98
6.96
6.95
3.85
3.74
3.72
MeO
O
P(OMe)2
(3a)
2.00
2.02
3.07
6.05
f1 (ppm)


163.1
163.1
134.0
133.9
119.1
117.1
114.2
114.0
77.3
77.0
76.7
55.3
52.6
52.5
MeO
O
P(OMe)2
(3a)
f1 (ppm)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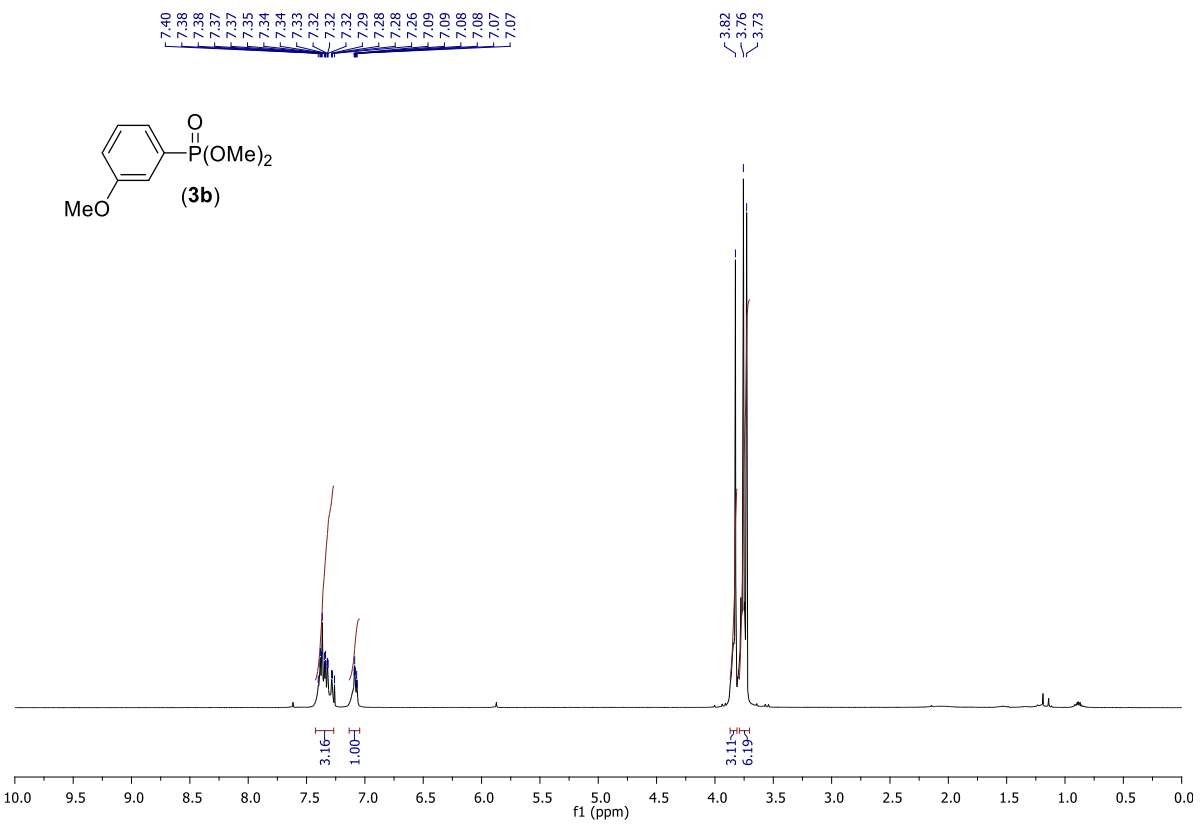
MeO
O
P(OMe)2
(3a)
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)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7.38
7.38
7.37
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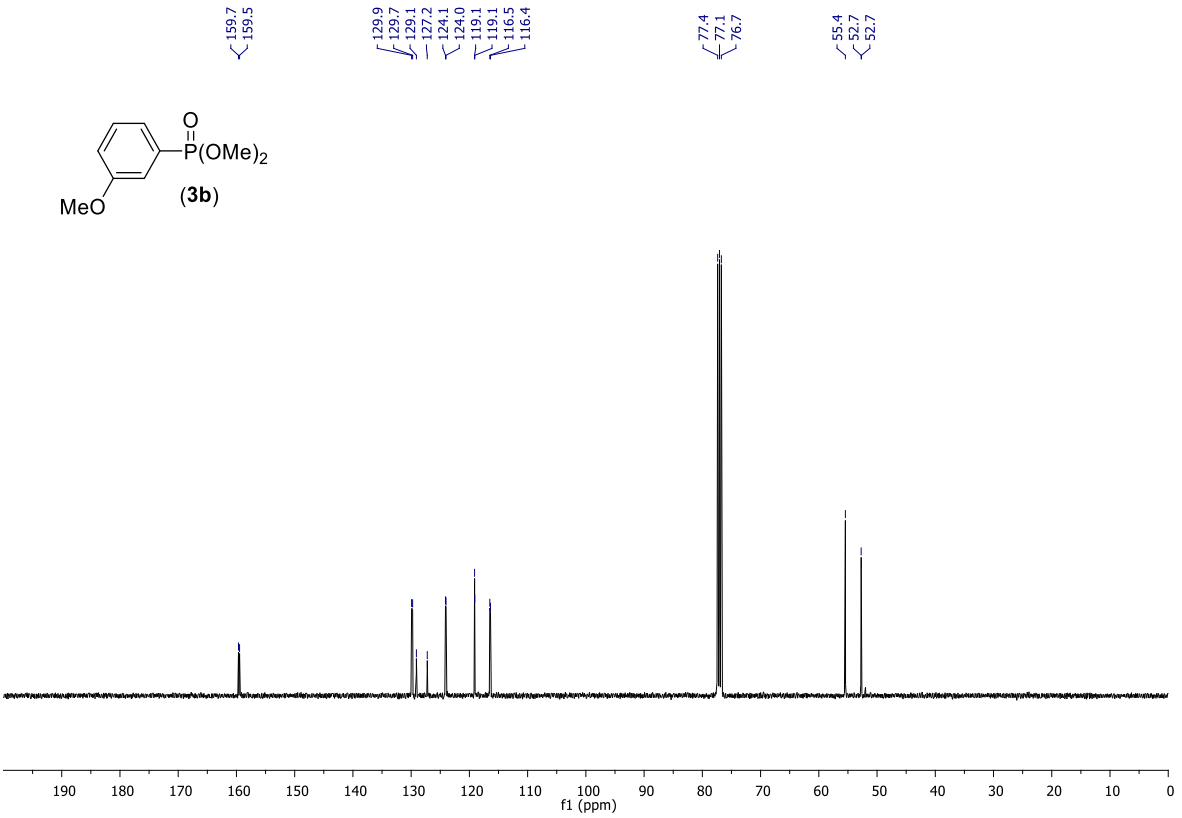
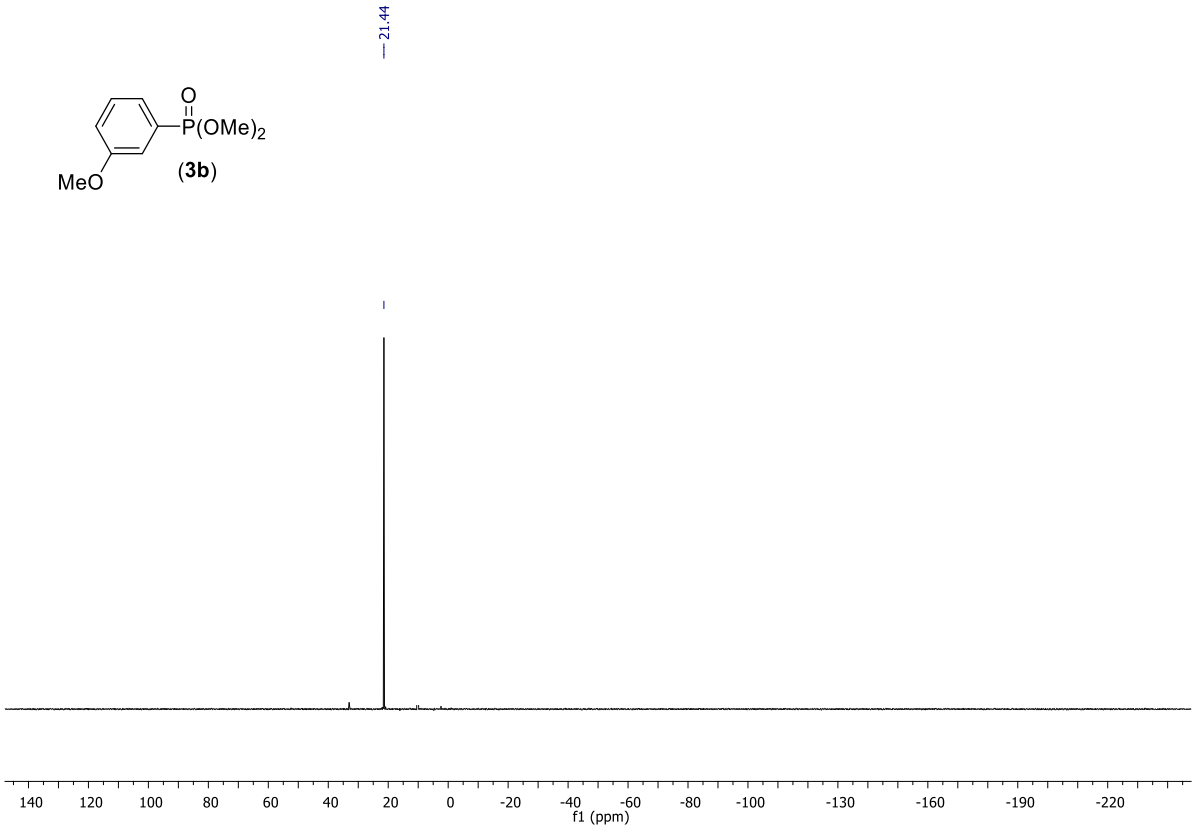
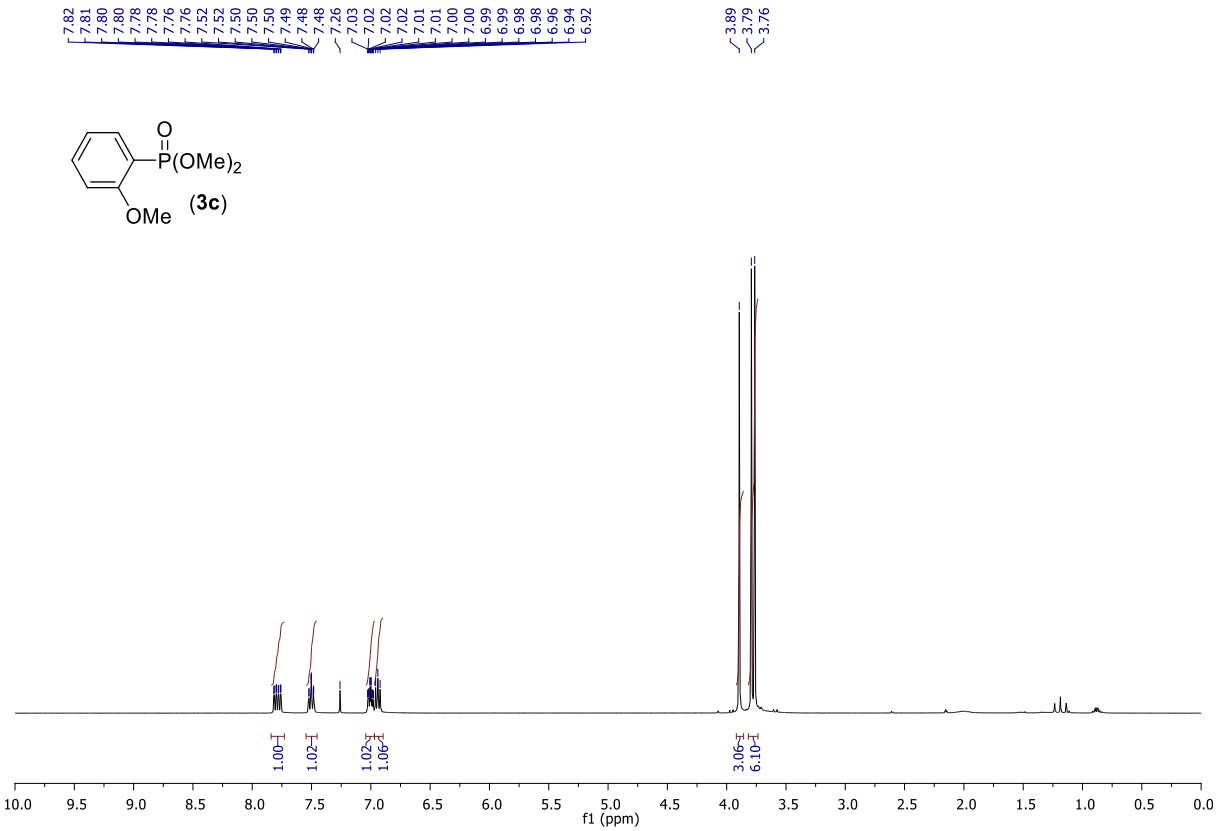
7.35
7.34
7.34
7.33
7.32
7.32
7.32
7.29
7.28
7.28
7.26
7.09
7.09
7.08
7.08
7.07
7.07
3.82
3.76
3.73
O
P(OMe)2
MeO
(3b)
3.16
1.00
3.11
6.19
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


159.7
159.5
129.9
129.7
129.1
127.2
124.1
124.0
119.1
119.1
116.5
116.4
77.4
77.1
76.7
55.4
52.7
52.7
O
P(OMe)2
MeO
(3b)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


21.44
O
P(OMe)2
MeO
(3b)
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)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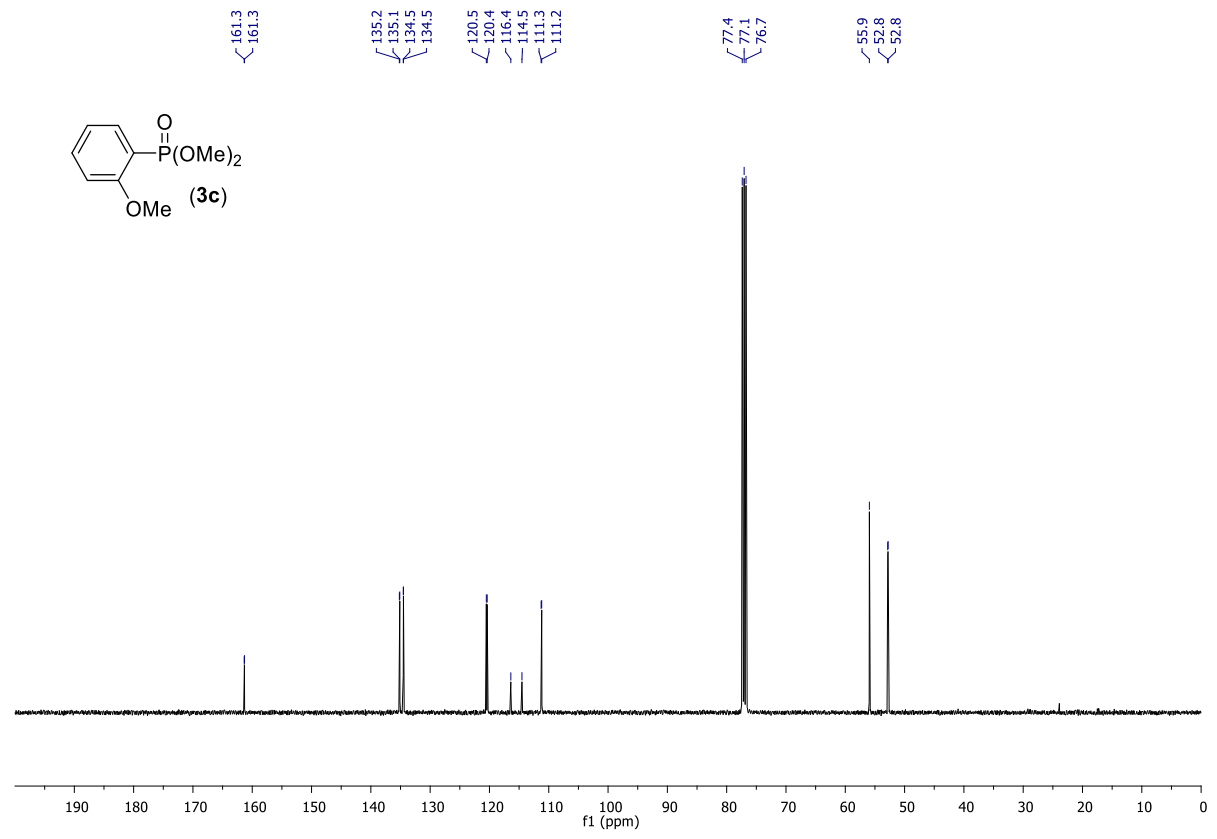
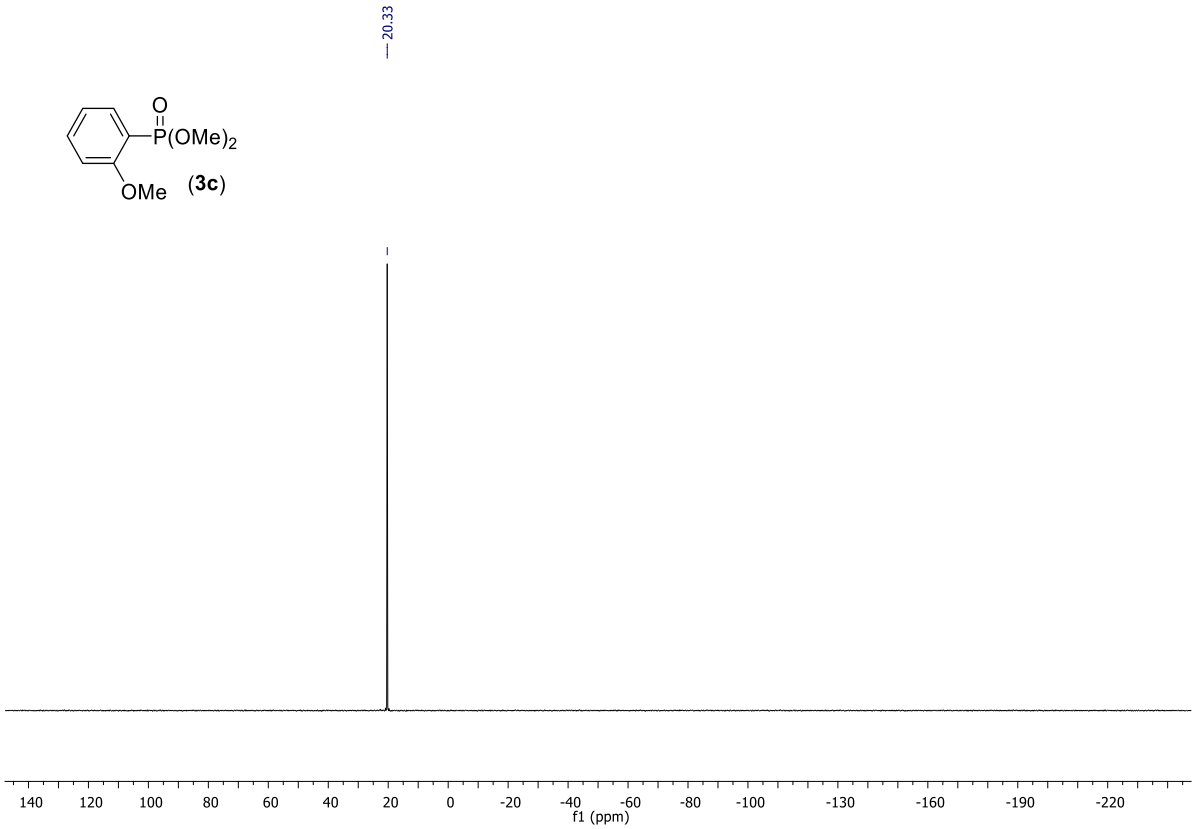
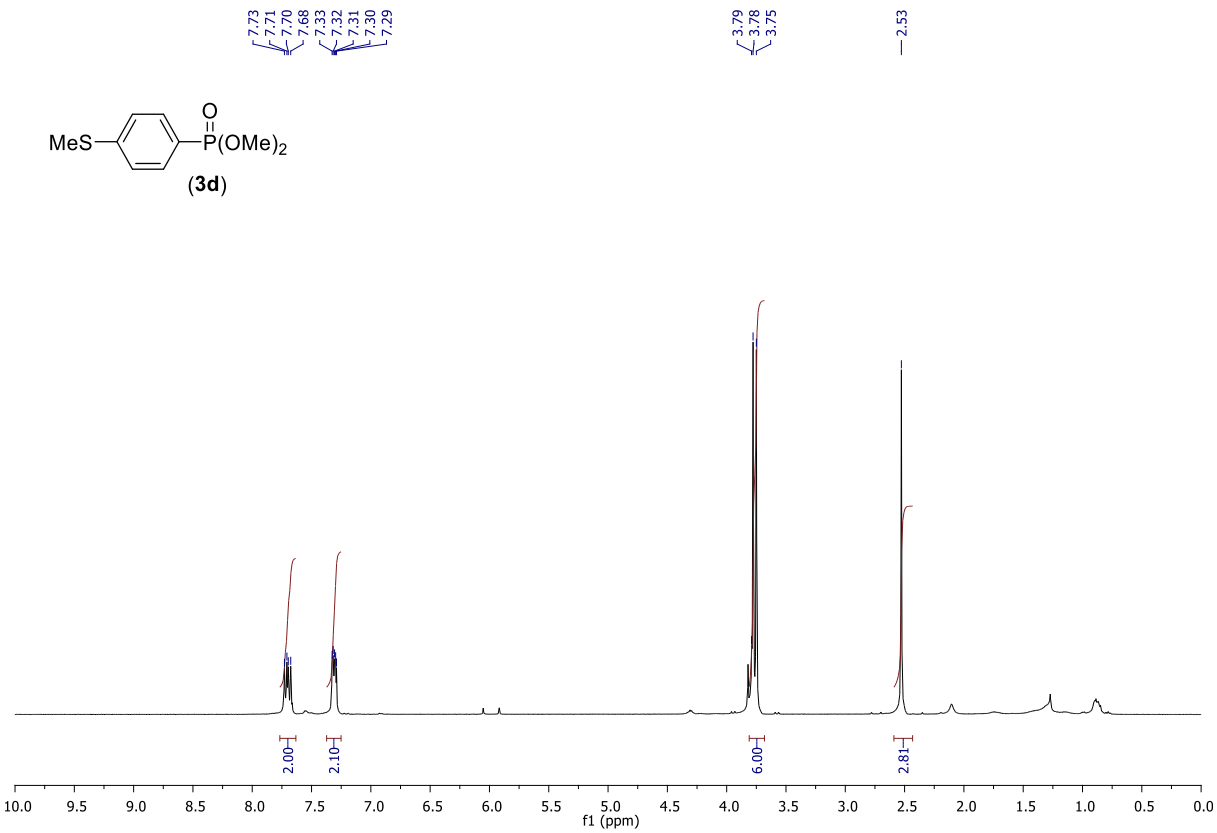
7.81
7.80
7.80
7.78
7.76
7.76
7.52
7.50
7.50
7.50
7.49
7.48
7.48
7.26
7.03
7.02
7.02
7.02
7.01
7.01
7.00
7.00
6.99
6.99
6.98
6.98
6.96
6.94
6.92
3.89
3.79
3.76
O
P(OMe)2
OMe
(3c)
1.00
1.02
1.02
1.06
3.06
6.10
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


161.3
161.3
135.2
135.1
134.5
134.5
120.5
120.4
116.4
114.5
111.3
111.2
77.4
77.1
76.7
55.9
52.8
52.8
O
P(OMe)2
OMe
(3c)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


20.33
O
P(OMe)2
OMe
(3c)
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)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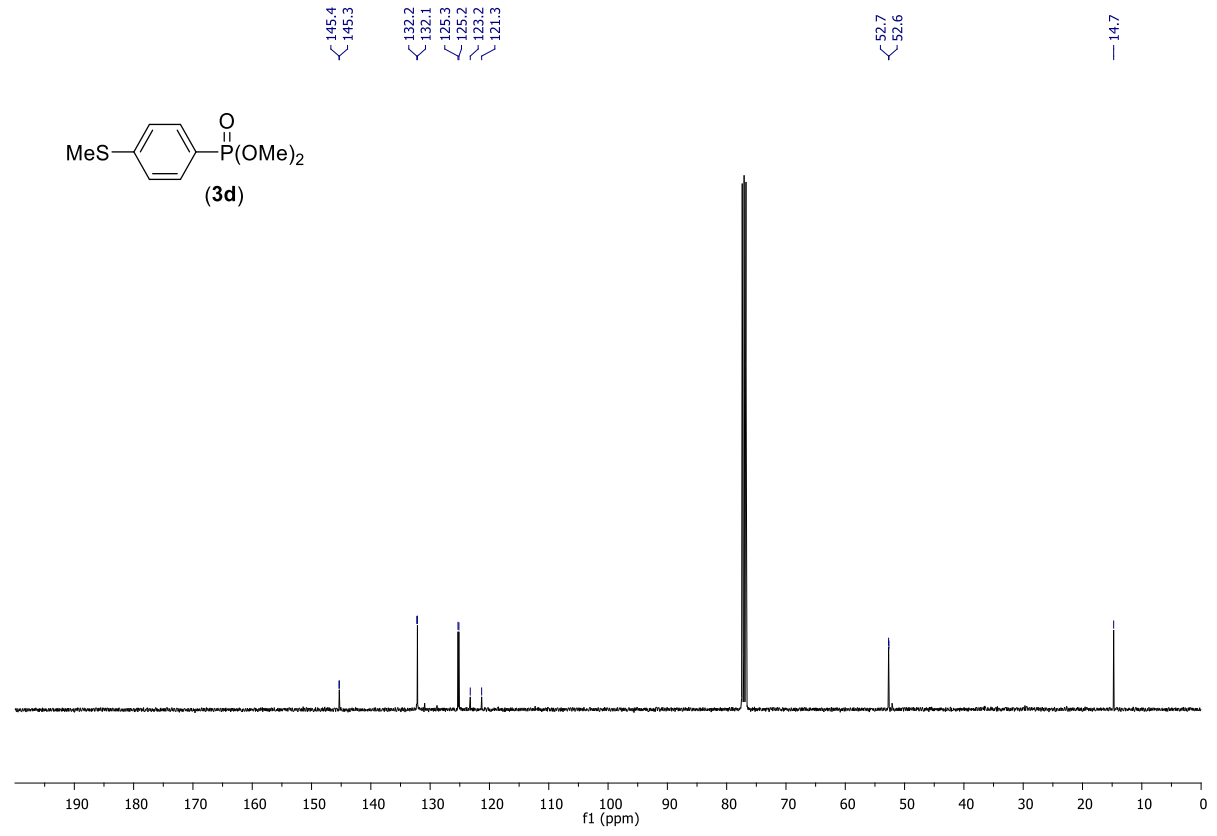
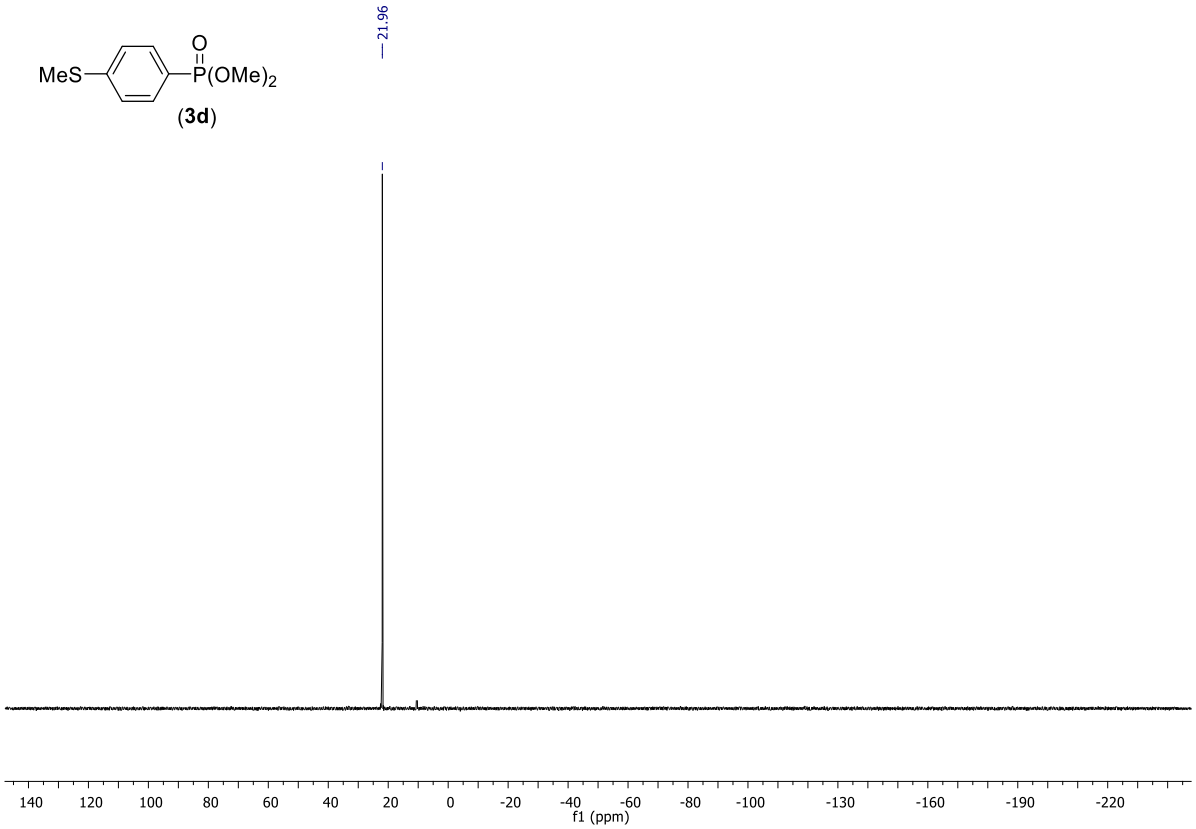
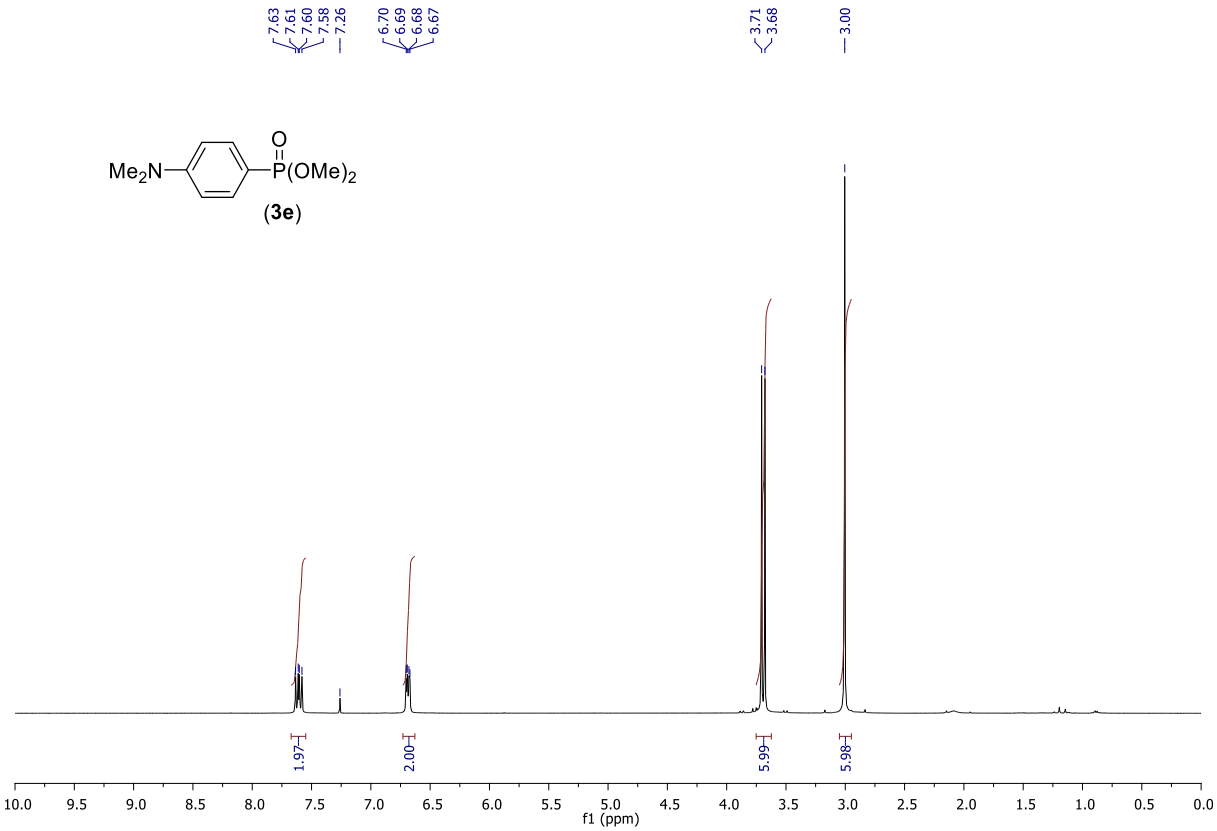
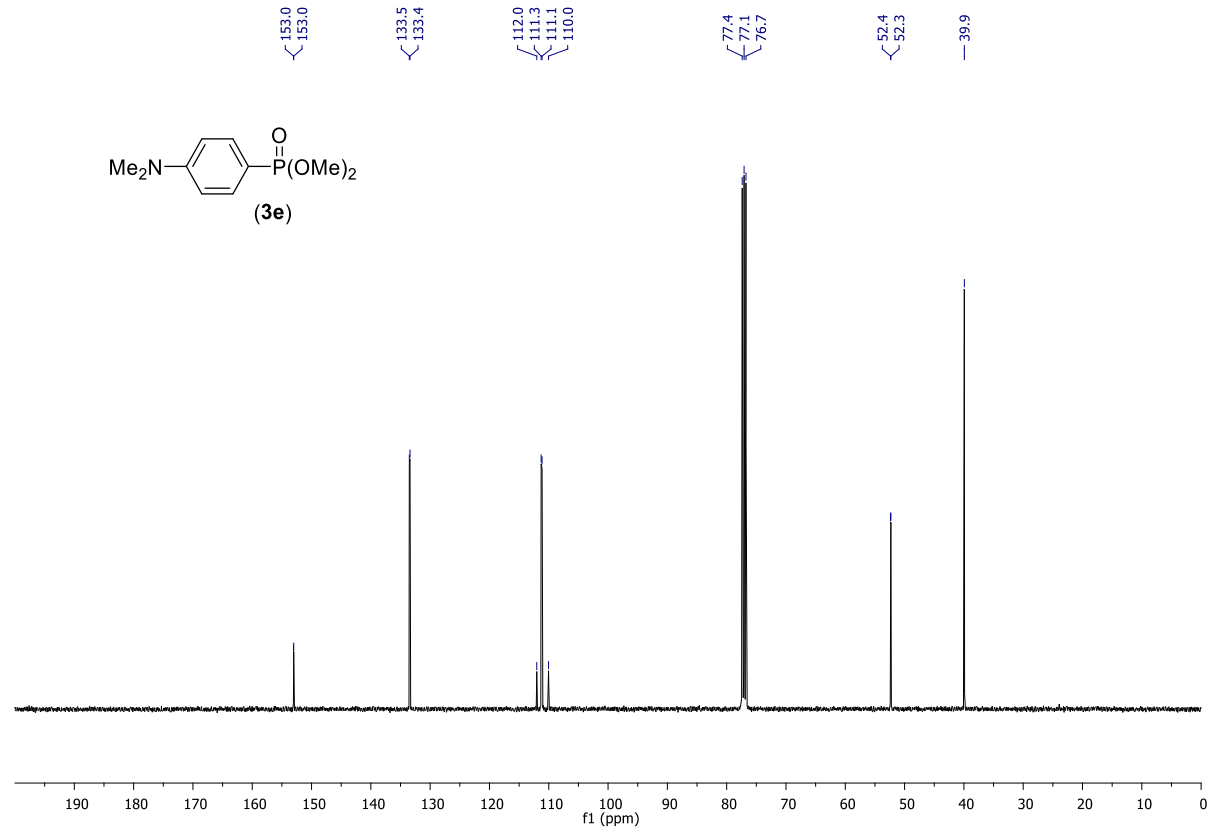
7.71
7.70
7.68
7.33
7.32
7.31
7.30
7.29
3.79
3.78
3.75
2.53
MeS
O
P(OMe)2
(3d)
2.00
2.10
6.00
2.81
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


145.4
145.3
132.2
132.1
125.3
125.2
123.2
121.3
52.7
52.6
14.7
MeS
O
P(OMe)2
(3d)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


MeS
O
P(OMe)2
(3d)
21.96
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)


7.63
7.61
7.60
7.58
7.26
6.70
6.69
6.68
6.67
3.71
3.68
3.00
O
Me2N
P(OMe)2
(3e)
1.97
2.00
5.99
5.98
10.0 9.5 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 0.0
f1 (ppm)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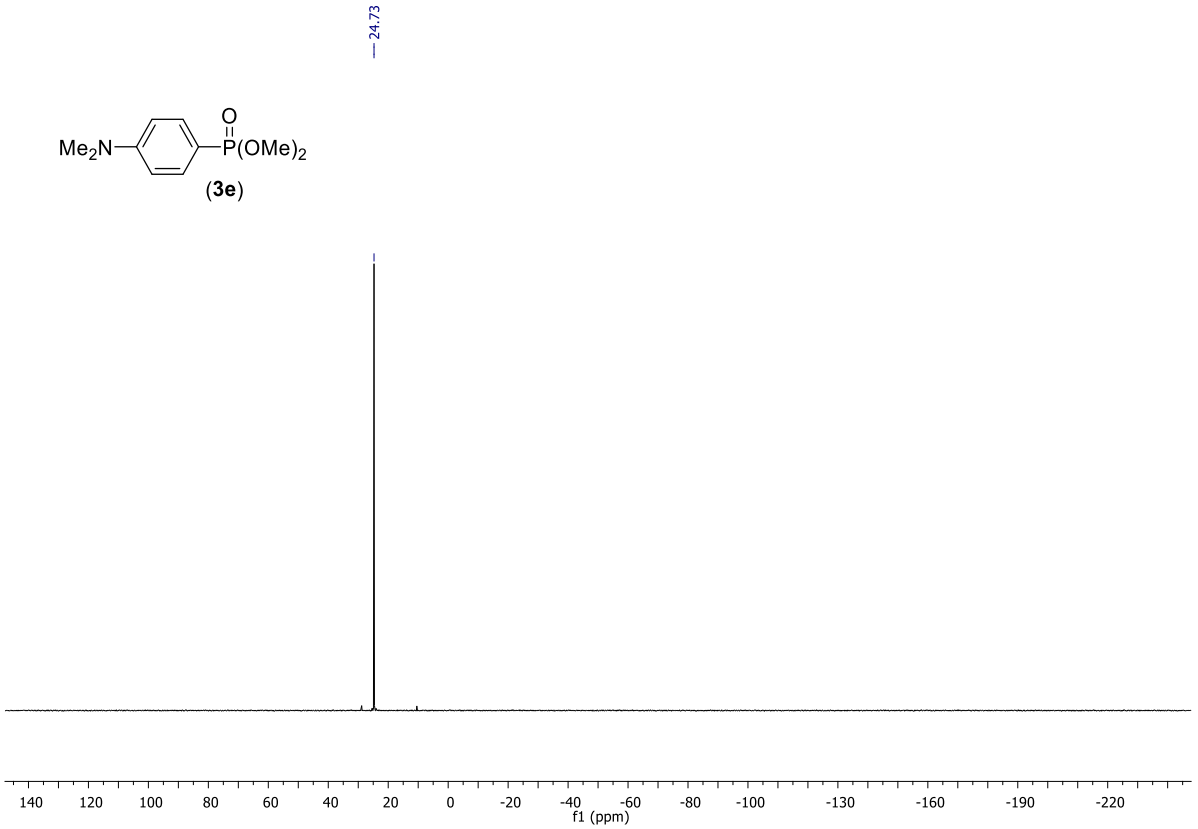
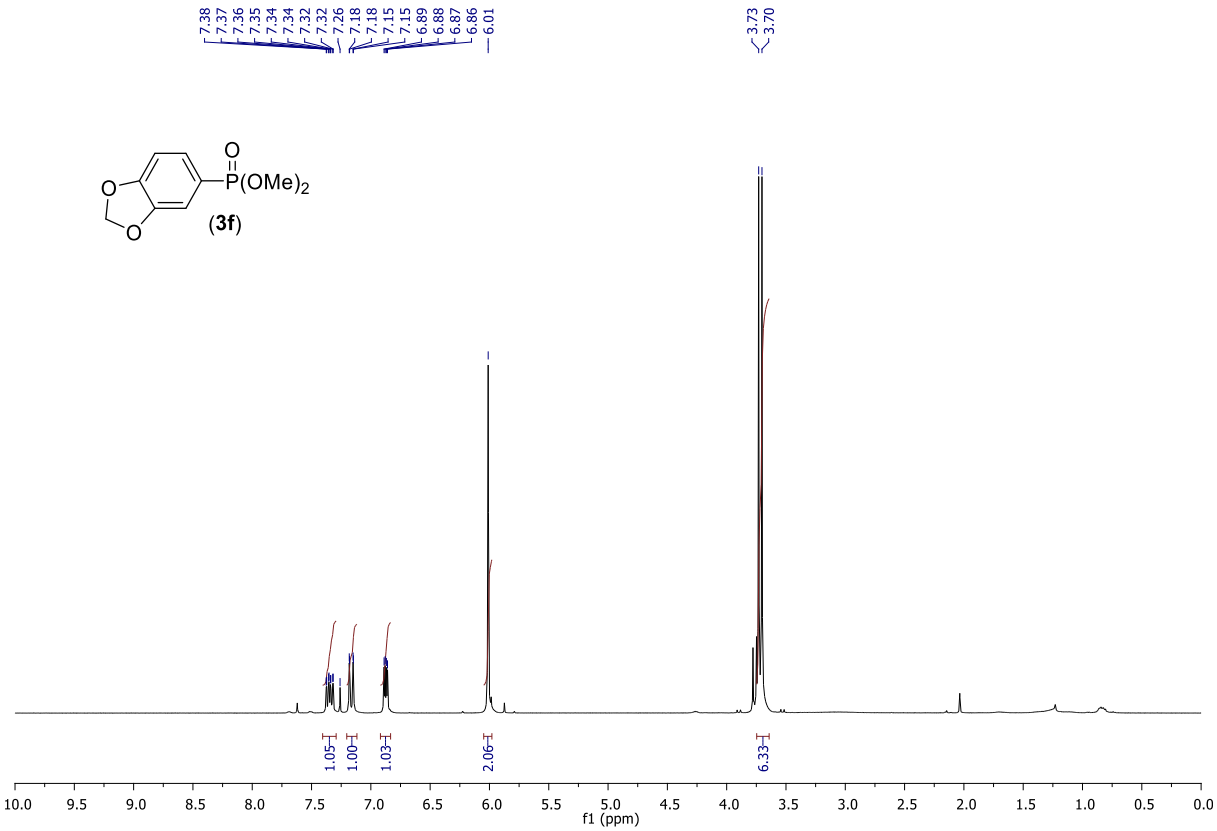
153.0
133.5
133.4
112.0
111.3
111.1
110.0
77.4
77.1
76.7
52.4
52.3
39.9
O
Me2N
P(OMe)2
(3e)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


24.73
Me2N
O
P(OMe)2
(3e)
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)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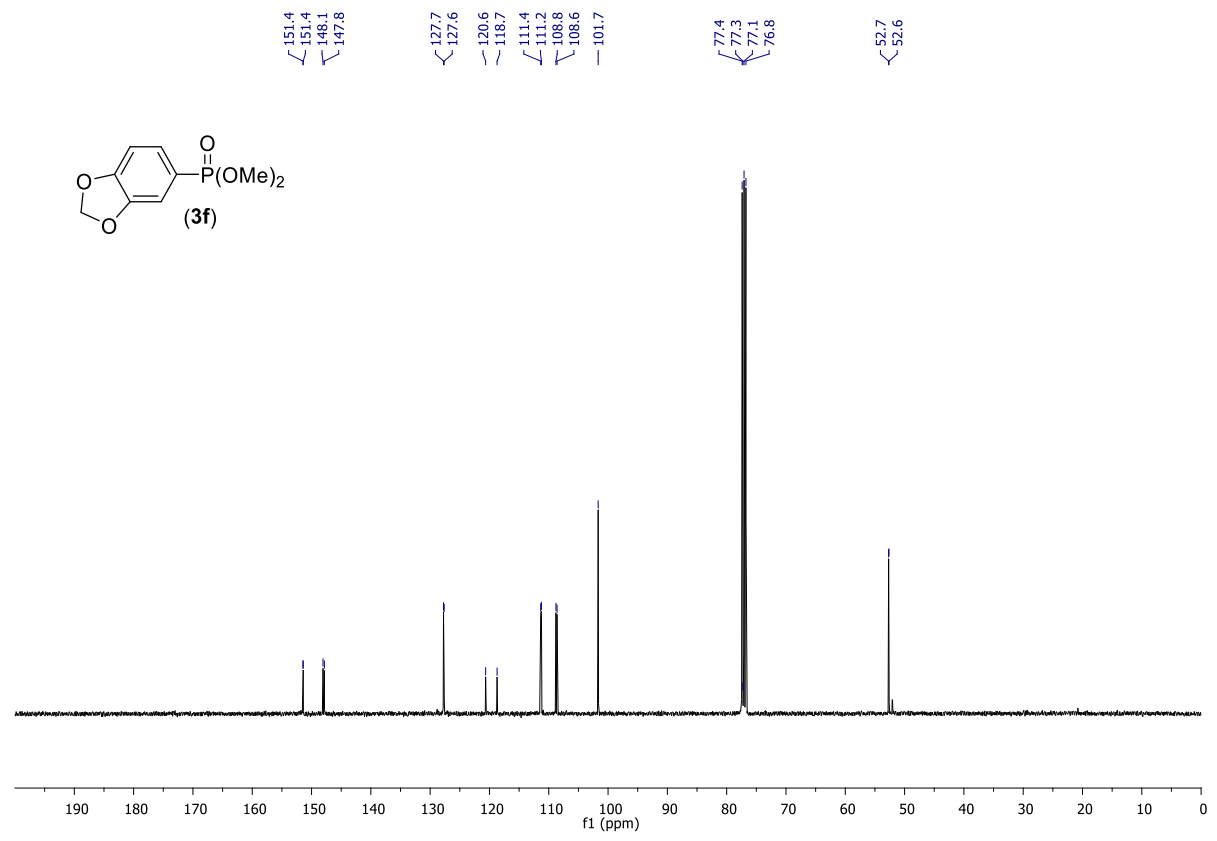
7.34
7.32
7.32
7.26
7.18
7.18
7.15
7.15
6.89
6.88
6.87
6.86
6.01
3.73
3.70
O
P(OMe)2
O
O
(3f)
1.05
1.00
1.03
2.06
6.33
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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127.6
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111.2
108.8
108.6
101.7
77.4
77.3
77.1
76.8
52.7
52.6
O
P(OMe)2
O
O
(3f)
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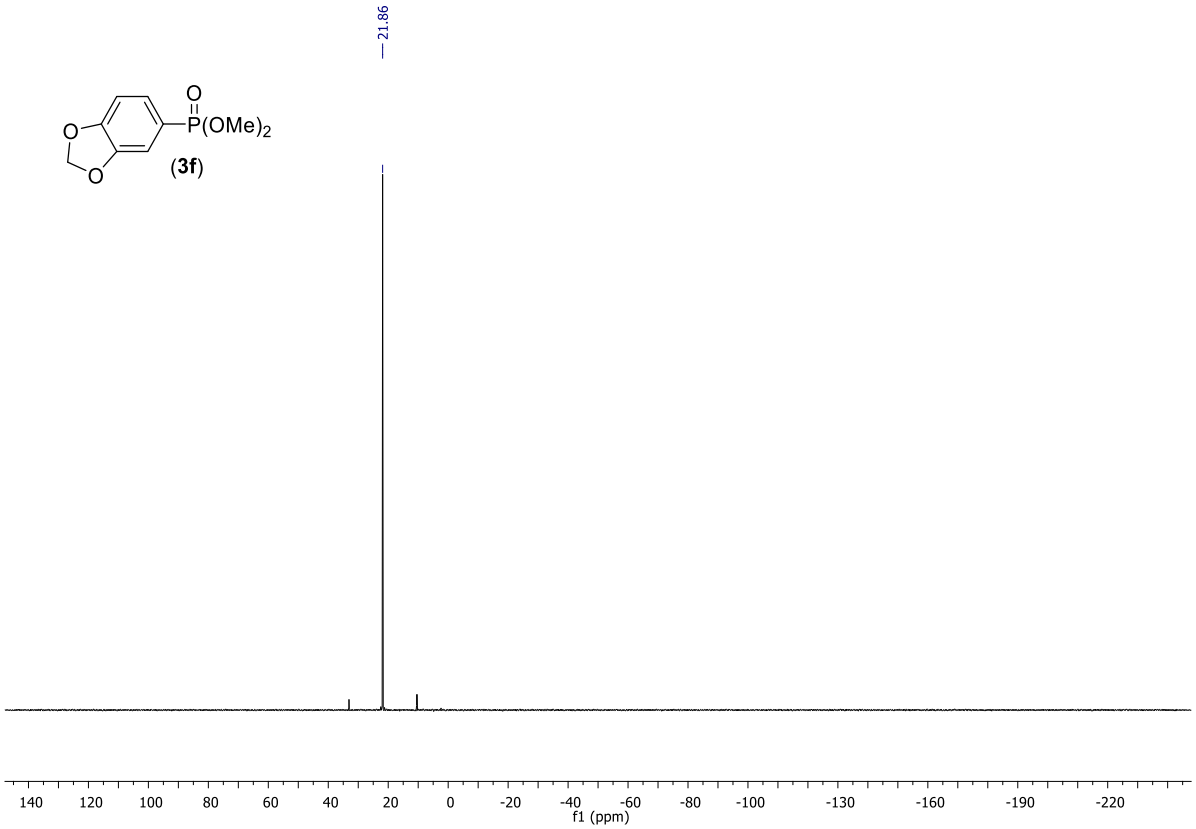
f1 (ppm)


21.86
O
P(OMe)2
O
O
(3f)
140
120
100
80
60
40
20
0
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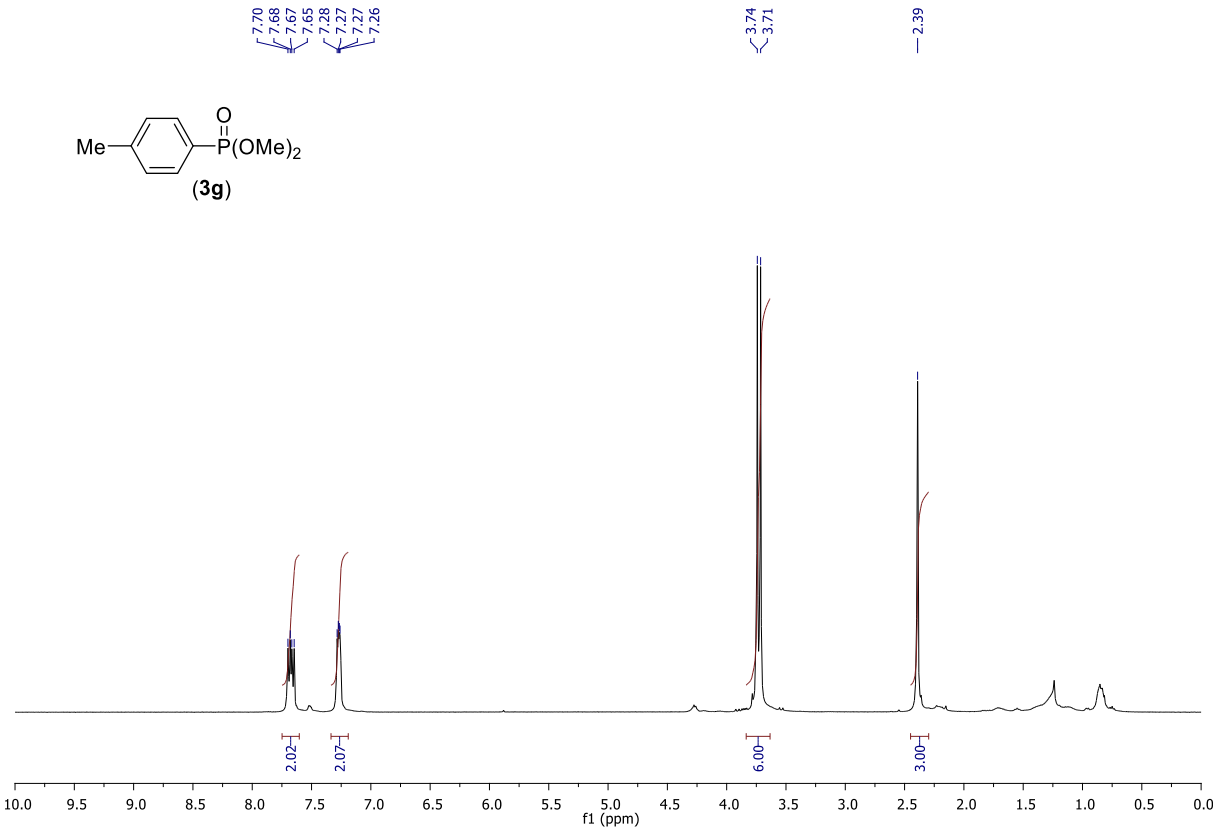
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)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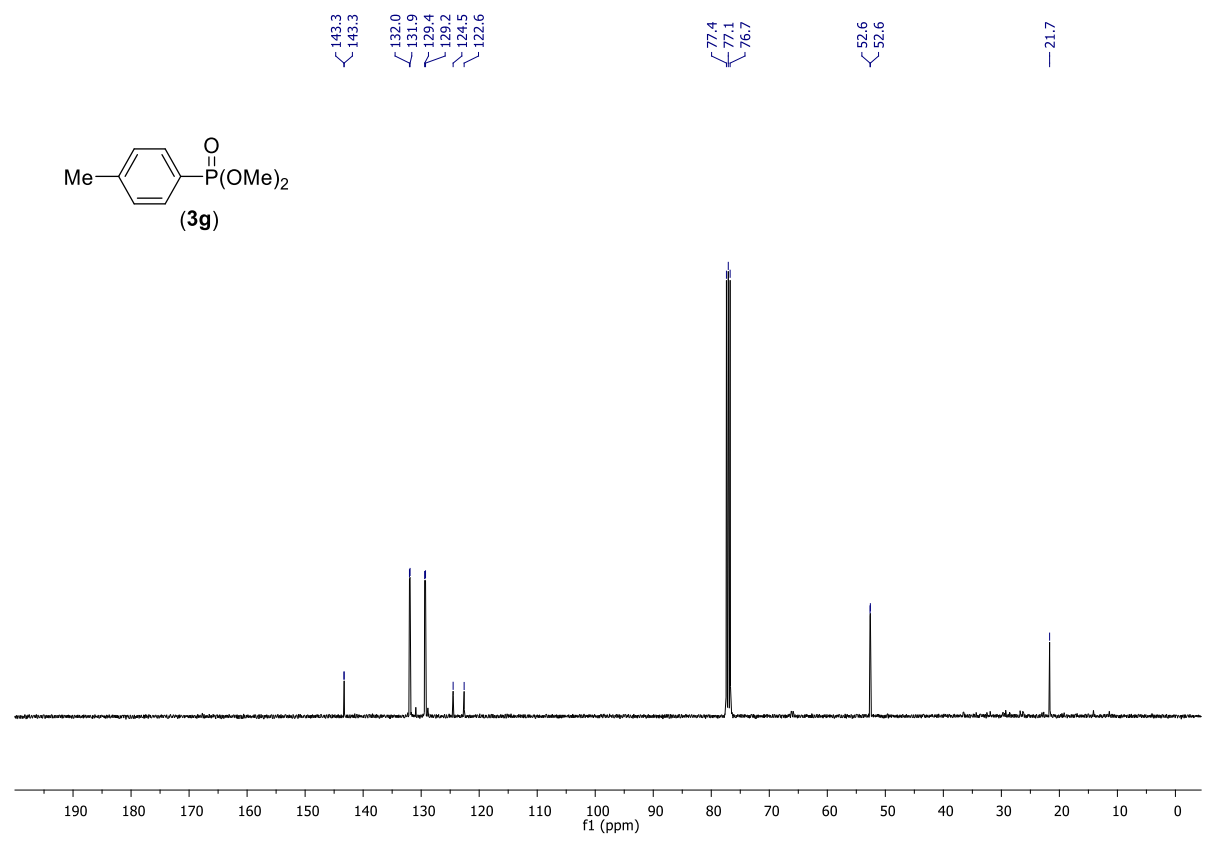
7.68
7.67
7.65
7.28
7.27
7.27
7.26
3.74
3.71
2.39
Me
O
P(OMe)2
(3g)
2.02
2.07
6.00
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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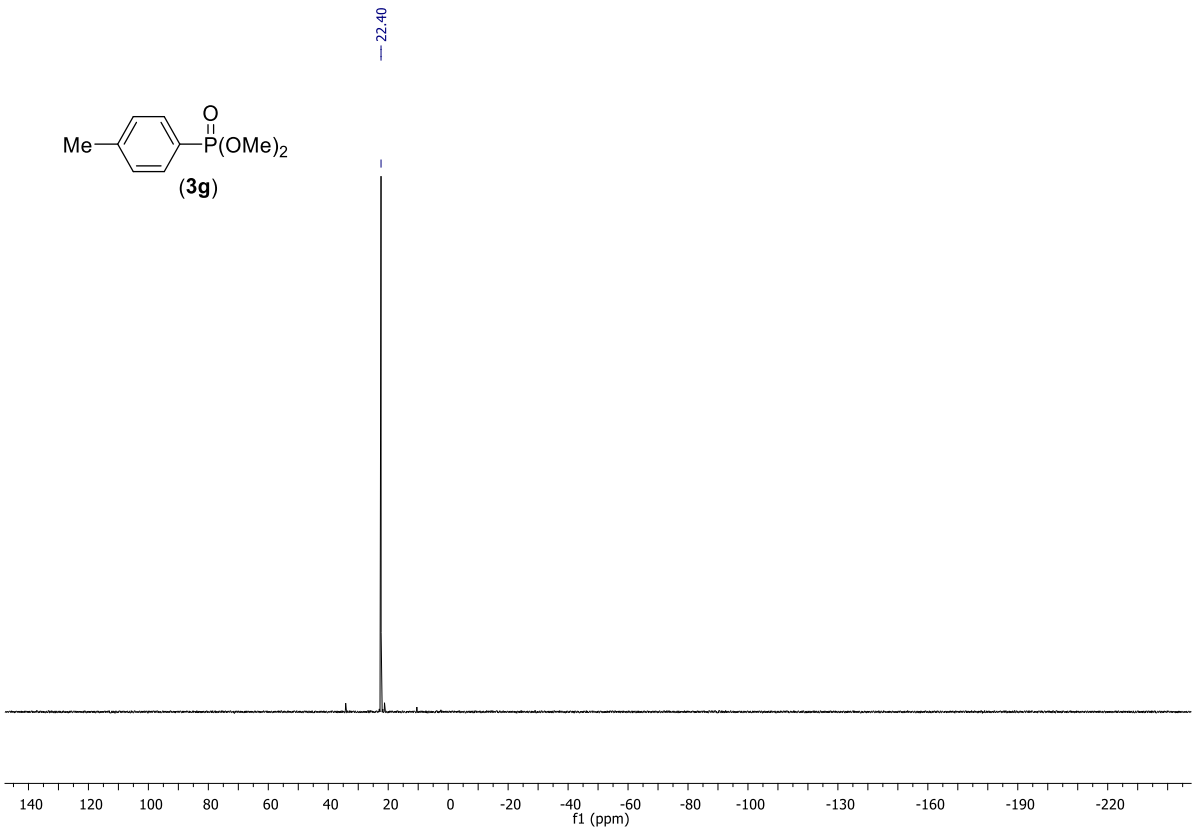
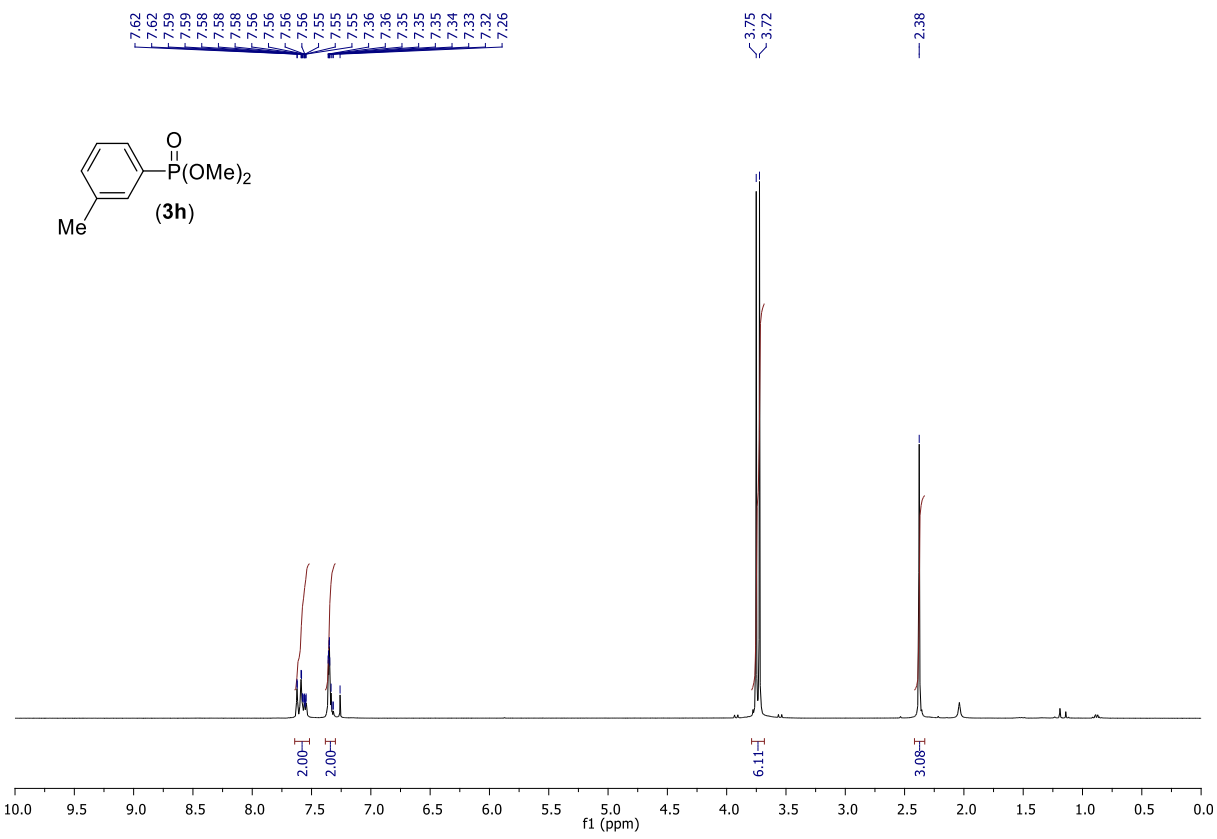
143.3
132.0
131.9
129.4
129.2
124.5
122.6
77.4
77.1
76.7
52.6
52.6
21.7
Me
O
P(OMe)2
(3g)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


22.40
Me
O
P(OMe)2
(3g)
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)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7.58
7.58
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7.56
7.56
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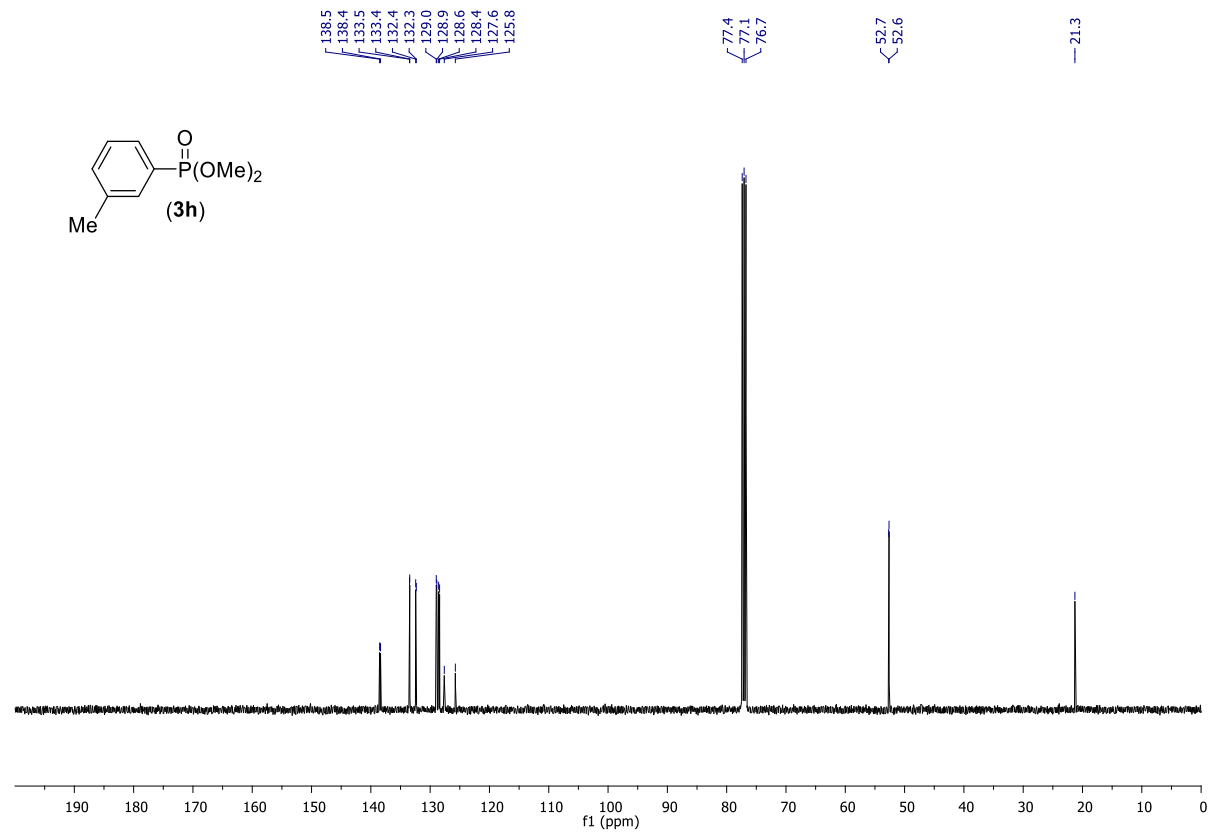
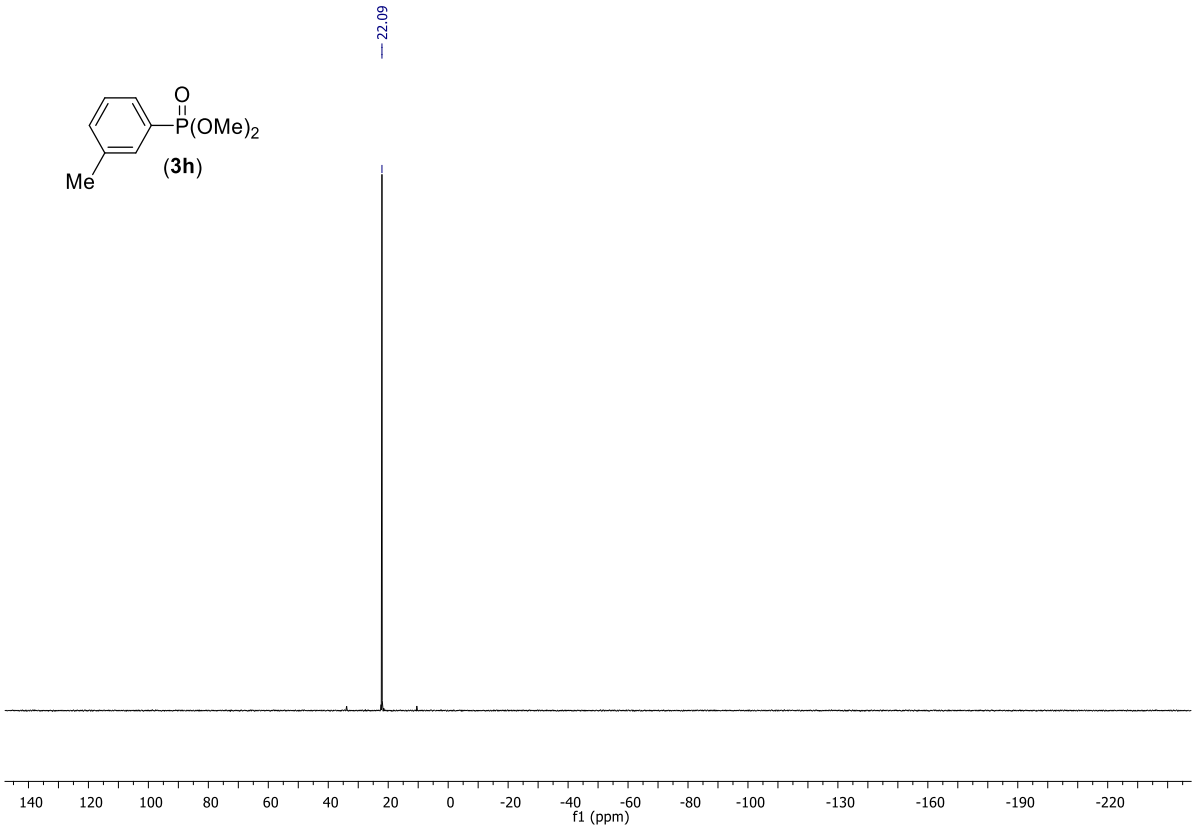
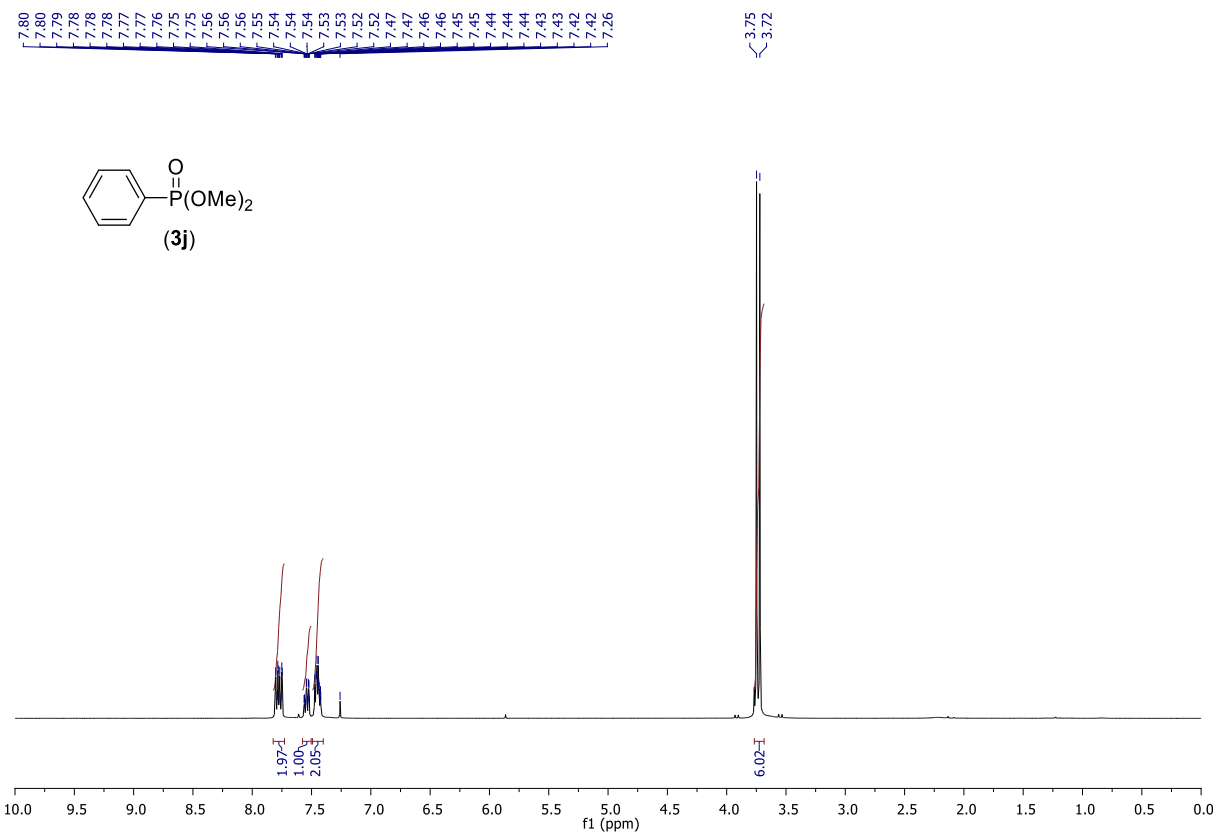
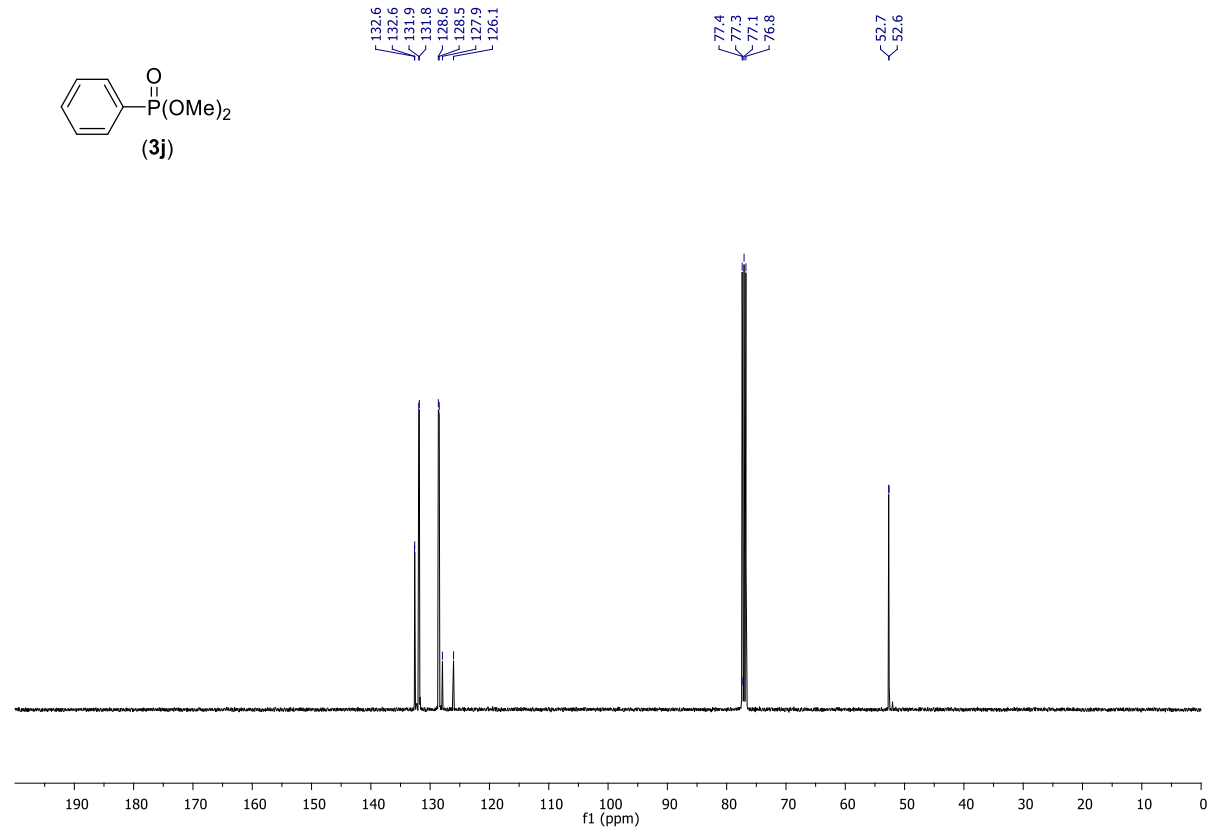
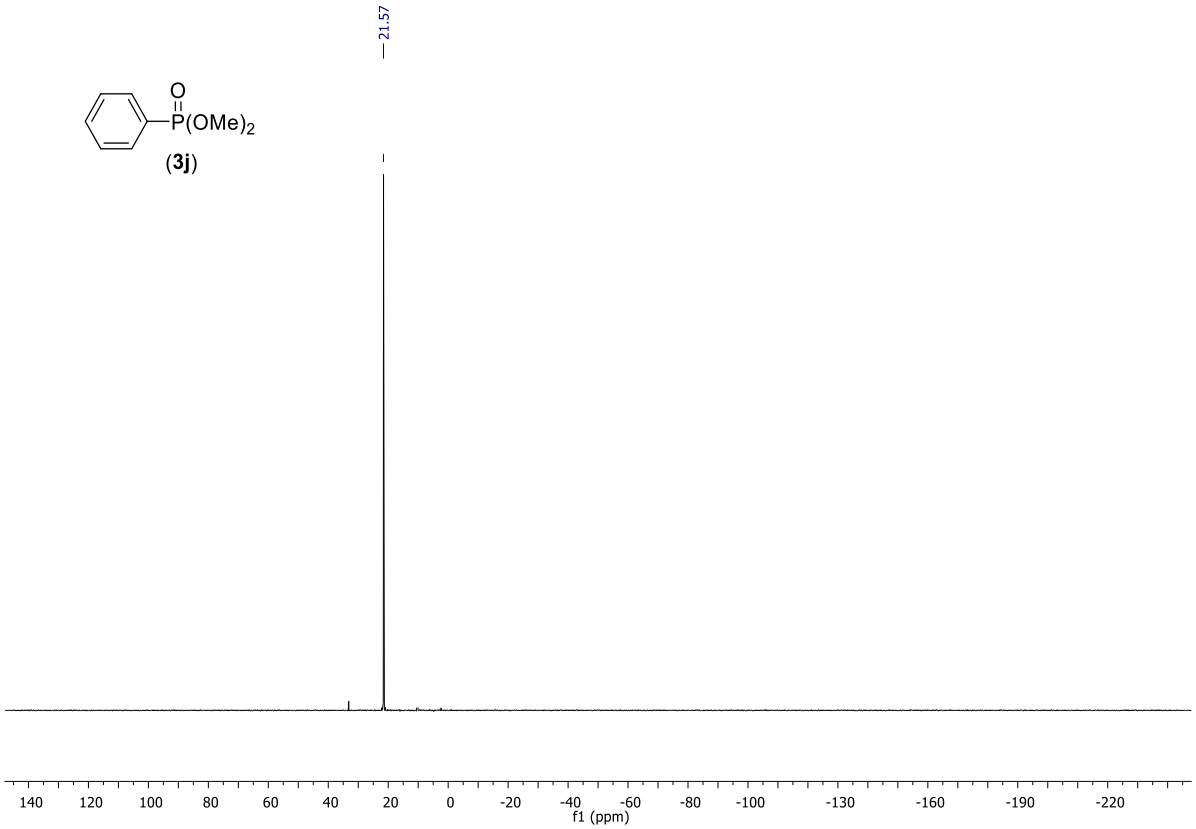
7.55
7.36
7.36
7.35
7.35
7.35
7.34
7.33
7.32
7.26
3.75
3.72
2.38
O
P(OMe)2
Me
(3h)
2.00
2.00
6.11
3.08
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


138.5
138.4
133.5
133.4
132.4
132.3
129.0
128.9
128.6
128.4
127.6
125.8
77.4
77.1
76.7
52.7
52.6
21.3
O
P(OMe)2
Me
(3h)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

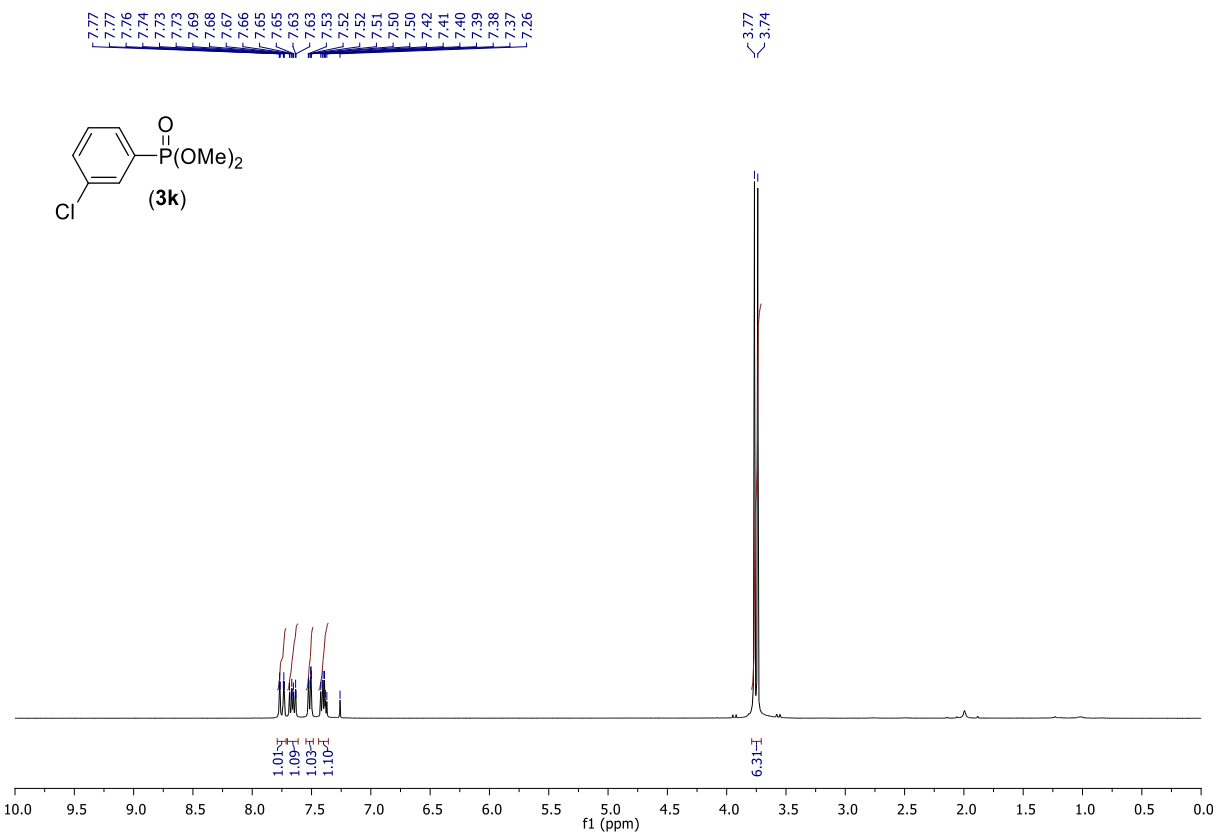

22.09
O
P(OMe)2
Me
(3h)
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)

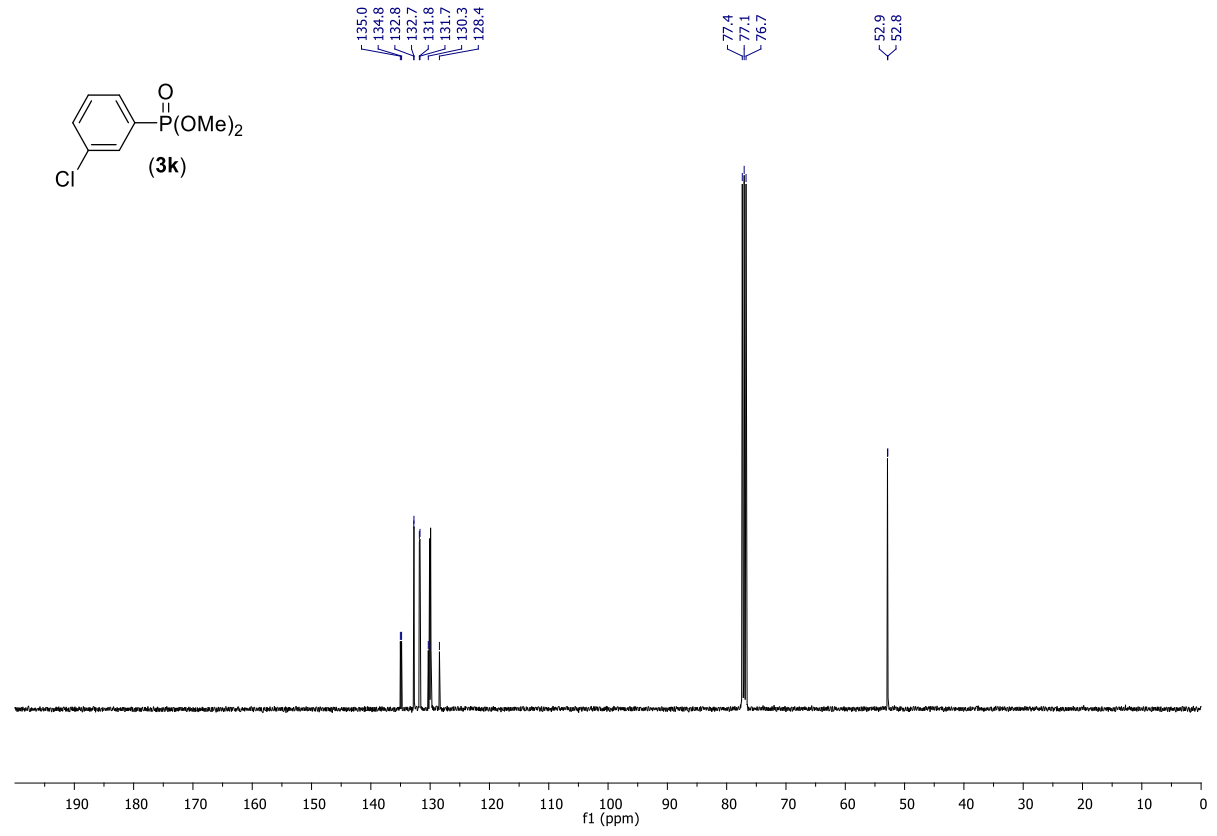

7.80
7.80
7.79
7.78
7.78
7.78
7.77
7.77
7.76
7.75
7.75
7.56
7.56
7.56
7.55
7.54
7.54
7.54
7.53
7.53
7.52
7.52
7.47
7.47
7.46
7.46
7.45
7.45
7.44
7.44
7.44
7.43
7.43
7.42
7.42
7.26
3.75
3.72
O
P(OMe)2
(3j)
1.97
1.00
2.05
6.02
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

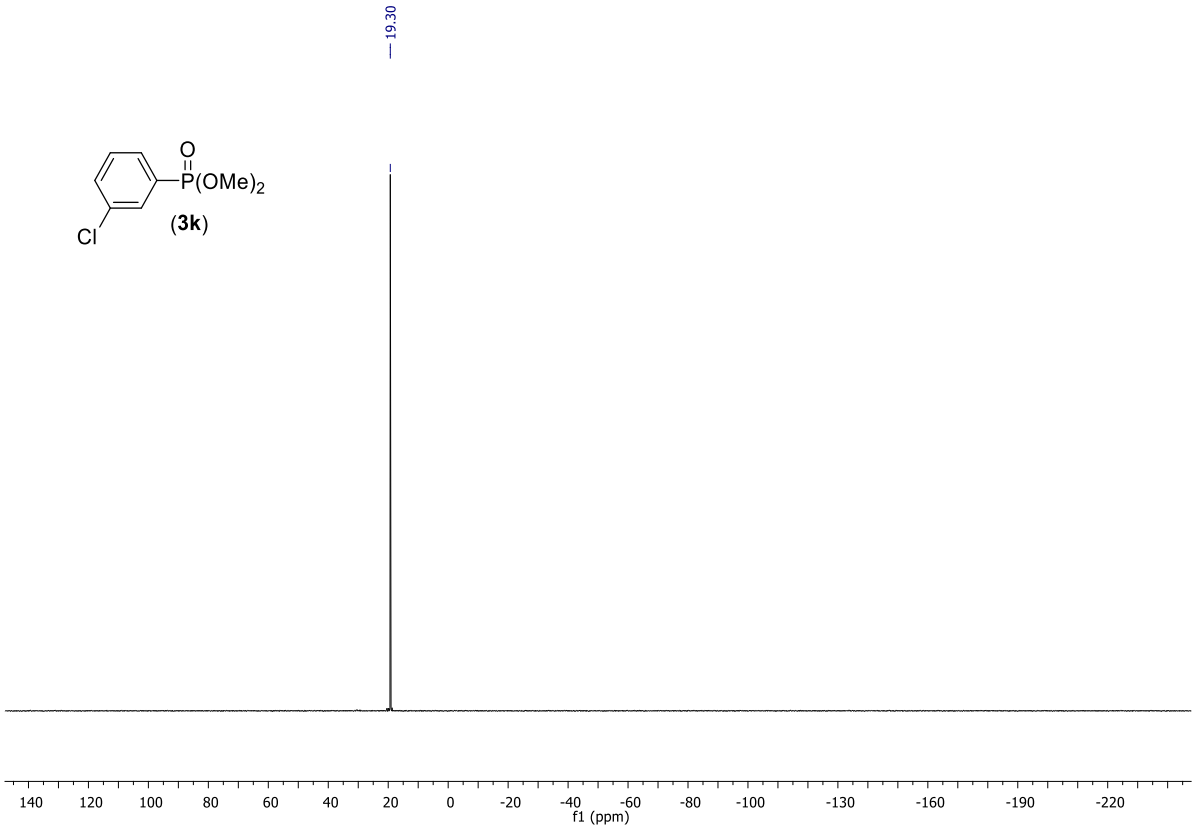

132.6
132.6
131.9
131.8
128.6
128.5
127.9
126.1
77.4
77.3
77.1
76.8
52.7
52.6
O
P(OMe)2
(3j)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

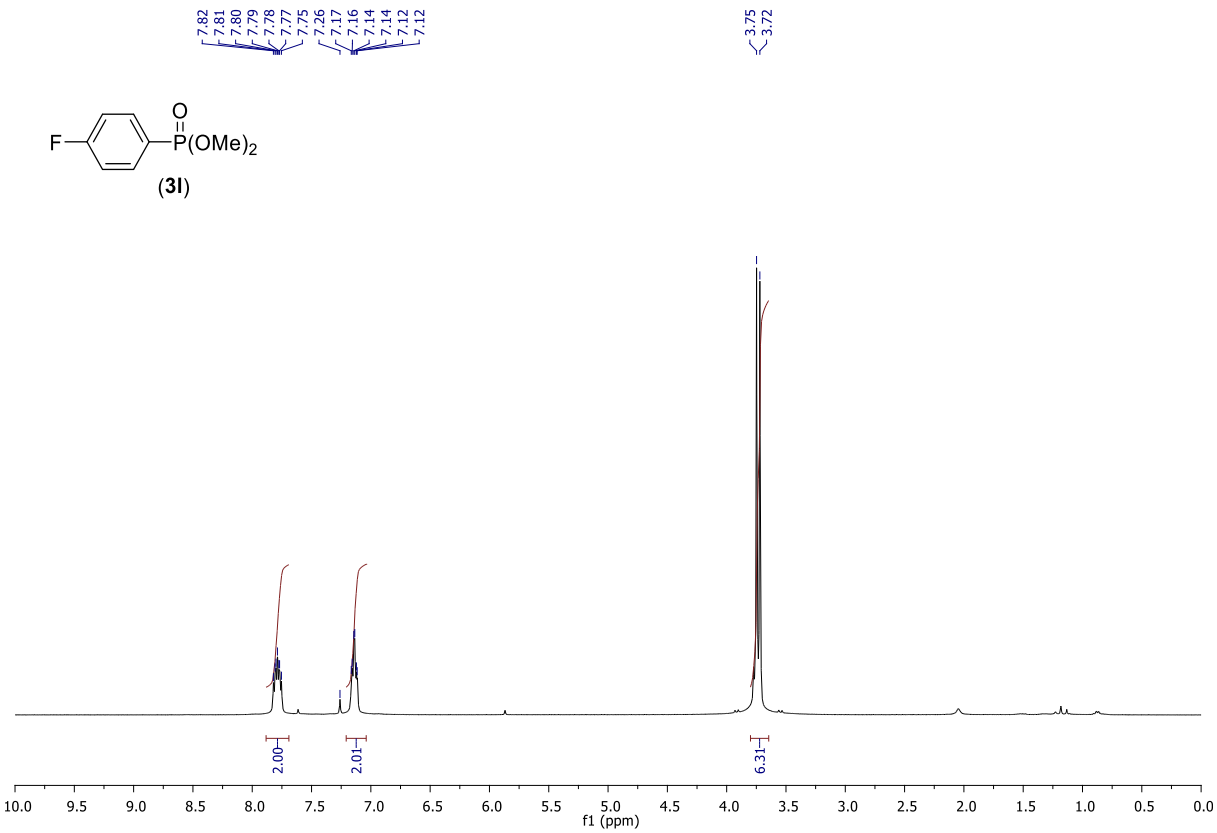

21.57
O
P(OMe)2
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)

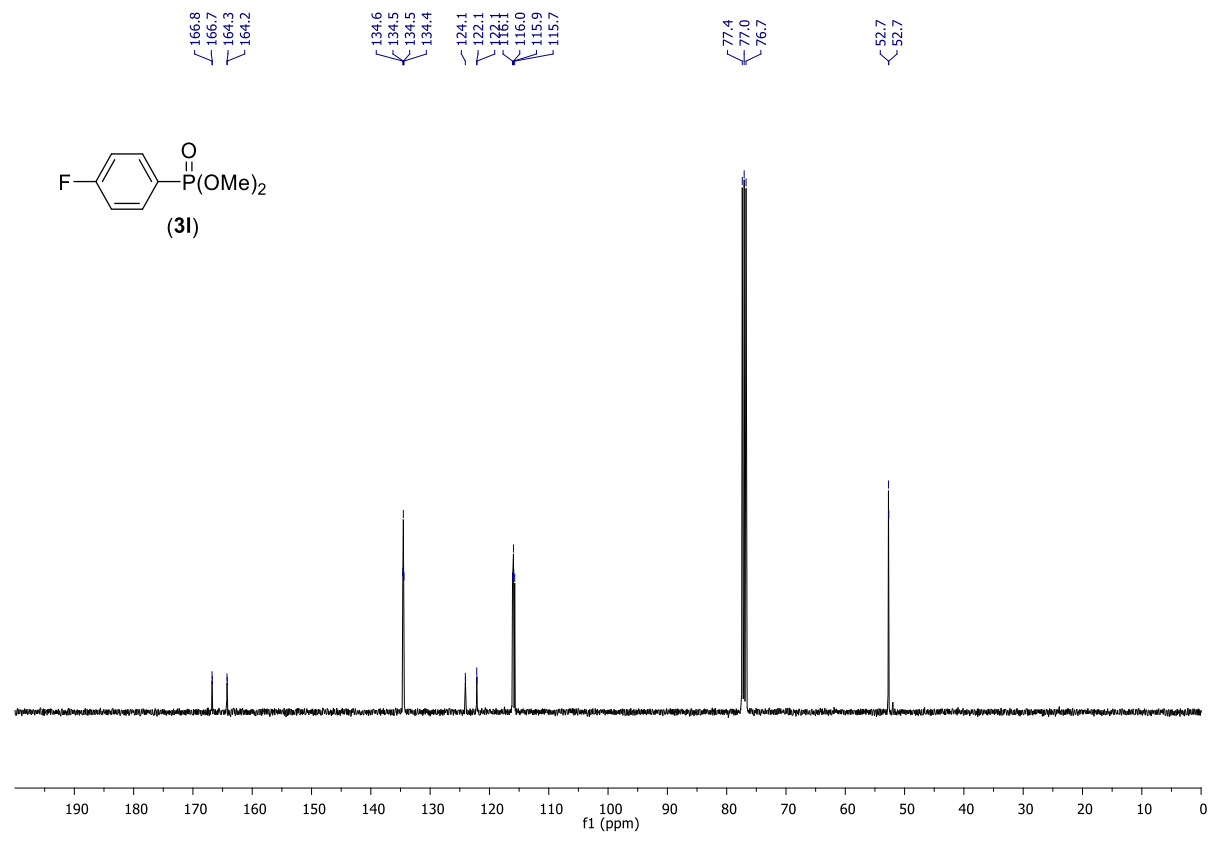
(**3j**)

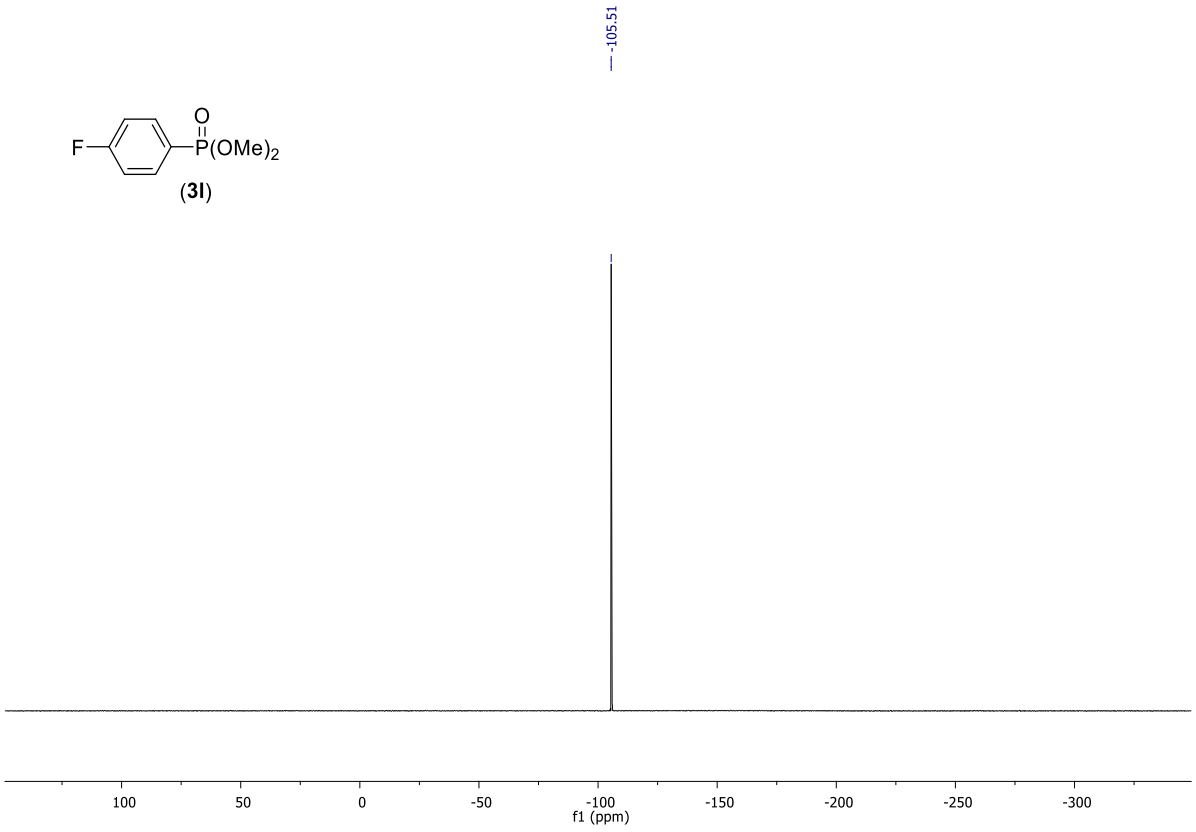

7.77
7.77
7.76
7.74
7.73
7.73
7.69
7.68
7.67
7.66
7.65
7.65
7.63
7.63
7.53
7.52
7.52
7.51
7.50
7.50
7.42
7.41
7.40
7.39
7.38
7.37
7.26
3.77
3.74
O
P(OMe)2
Cl
(3k)
1.01
1.09
1.03
1.10
6.31
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


135.0
134.8
132.8
132.7
131.8
131.7
130.3
128.4
77.4
77.1
76.7
52.9
52.8
O
P(OMe)2
Cl
(3k)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

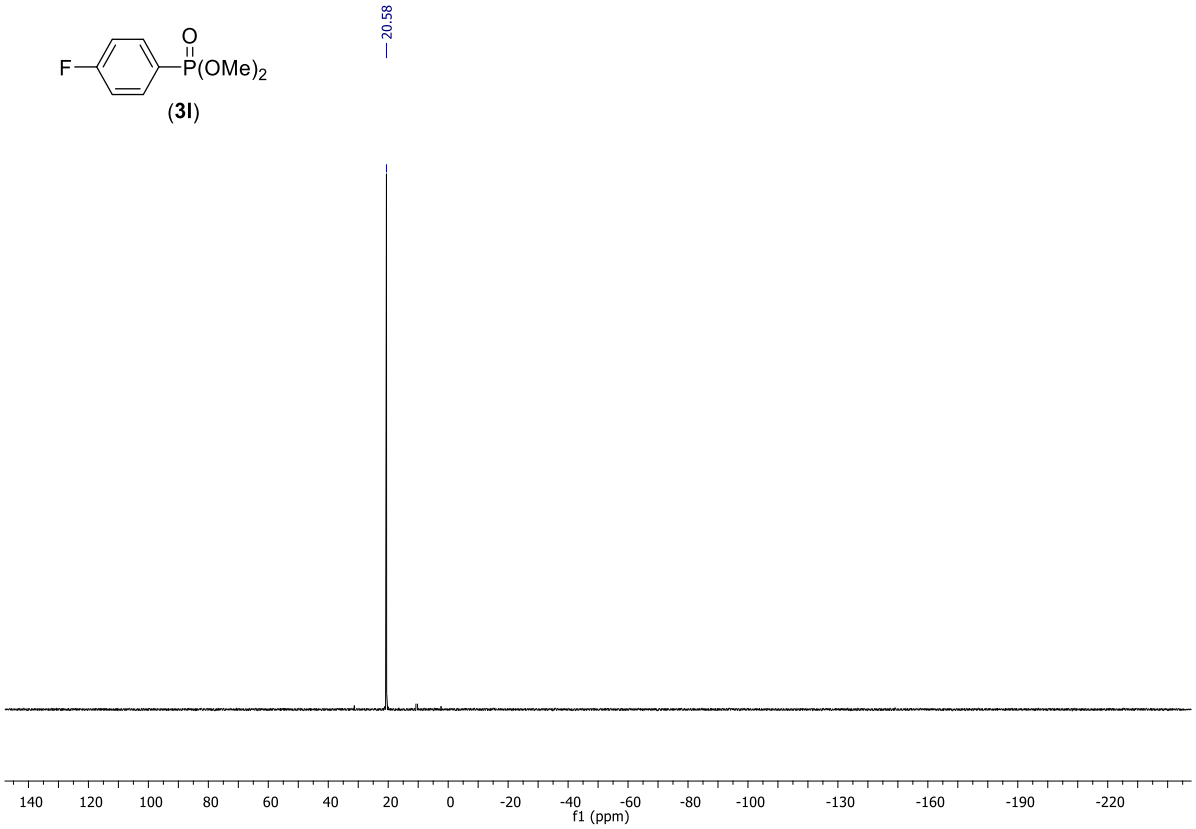

19.30
O
P(OMe)2
Cl
(3k)
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)


7.82
7.81
7.80
7.79
7.78
7.77
7.75
7.26
7.17
7.16
7.14
7.14
7.12
7.12
3.75
3.72
F
O
P(OMe)2
(3l)
2.00
2.01
6.31
10.0
9.5
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
0.0
f1 (ppm)

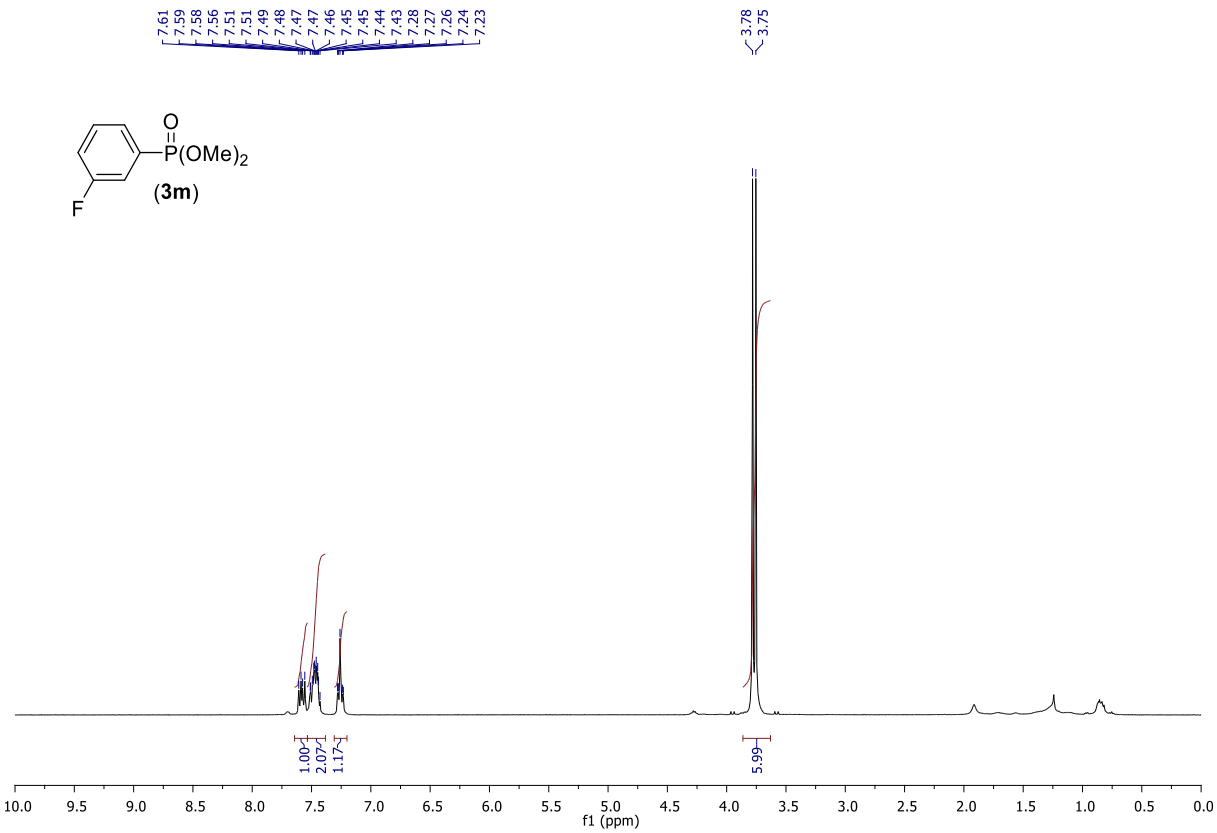

166.8
166.7
164.3
164.2
134.6
134.5
134.5
134.4
124.1
122.1
122.1
116.1
116.0
115.9
115.7
77.4
77.0
76.7
52.7
52.7
F
O
P(OMe)2
(3l)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

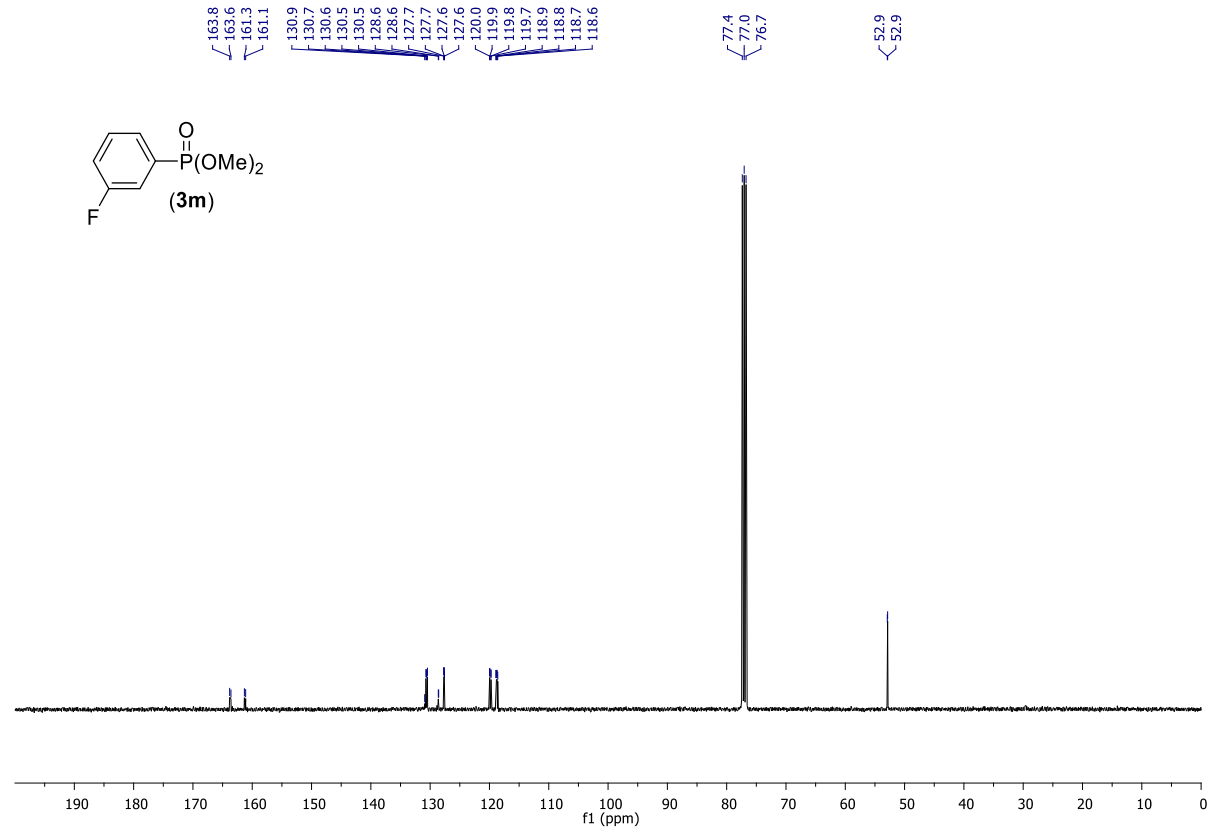

-105.51
F
O
P(OMe)2
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

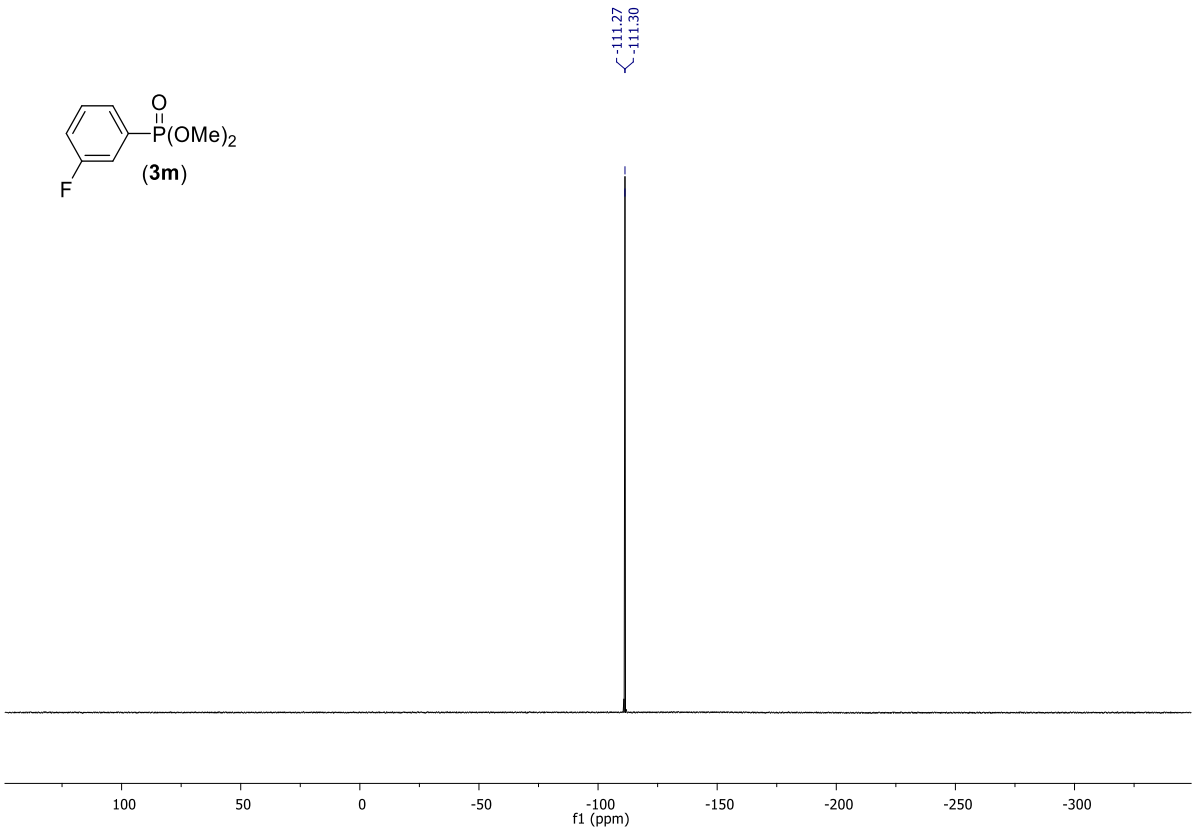
(**3l**)

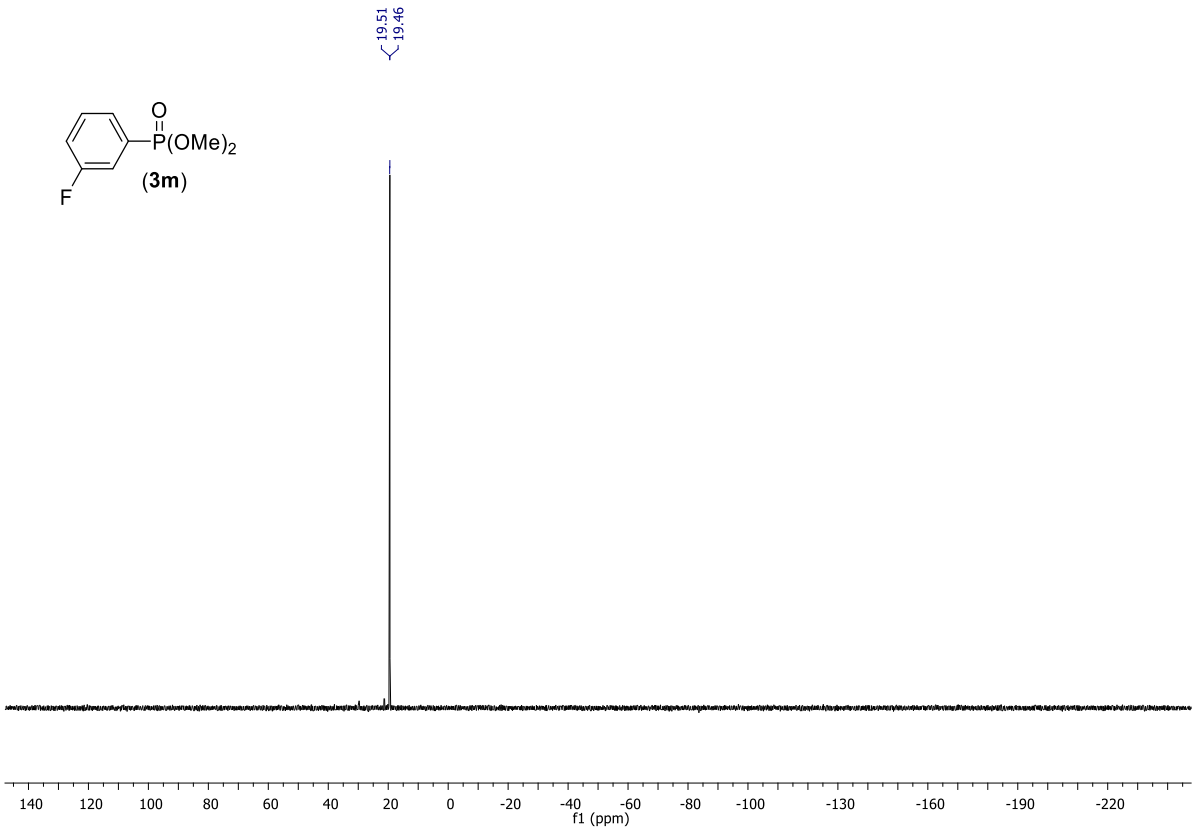

20.58
F
O
P(OMe)2
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)

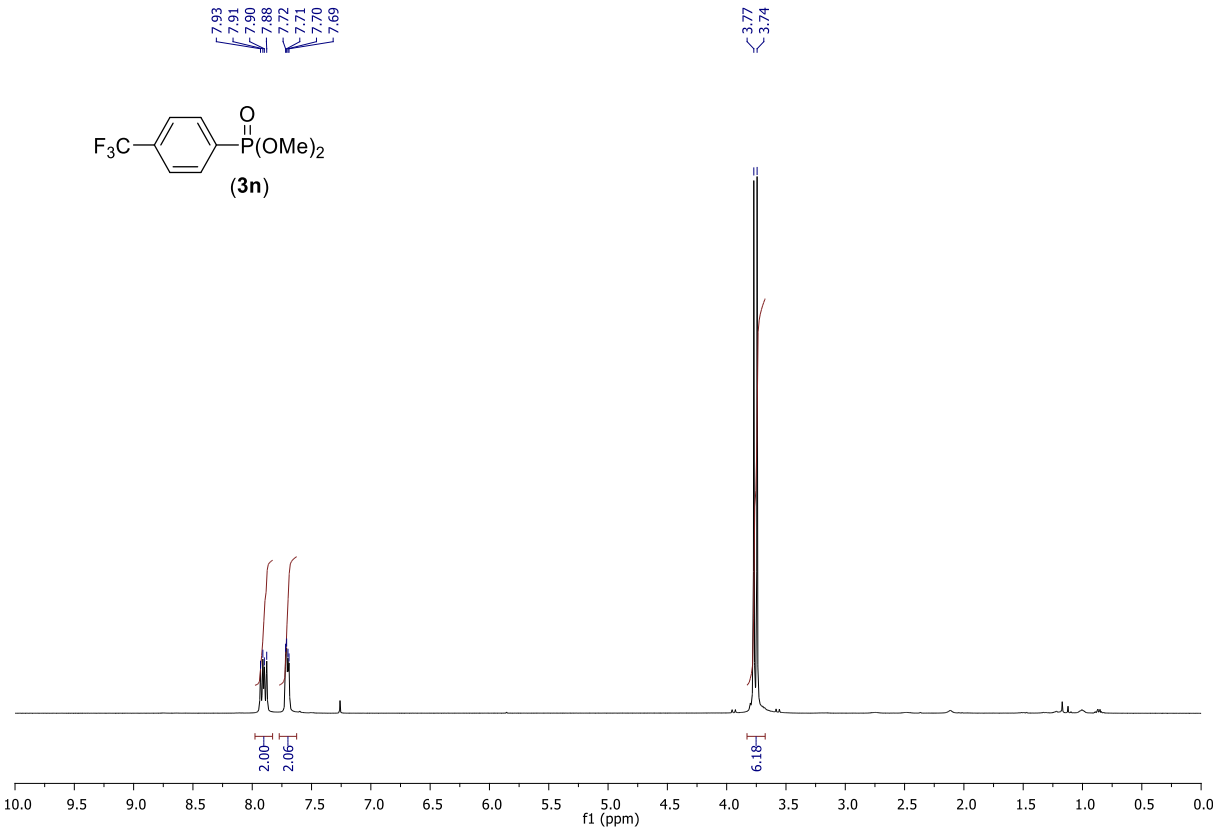
(**3l**)

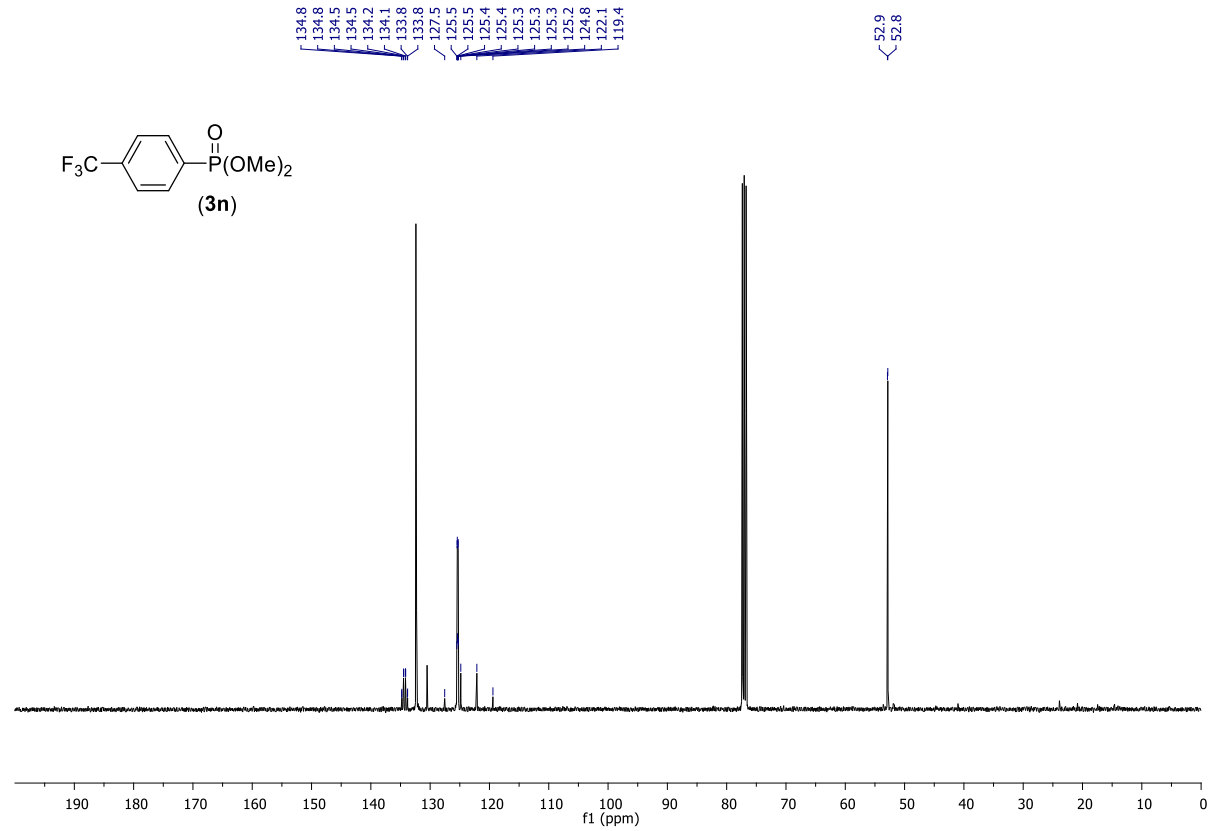

7.61
7.59
7.58
7.56
7.51
7.51
7.49
7.48
7.47
7.47
7.46
7.45
7.45
7.44
7.43
7.28
7.27
7.26
7.24
7.23
3.78
3.75
O
P(OMe)2
F
(3m)
1.00
2.07
1.17
5.99
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

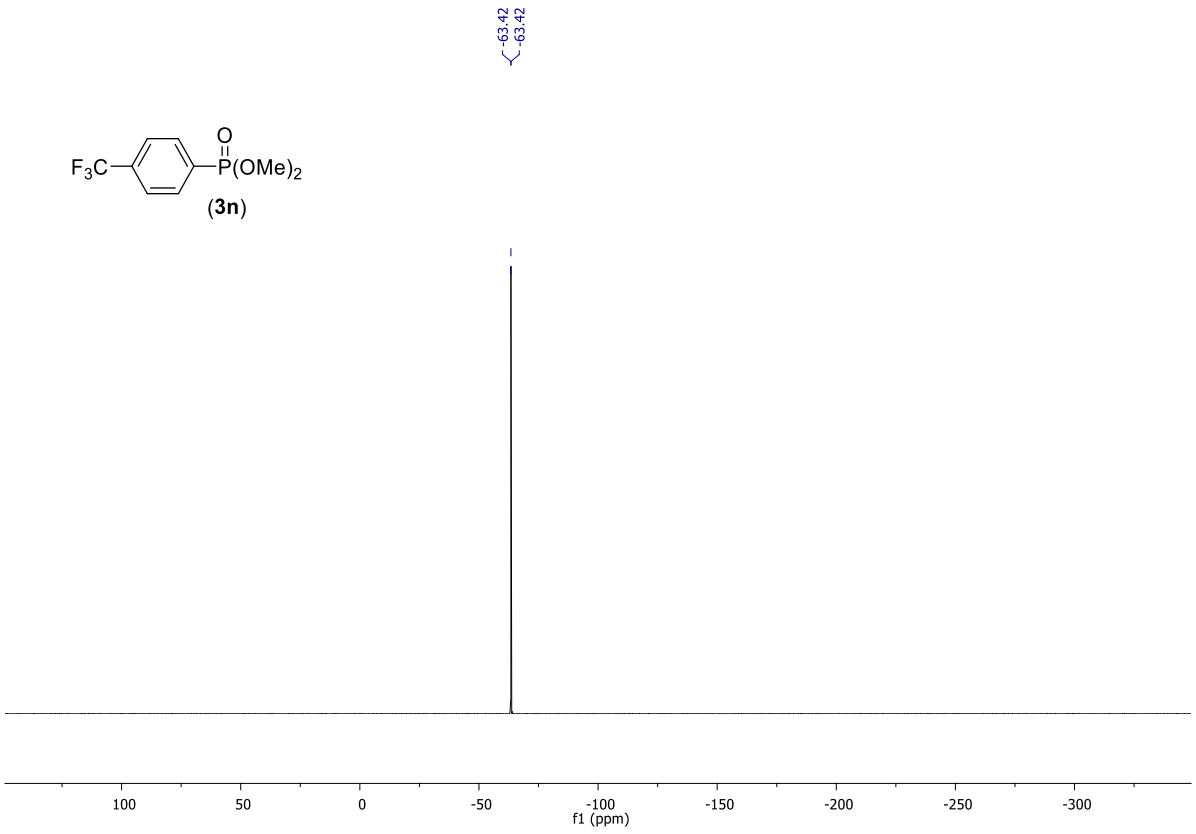

163.8
163.6
161.3
161.1
130.9
130.7
130.6
130.5
130.5
128.6
128.6
127.7
127.7
127.6
127.6
120.0
119.9
119.8
119.7
118.9
118.8
118.7
118.6
77.4
77.0
76.7
52.9
52.9
O
P(OMe)2
F
(3m)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

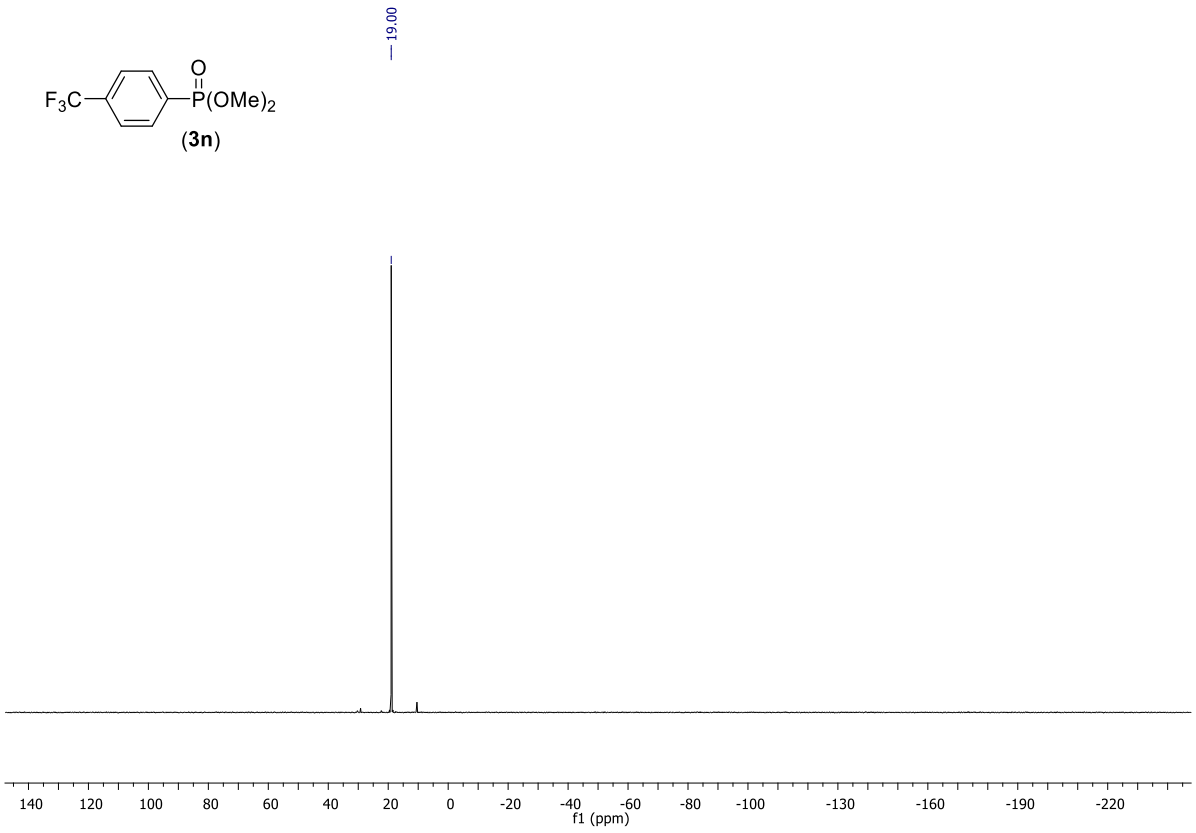

-111.27
-111.30
O
P(OMe)2
(3m)
F
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

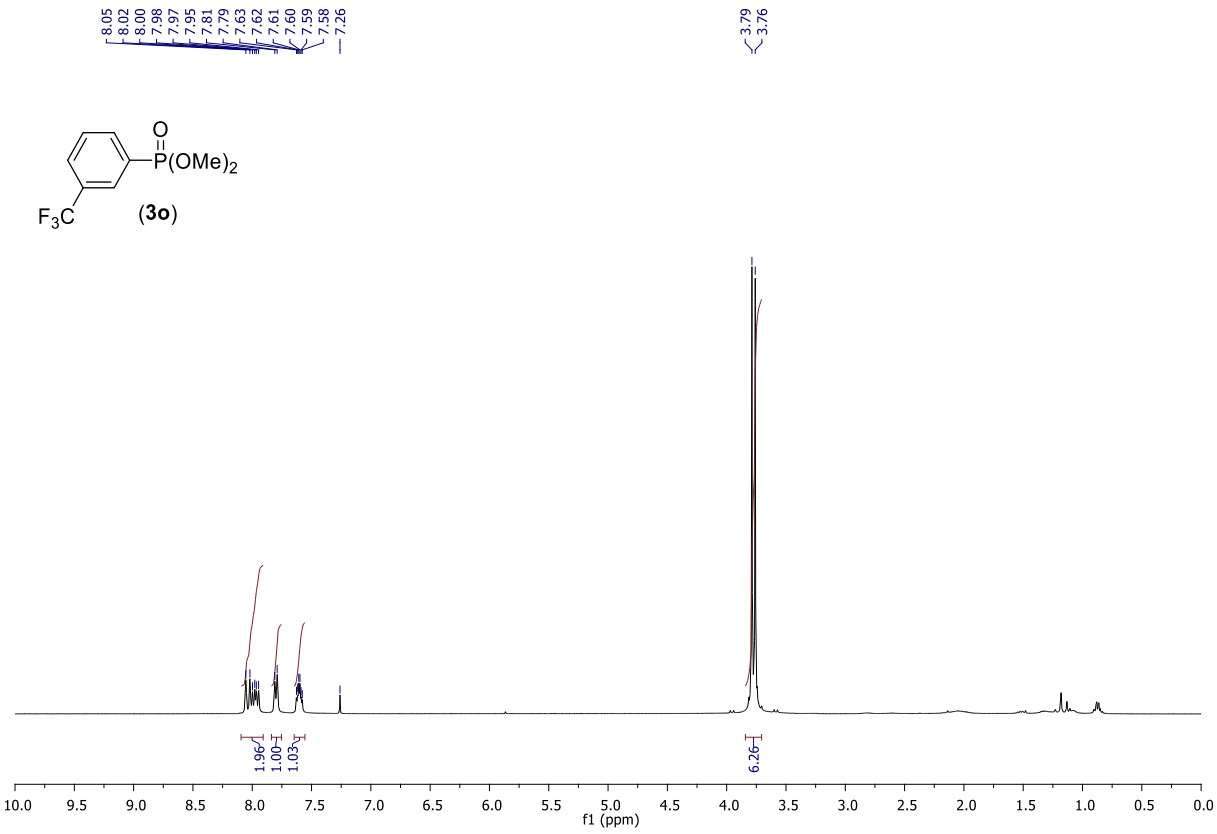

19.51
19.46
O
P(OMe)2
(3m)
F
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)

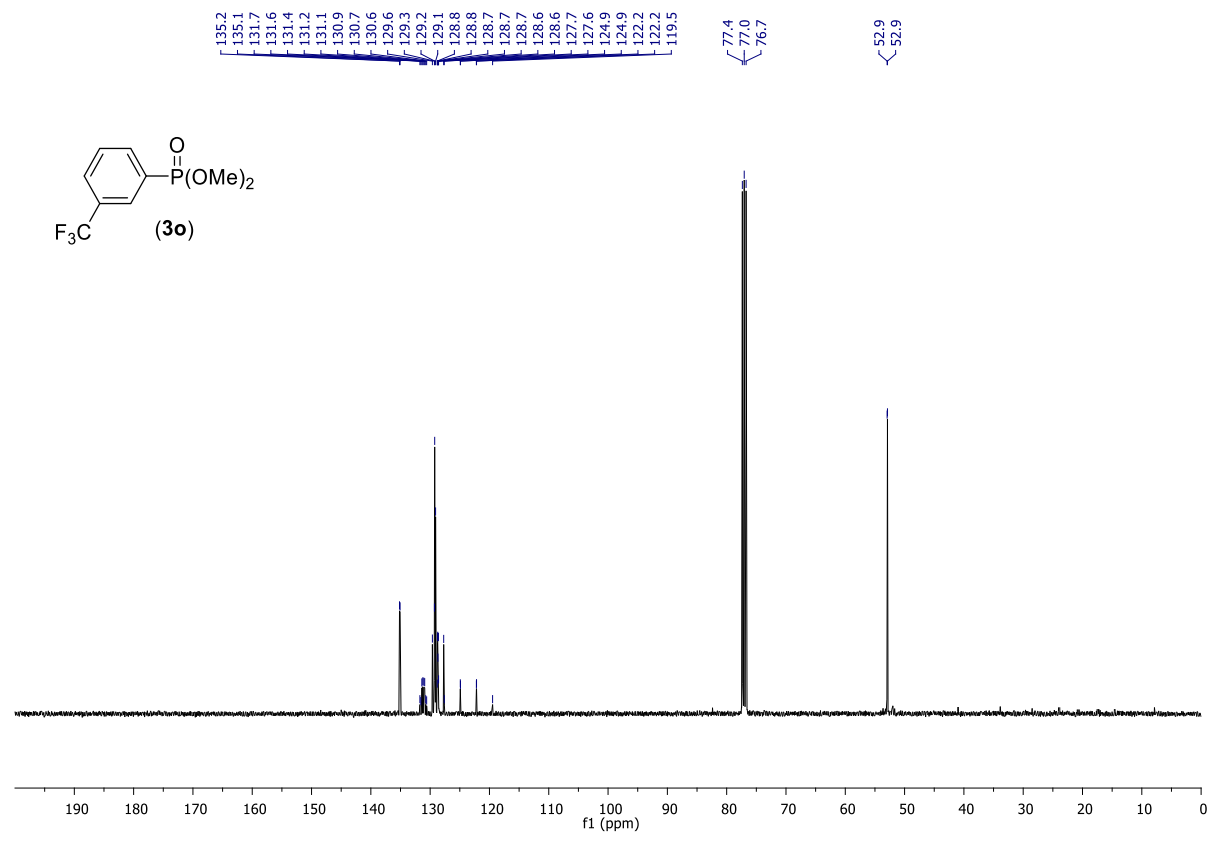

7.93
7.91
7.90
7.88
7.72
7.71
7.70
7.69
3.77
3.74
O
F3C
P(OMe)2
(3n)
2.00
2.06
6.18
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

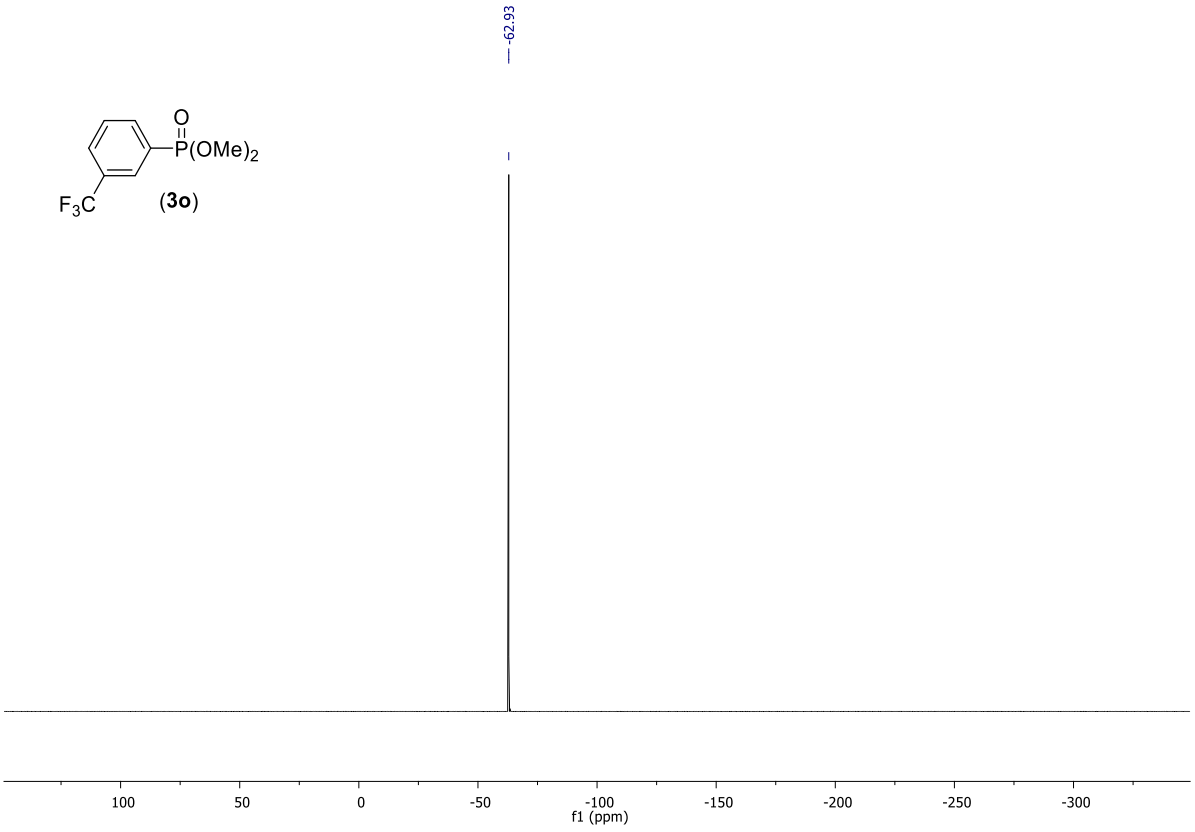

134.8
134.8
134.5
134.5
134.2
134.1
133.8
133.8
127.5
125.5
125.5
125.4
125.4
125.3
125.3
125.3
125.2
124.8
122.1
119.4
52.9
52.8
O
F3C
P(OMe)2
(3n)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

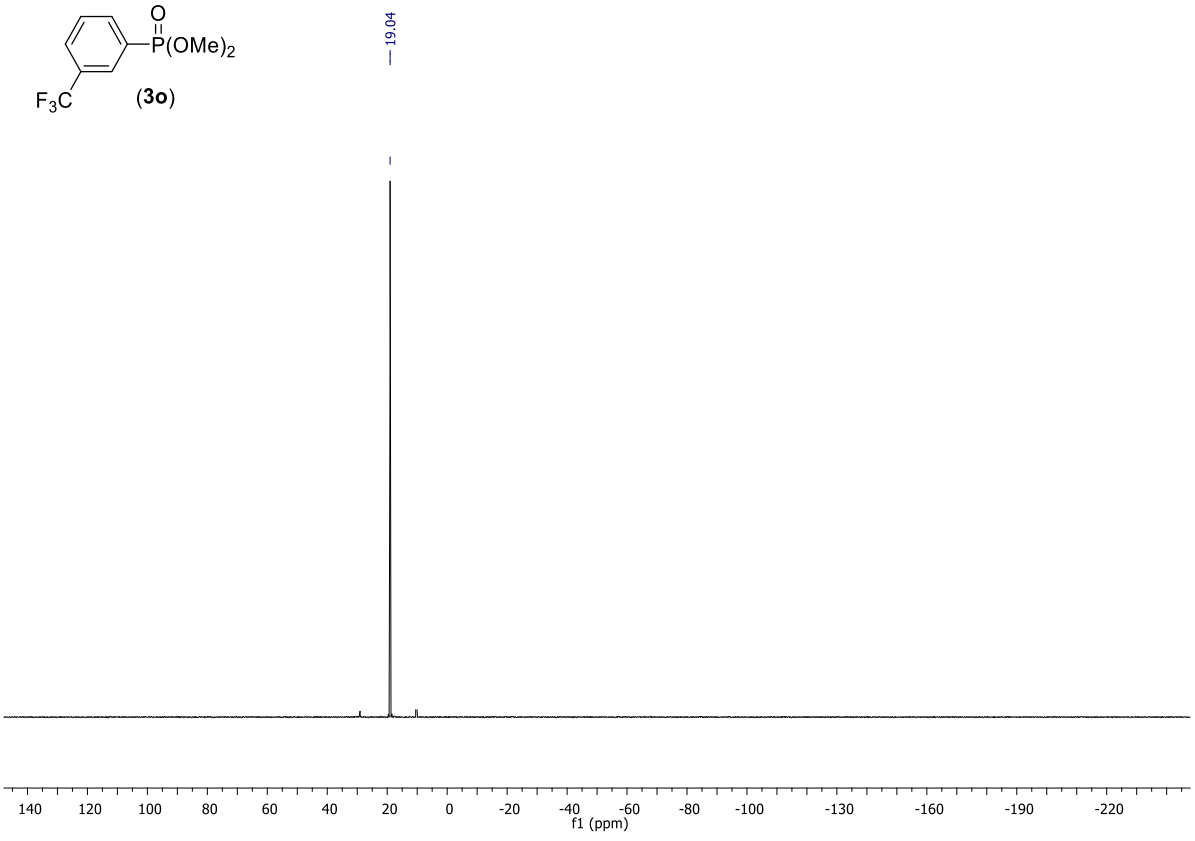

-63.42
-63.42
O
F3C
P(OMe)2
(3n)
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

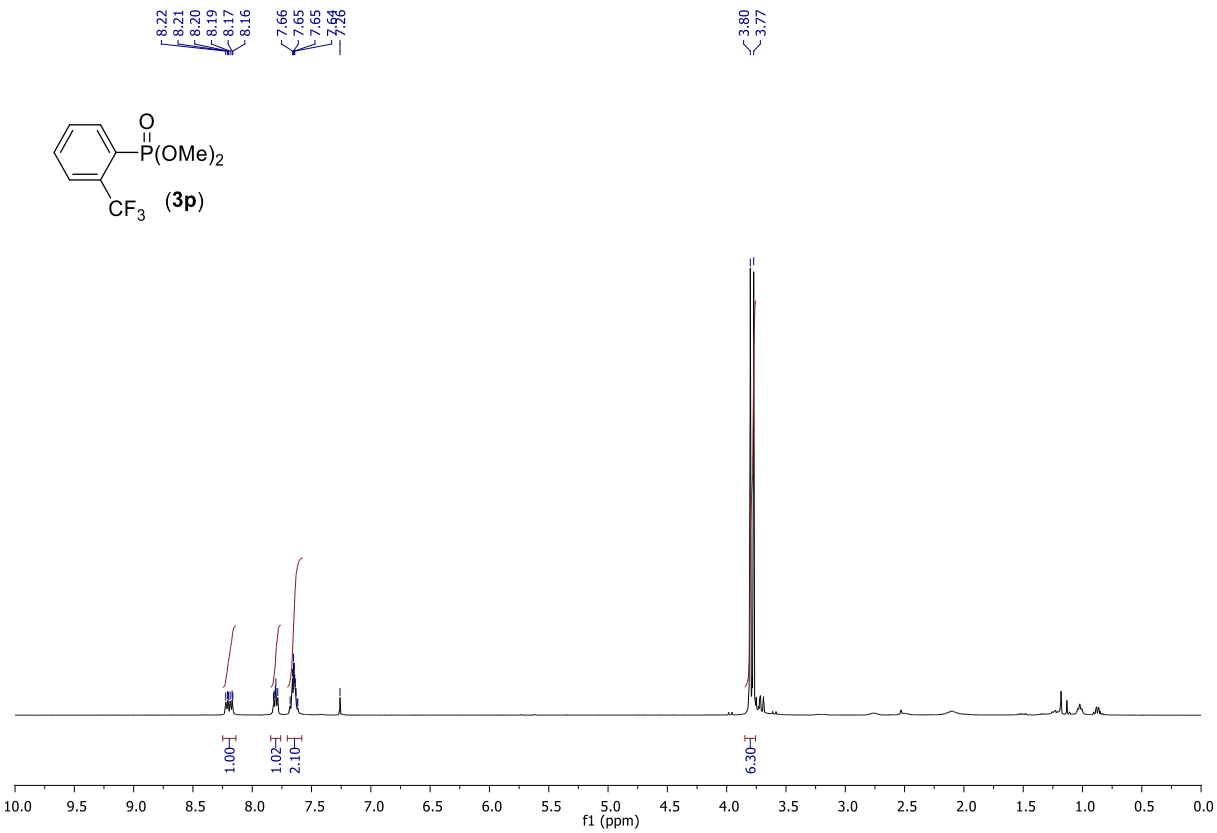

19.00
O
F3C
P(OMe)2
(3n)
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)

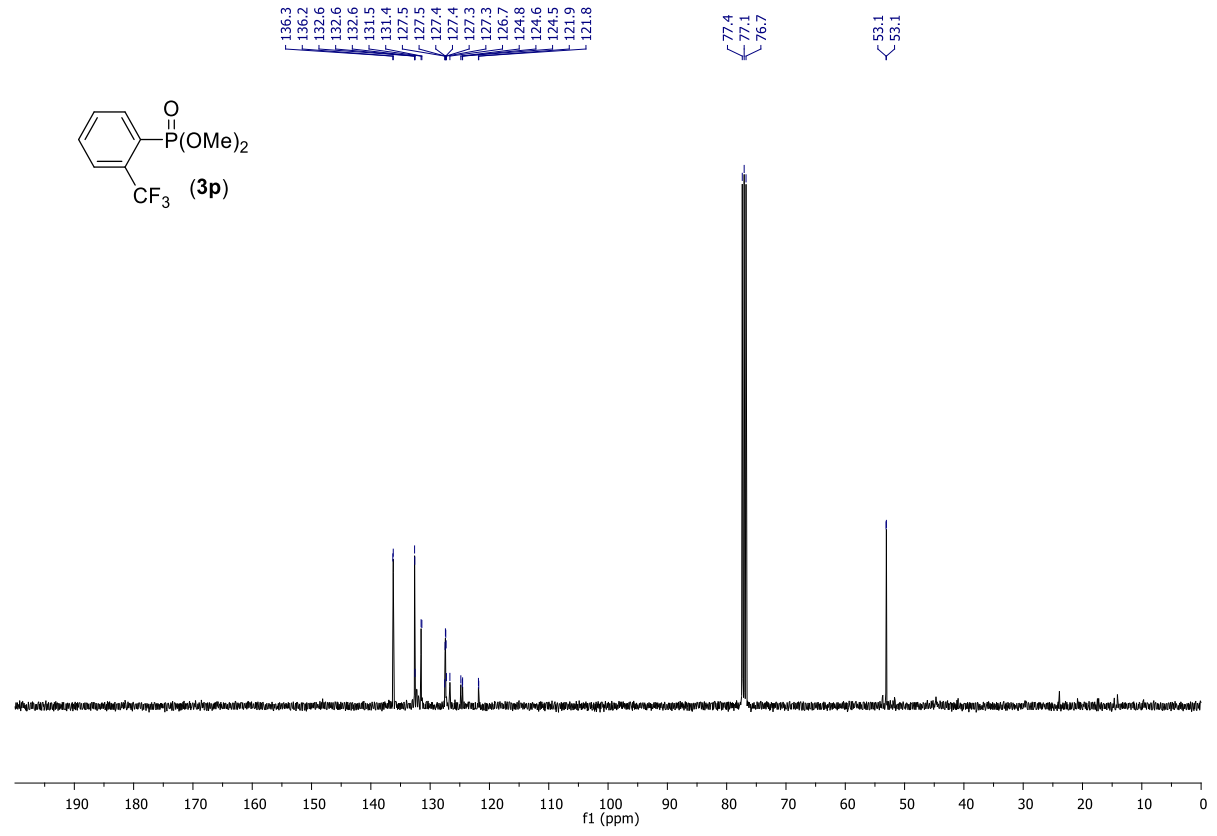

8.05
8.02
8.00
7.98
7.97
7.95
7.81
7.79
7.63
7.62
7.61
7.60
7.59
7.58
7.26
3.79
3.76
O
P(OMe)2
F3C
(3o)
1.96
1.00
1.03
6.26
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

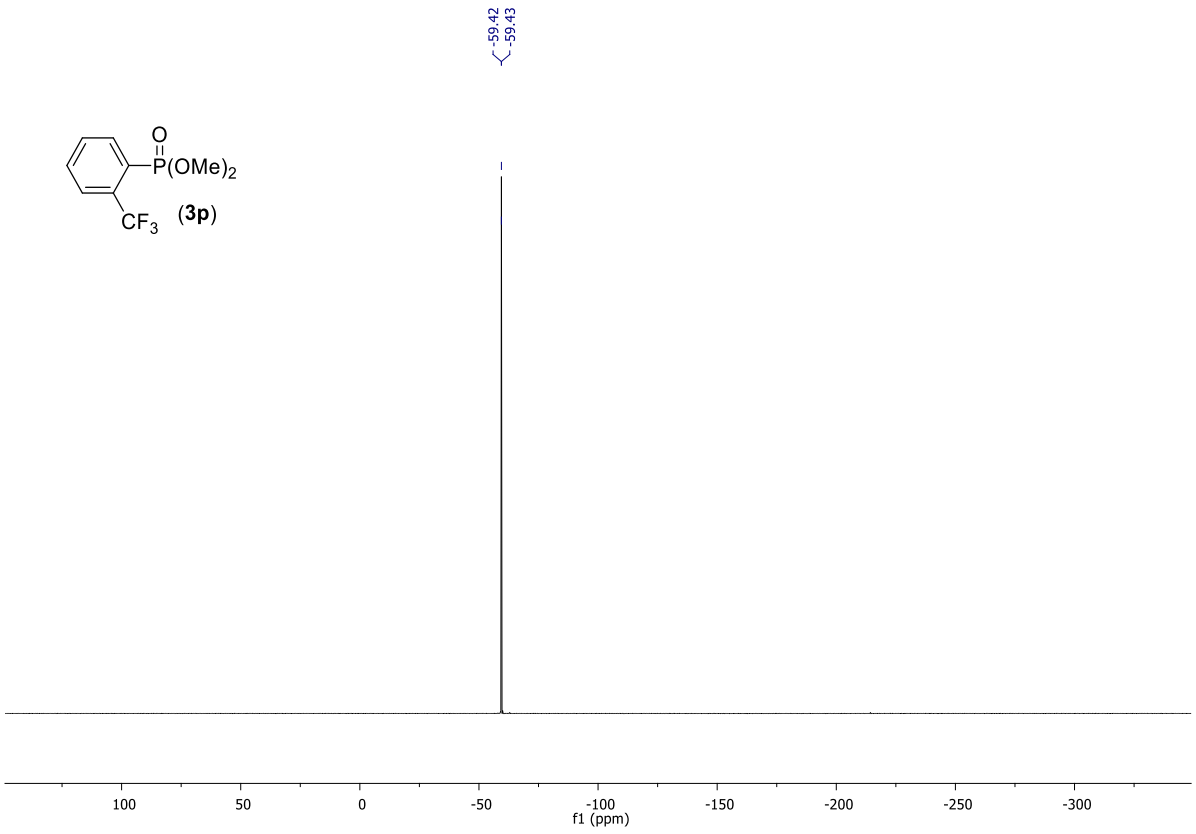

135.2
135.1
131.7
131.6
131.4
131.2
131.1
130.9
130.7
130.6
129.6
129.3
129.2
129.1
128.8
128.8
128.7
128.7
128.7
128.6
128.6
127.7
127.6
124.9
124.9
122.2
122.2
119.5
77.4
77.0
76.7
52.9
52.9
O
P(OMe)2
F3C
(3o)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

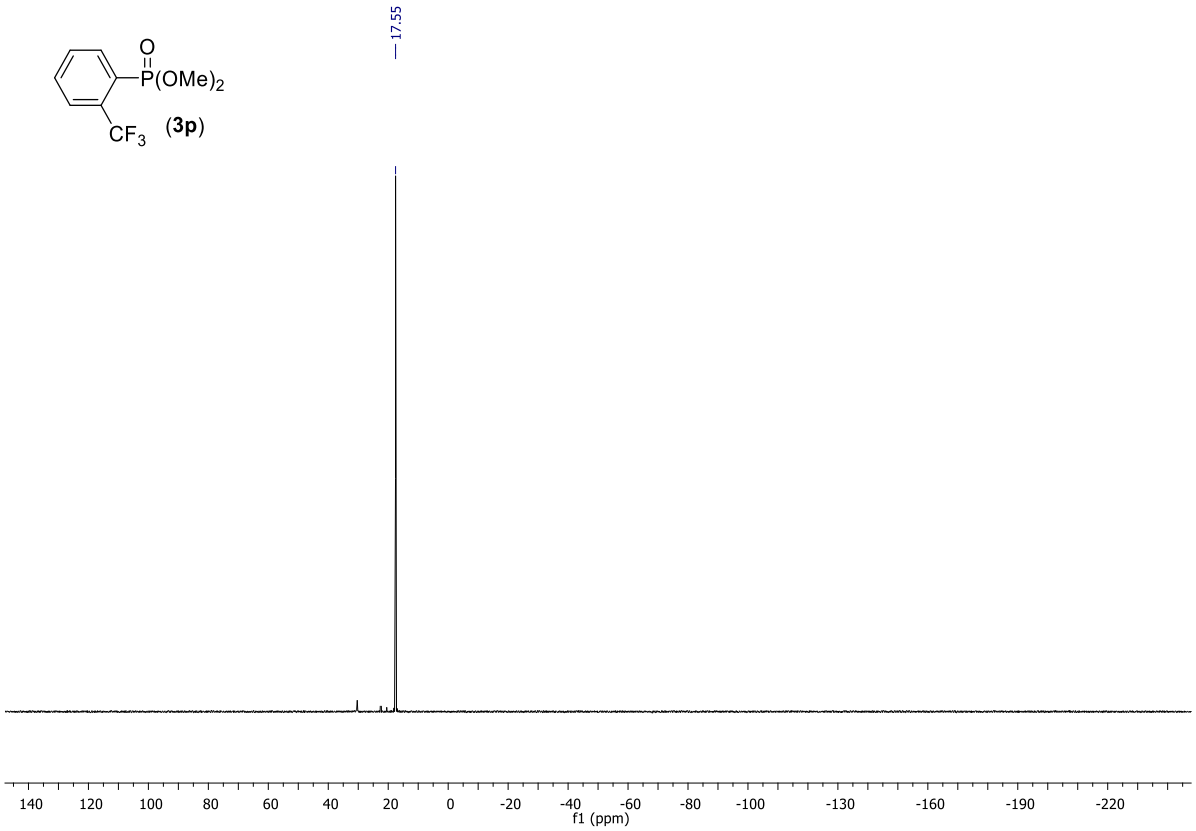

-62.93
O
P(OMe)2
F3C
(3o)
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

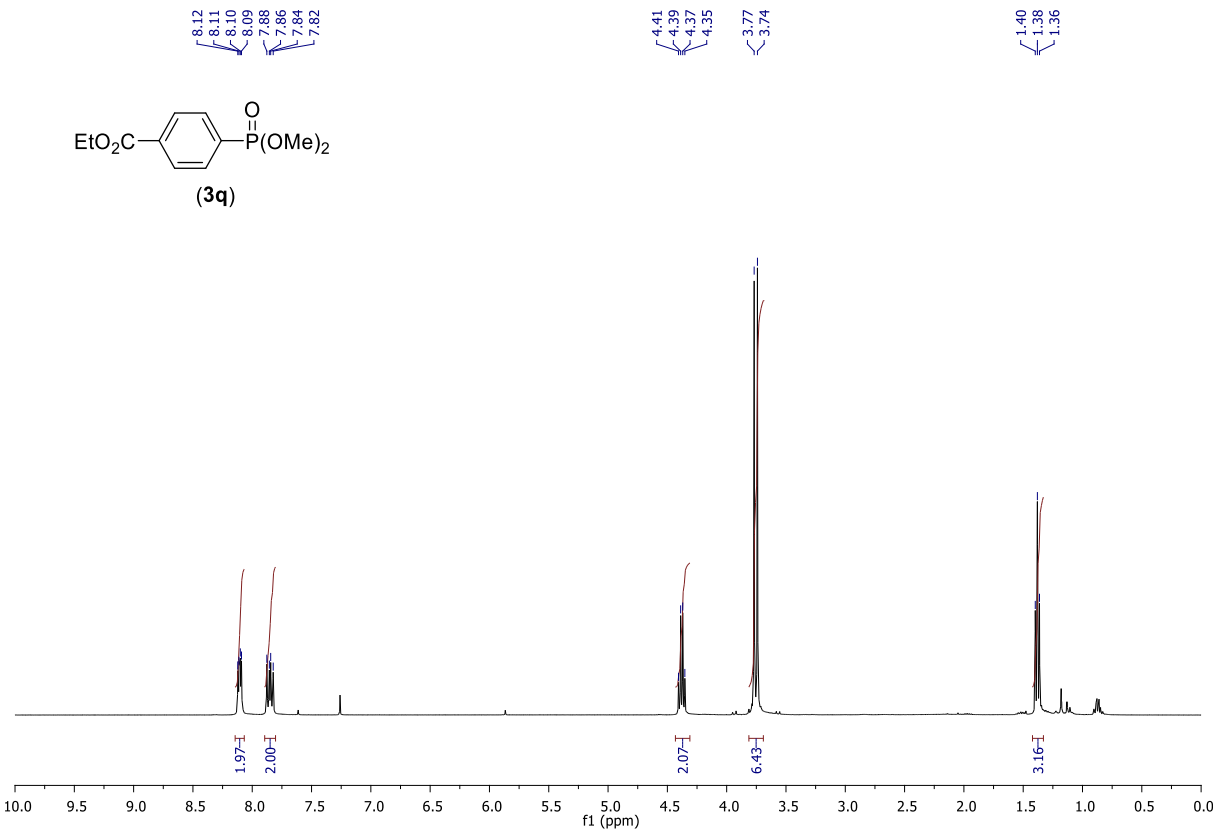

19.04
O
P(OMe)2
F3C
(3o)
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)

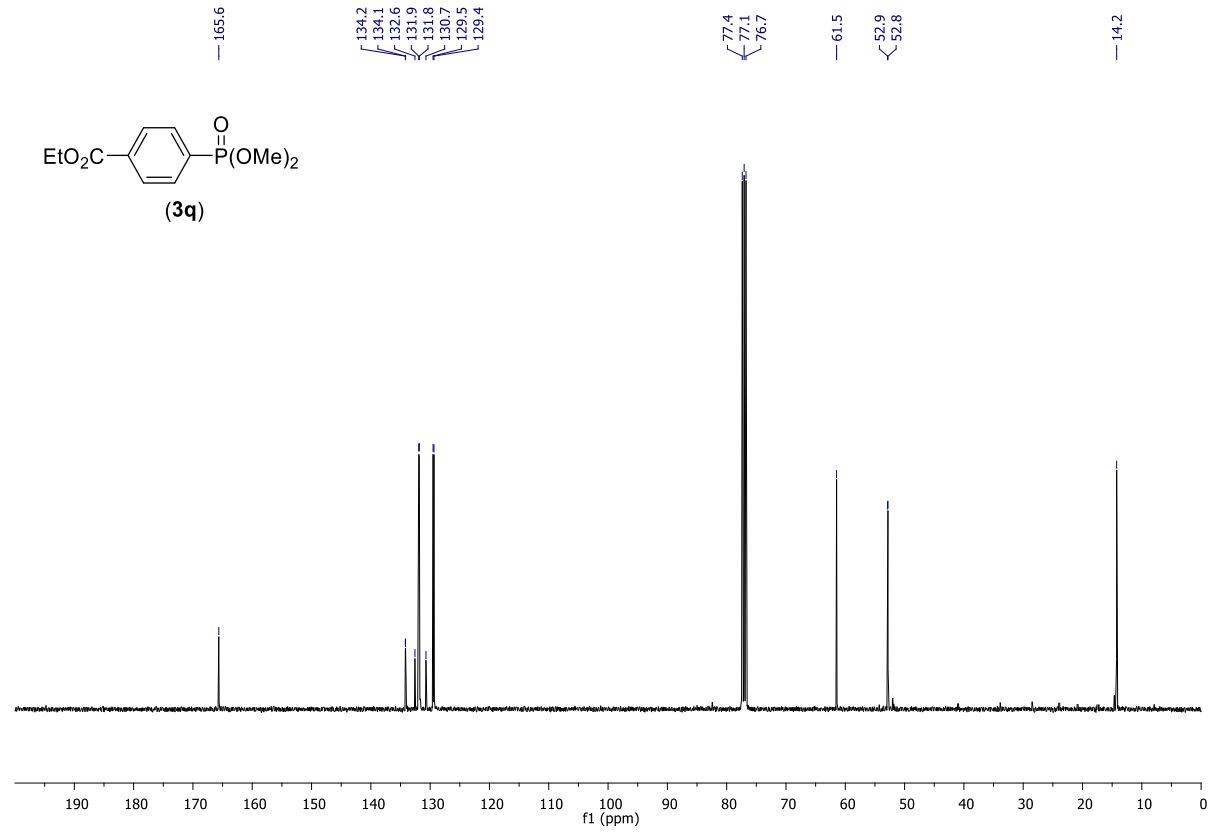

8.22
8.21
8.20
8.19
8.17
8.16
7.66
7.65
7.65
7.64
3.80
3.77
O
P(OMe)2
CF3
(3p)
1.00
1.02
2.10
6.30
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

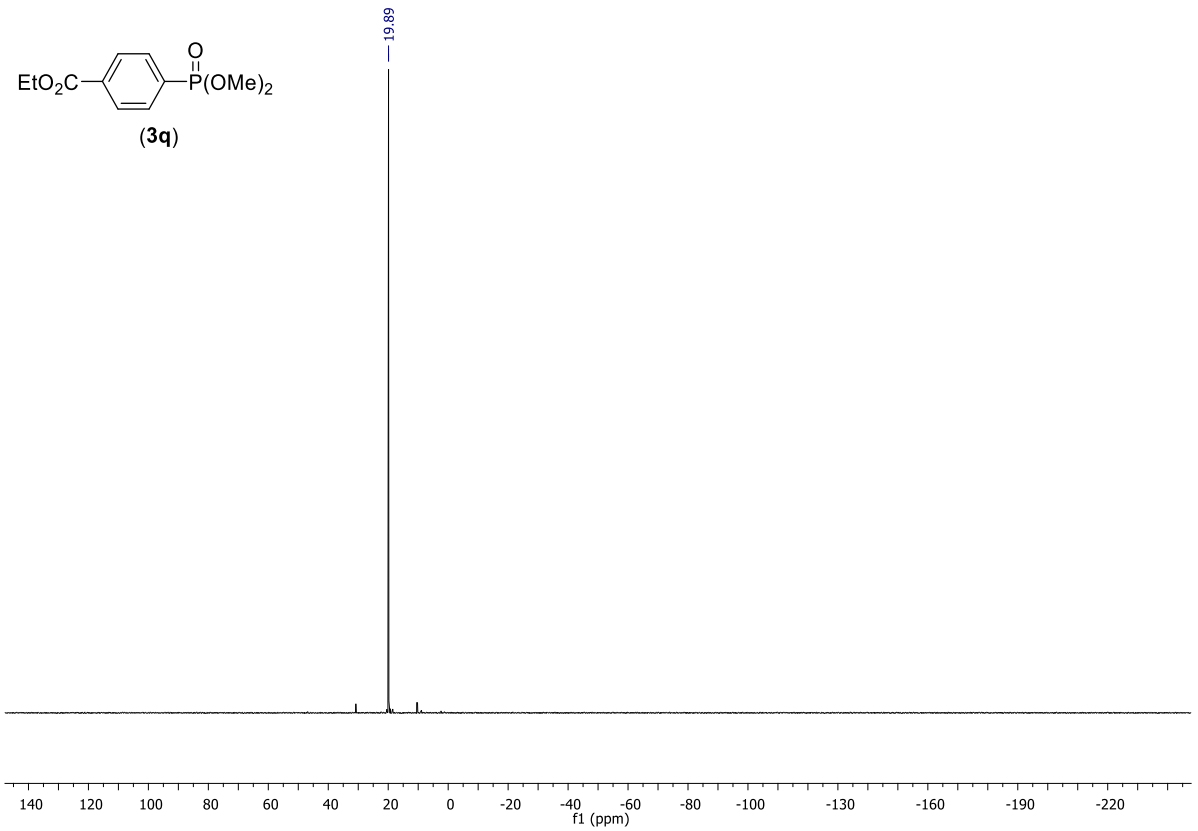

136.3
136.2
132.6
132.6
132.6
131.5
131.4
127.5
127.5
127.4
127.4
127.3
127.3
126.7
124.8
124.6
124.5
121.9
121.8
77.4
77.1
76.7
53.1
53.1
O
P(OMe)2
CF3
(3p)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

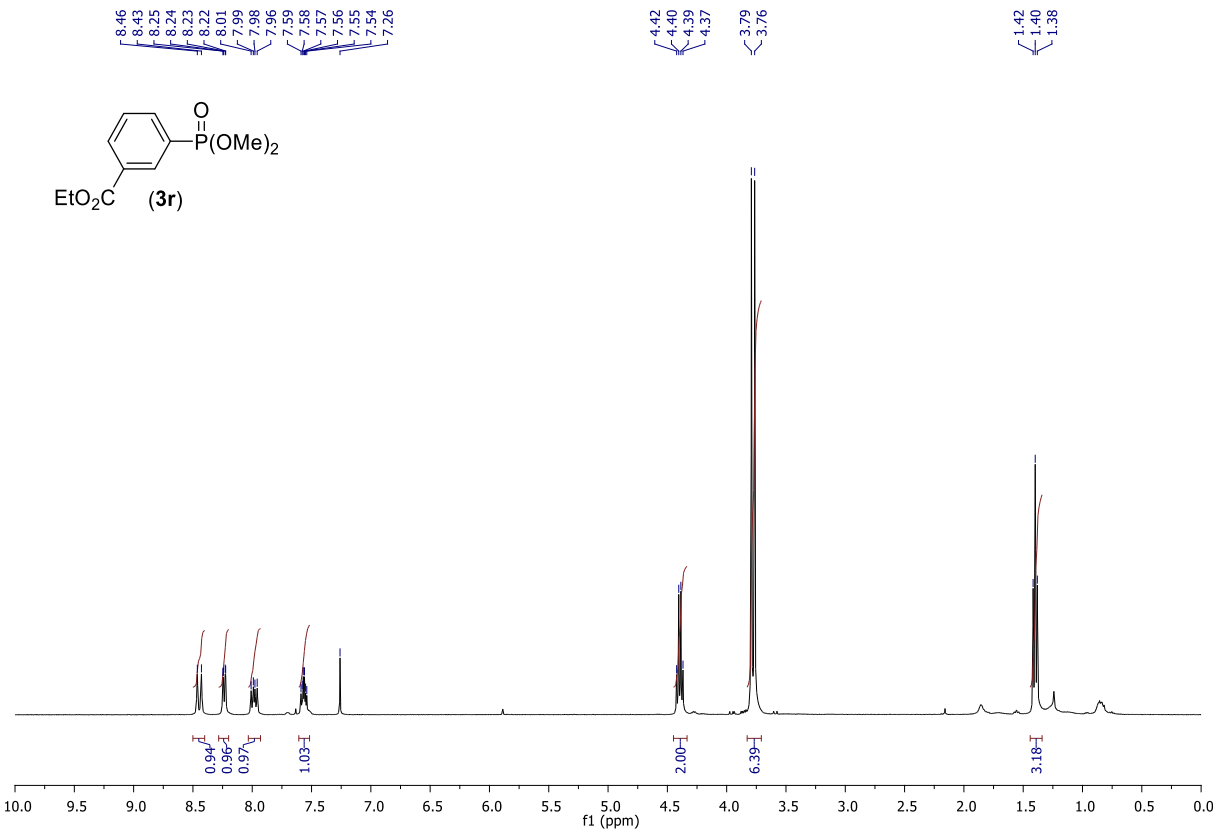

-59.42
-59.43
O
P(OMe)2
CF3
(3p)
100
50
0
-50
-100
-150
-200
-250
-300
f1 (ppm)

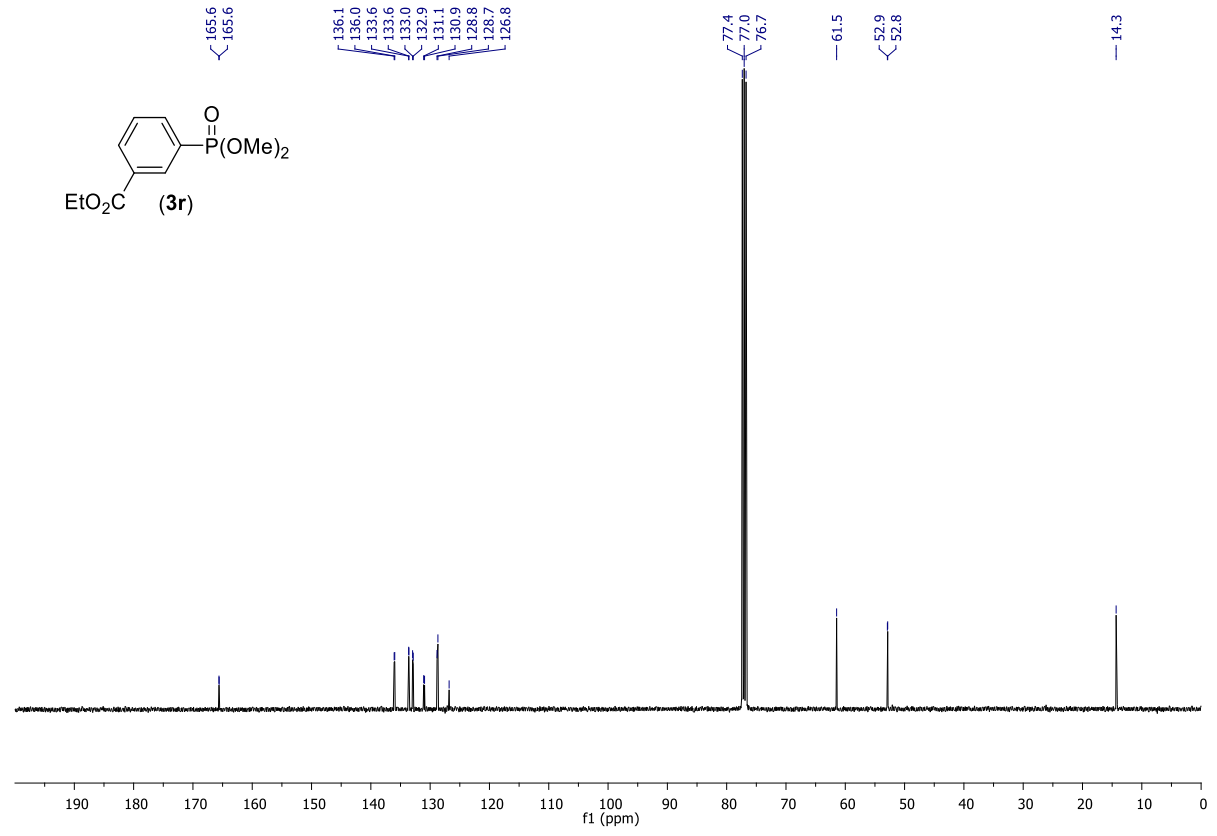

17.55
O
P(OMe)2
CF3
(3p)
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)

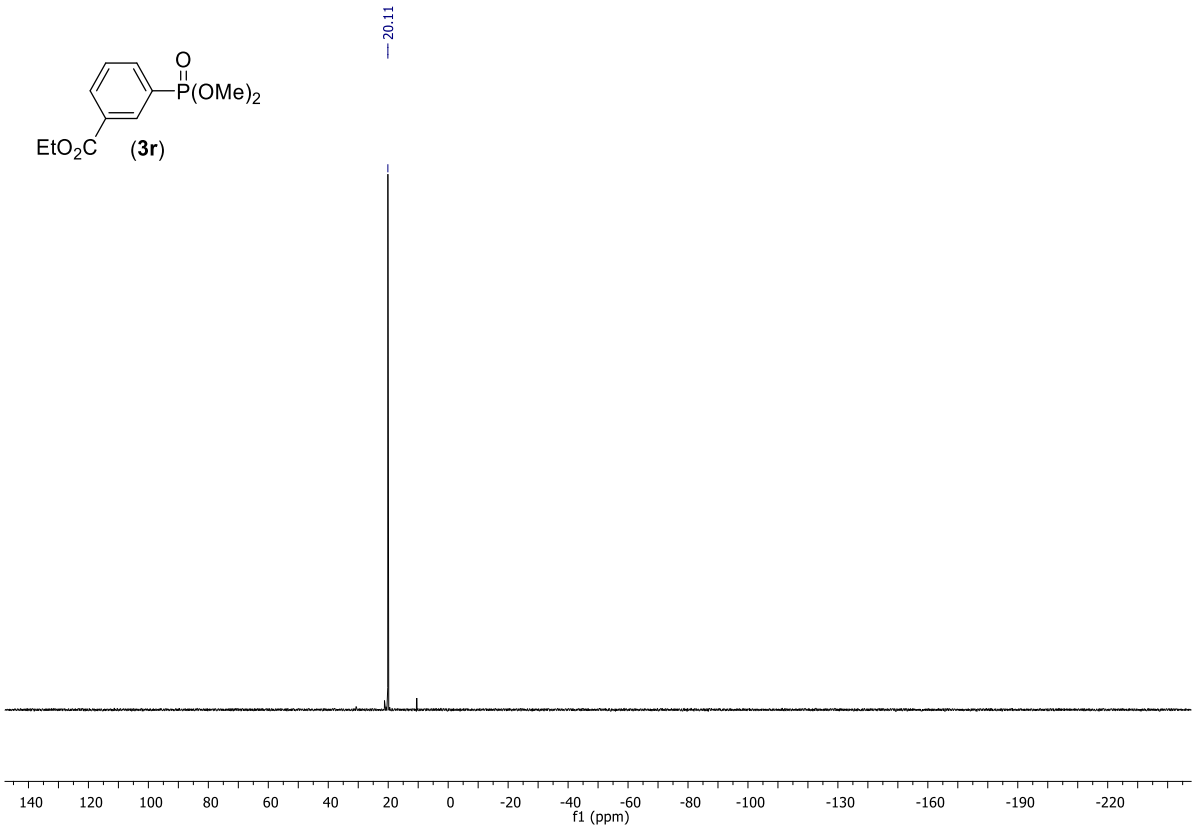

8.12
8.11
8.10
8.09
7.88
7.86
7.84
7.82
4.41
4.39
4.37
4.35
3.77
3.74
1.40
1.38
1.36
EtO2C
O
P(OMe)2
(3q)
1.97
2.00
2.07
6.43
3.16
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

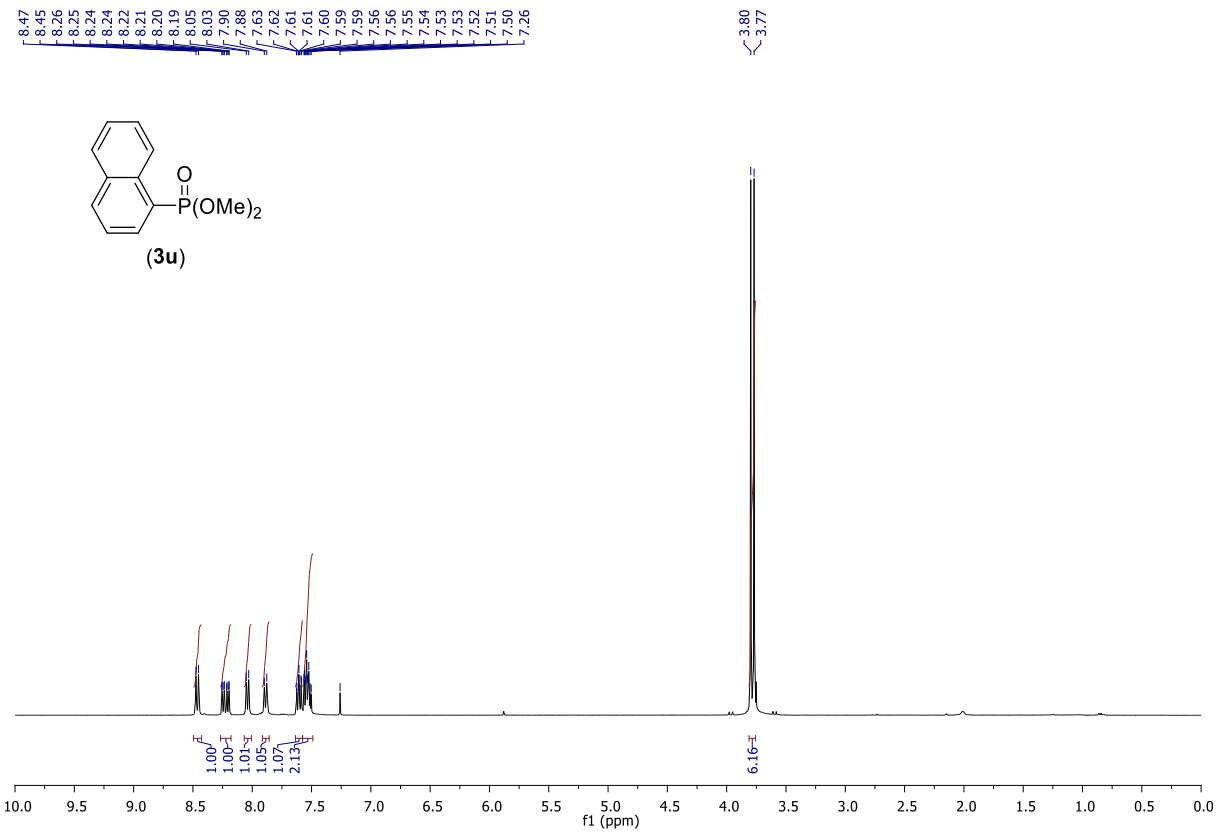

165.6
134.2
134.1
132.6
131.9
131.8
130.7
129.5
129.4
77.4
77.1
76.7
61.5
52.9
52.8
14.2
EtO2C
O
P(OMe)2
(3q)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


19.89
EtO2C
O
P(OMe)2
(3q)
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)

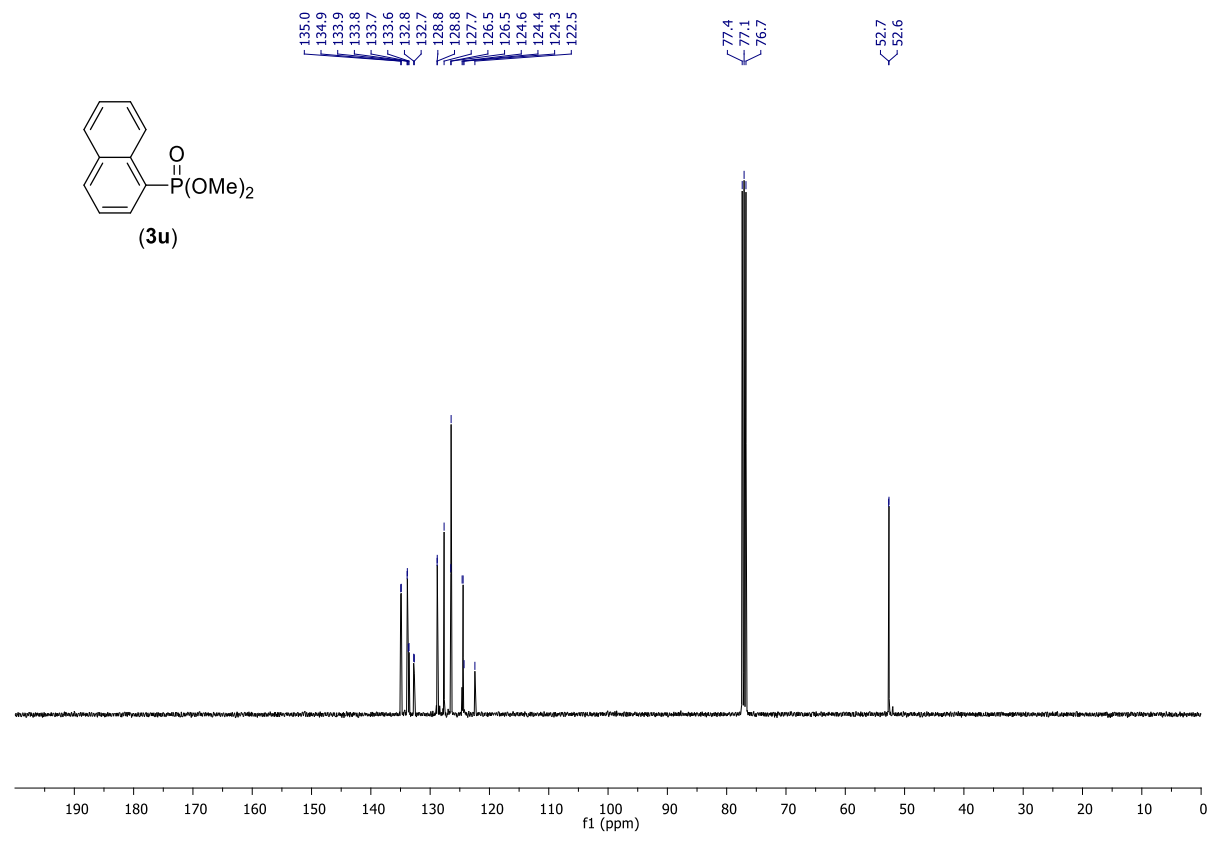

8.46
8.43
8.25
8.24
8.23
8.22
8.01
7.99
7.98
7.96
7.59
7.58
7.57
7.56
7.55
7.54
7.26
4.42
4.40
4.39
4.37
3.79
3.76
1.42
1.40
1.38
O
P(OMe)2
EtO2C
(3r)
0.94
0.96
0.97
1.03
2.00
6.39
3.18
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


165.6
165.6
136.1
136.0
133.6
133.6
133.0
132.9
131.1
130.9
128.8
128.7
126.8
77.4
77.0
76.7
61.5
52.9
52.8
14.3
O
P(OMe)2
EtO2C
(3r)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

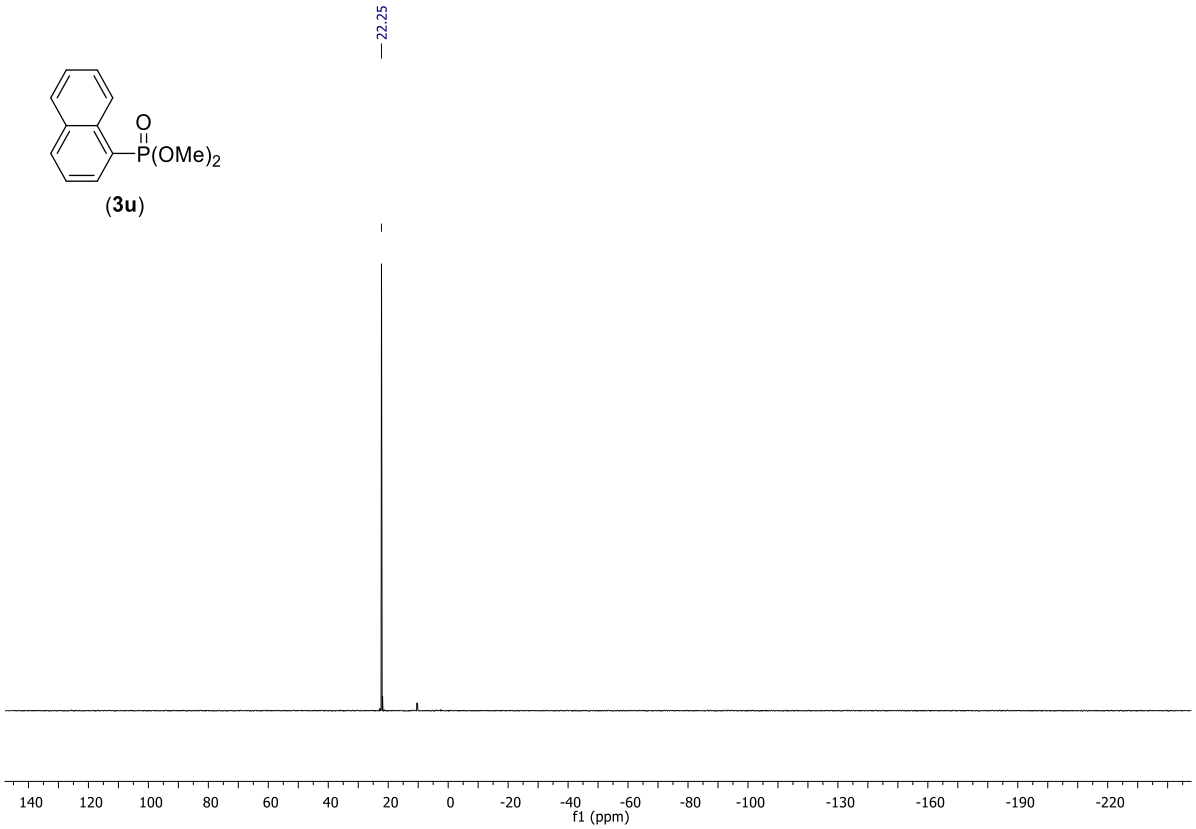

20.11
O
P(OMe)2
EtO2C
(3r)
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)


8.47
8.45
8.26
8.25
8.24
8.24
8.22
8.21
8.20
8.19
8.05
8.03
7.90
7.88
7.63
7.62
7.61
7.61
7.60
7.59
7.59
7.56
7.56
7.55
7.54
7.53
7.53
7.52
7.51
7.50
7.26
3.80
3.77
O
P(OMe)2
1.00
1.00
1.01
1.05
1.07
2.13
6.16
f1 (ppm)

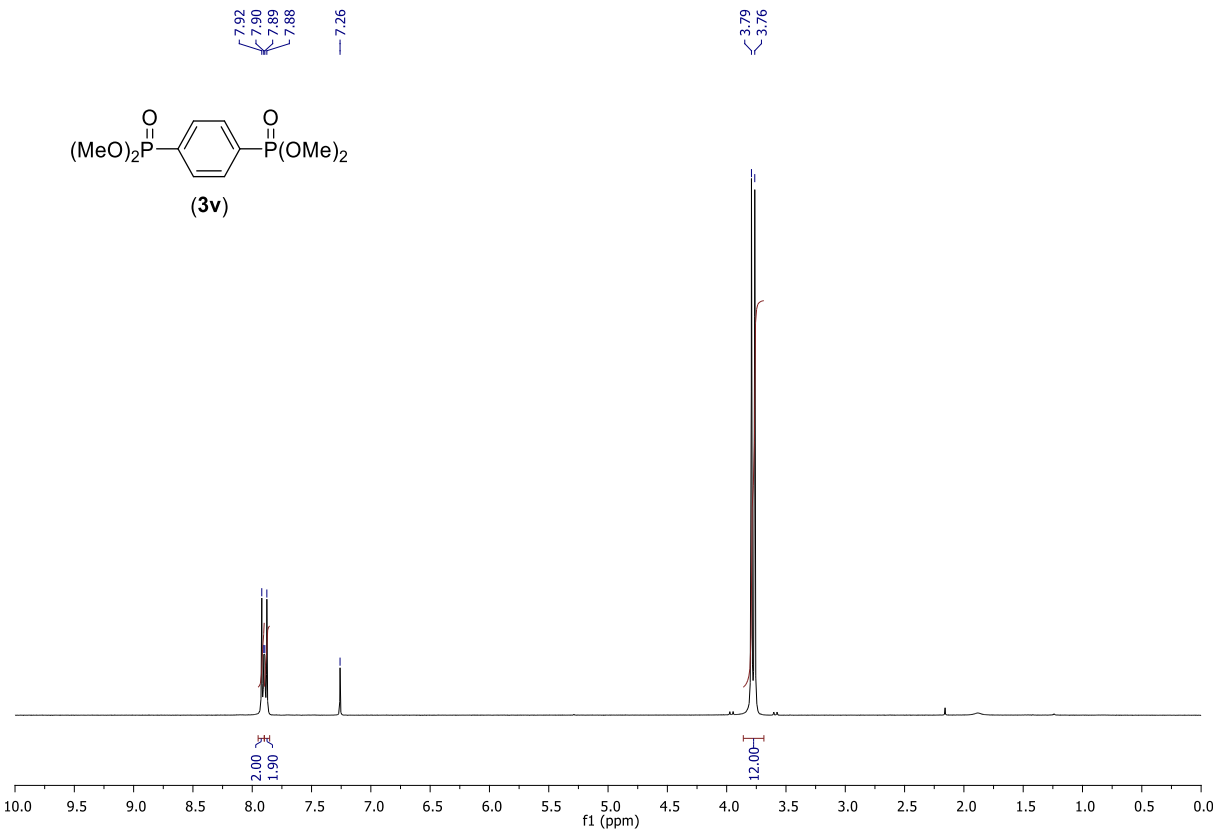
(**3u**)

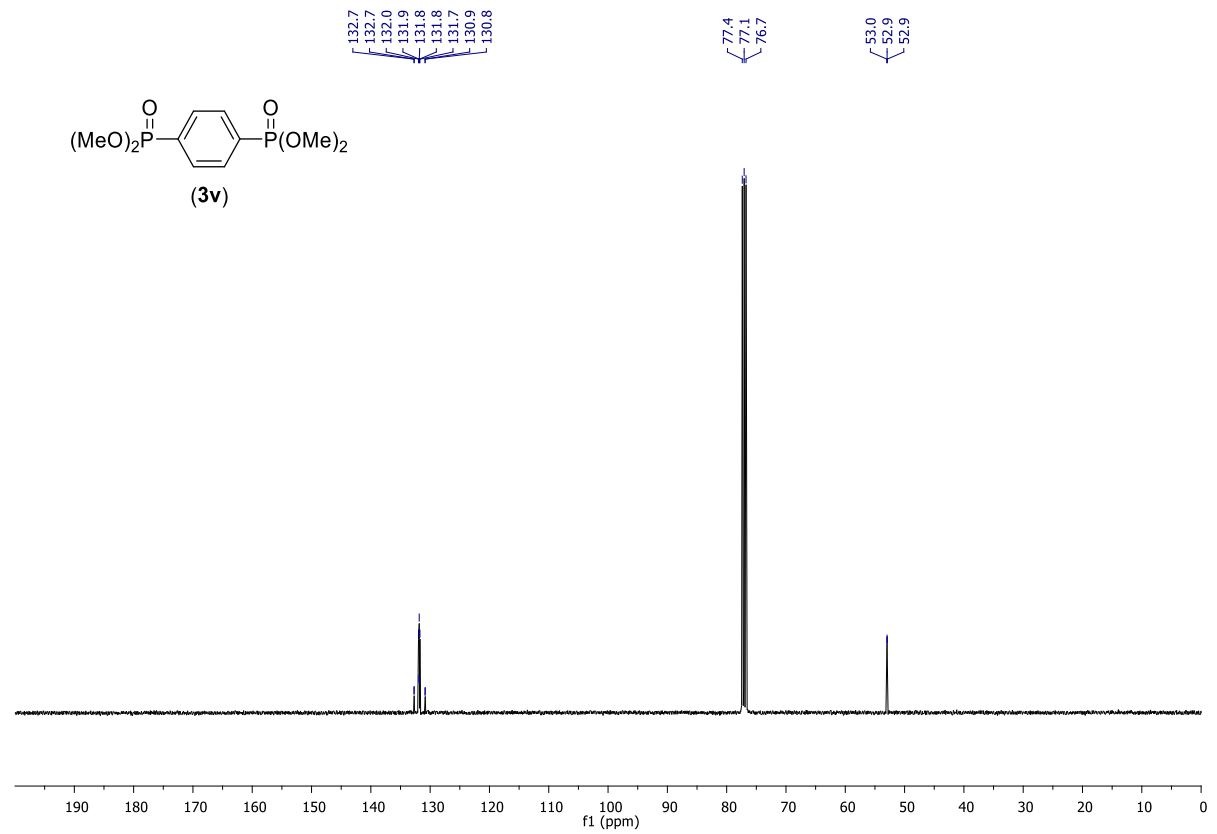

135.0
134.9
133.9
133.8
133.7
133.6
132.8
132.7
128.8
128.8
127.7
126.5
126.5
124.6
124.4
124.3
122.5
77.4
77.1
76.7
52.7
52.6
O
P(OMe)2
f1 (ppm)

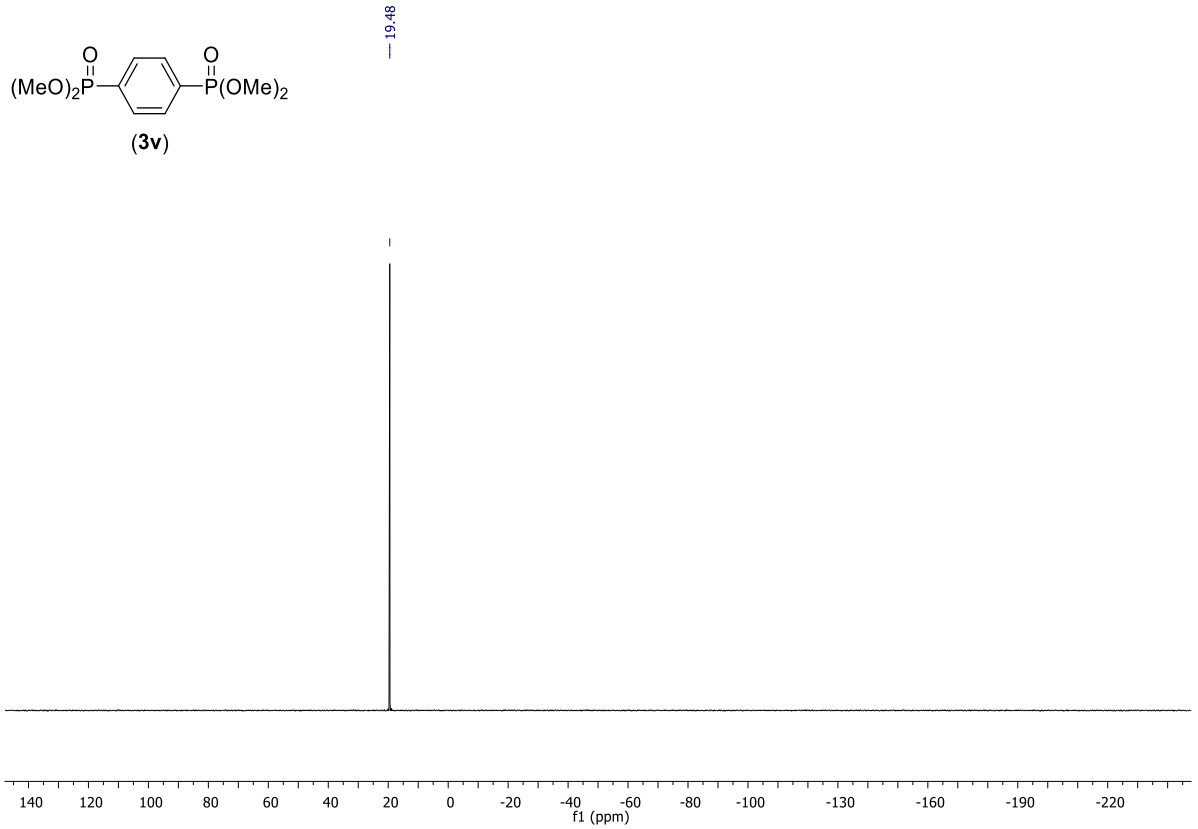
(**3u**)

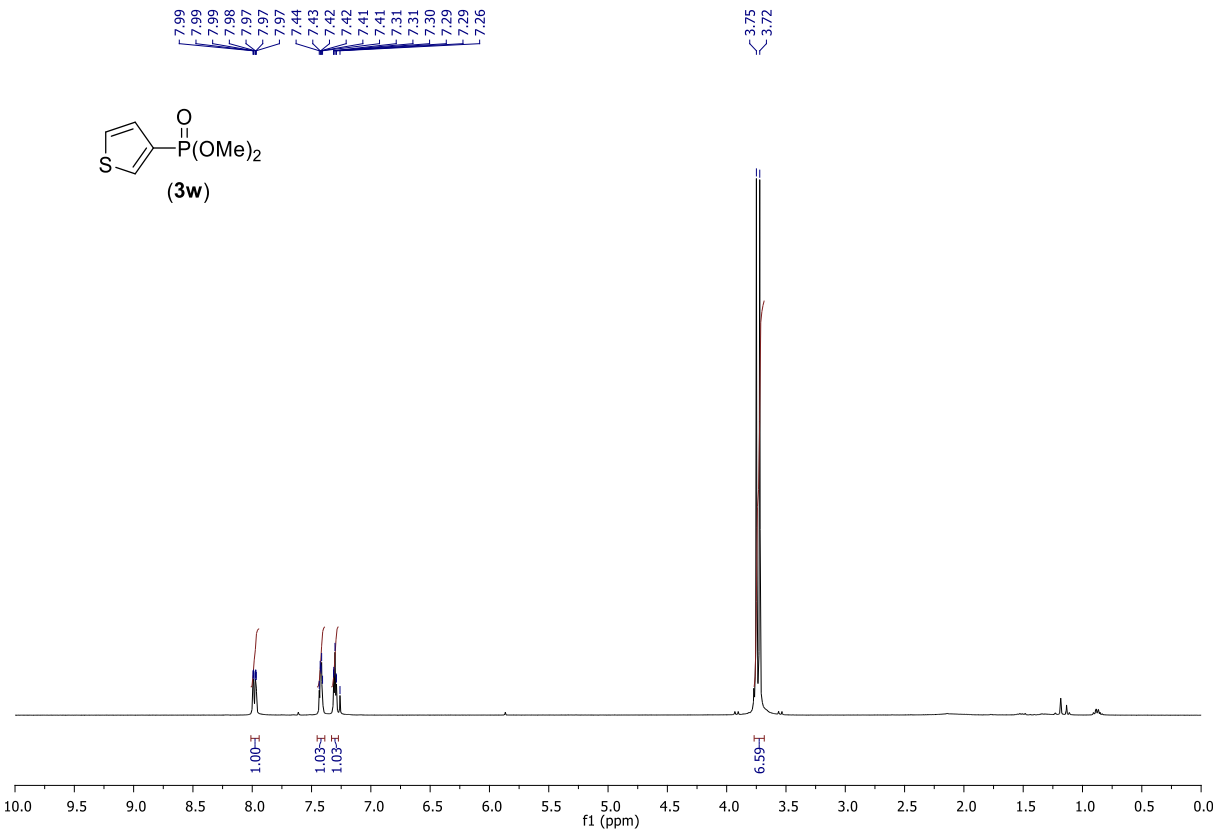

22.25
O
P(OMe)2
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)

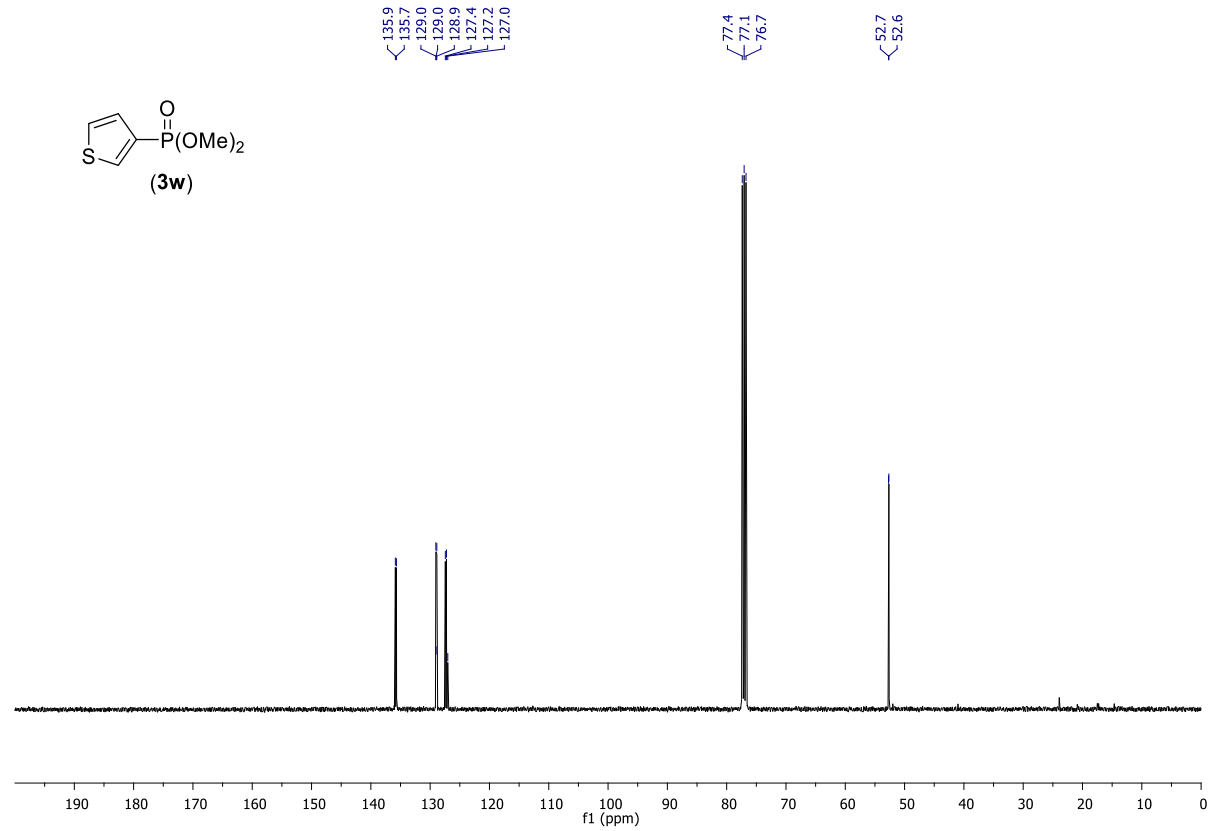
(**3u**)

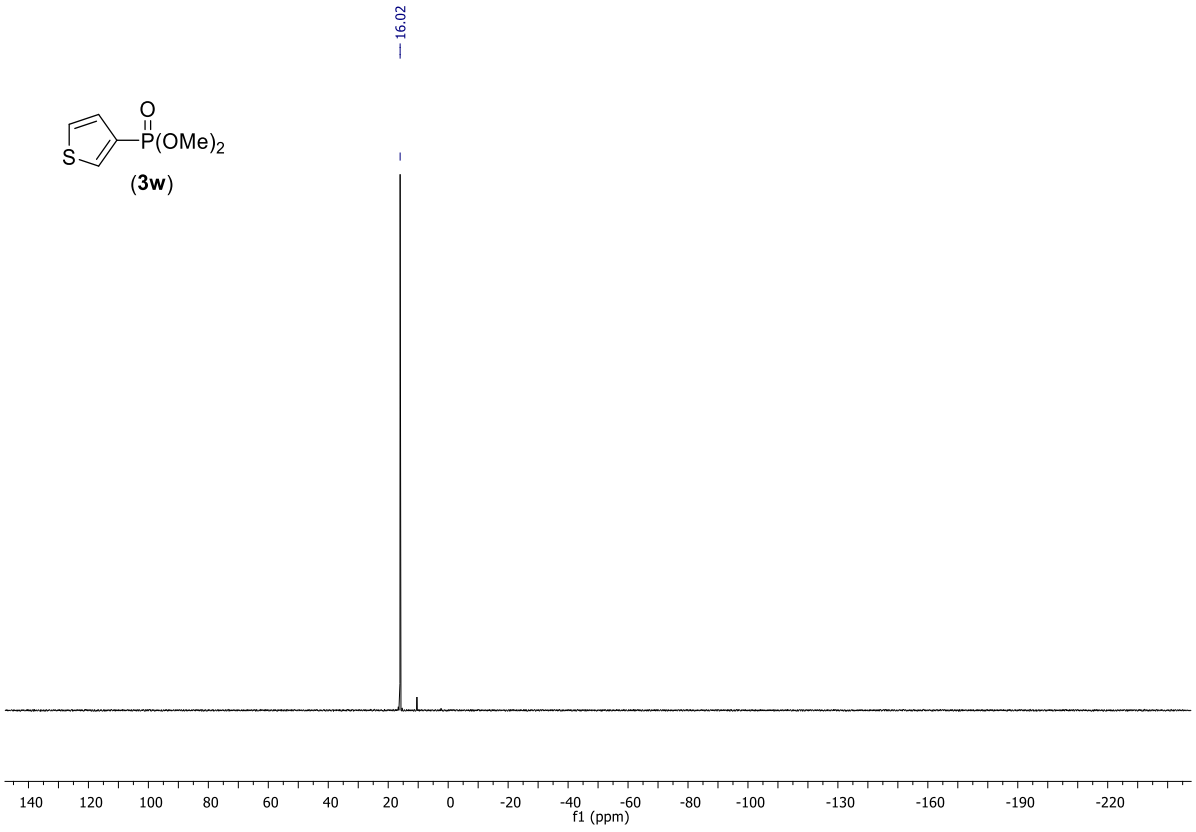

7.92
7.90
7.89
7.88
7.26
3.79
3.76
(MeO)2P
P(OMe)2
(3v)
2.00
1.90
12.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

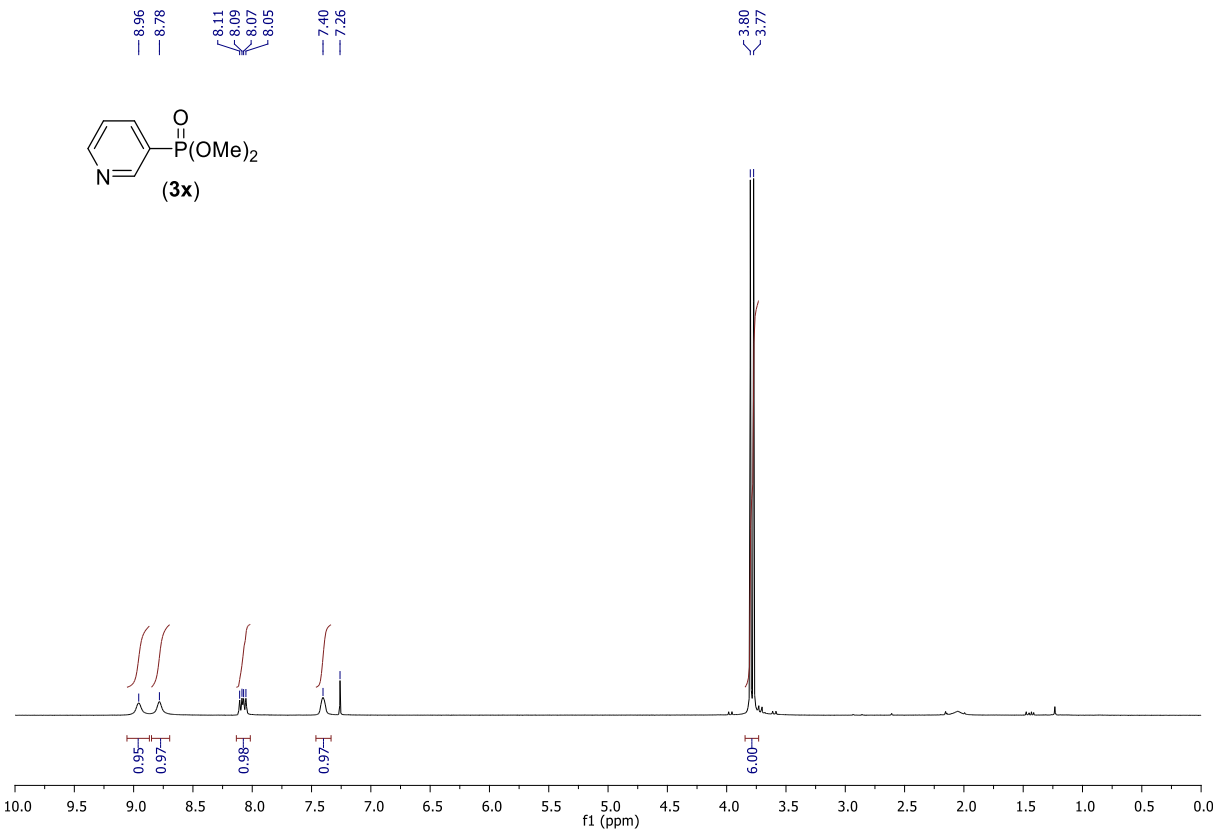

132.7
132.7
132.0
131.9
131.8
131.8
131.7
130.9
130.8
77.4
77.1
76.7
53.0
52.9
52.9
(MeO)2P
P(OMe)2
(3v)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

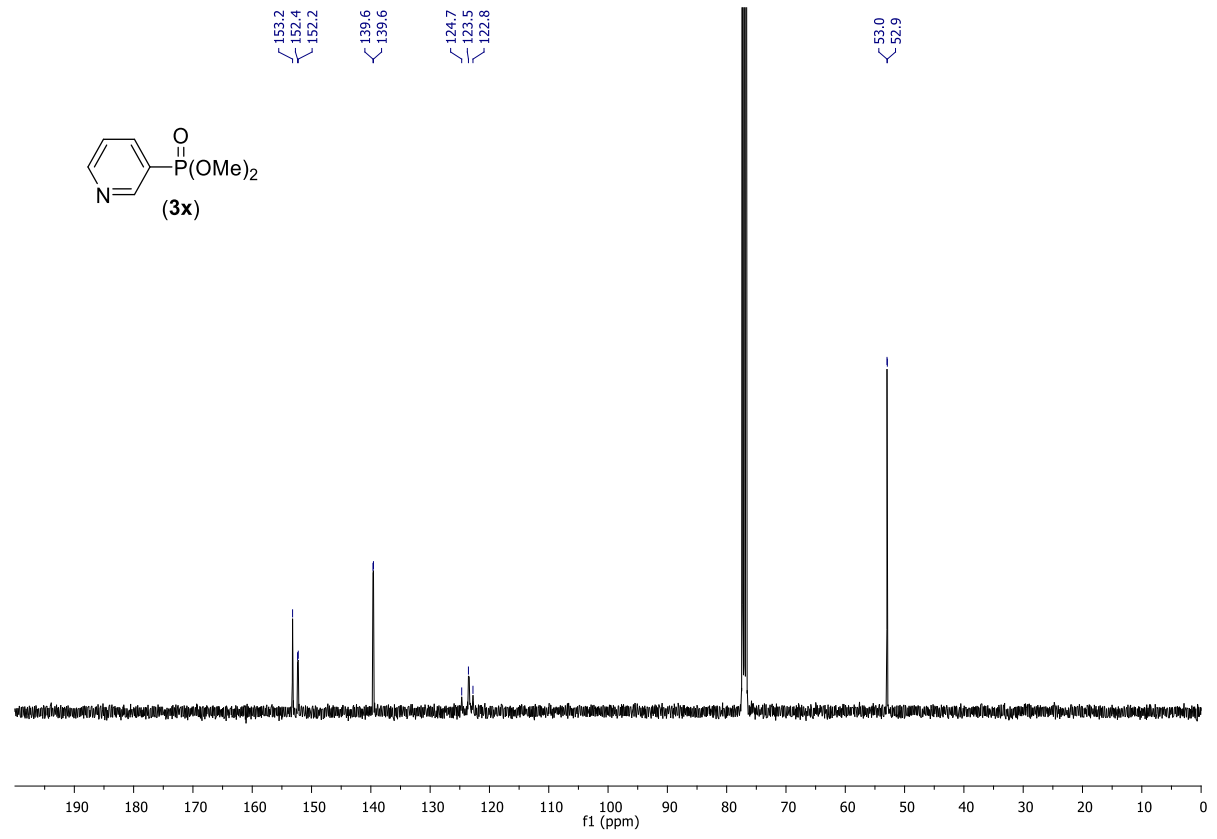

19.48
(MeO)2P
P(OMe)2
O
O
(3v)
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)

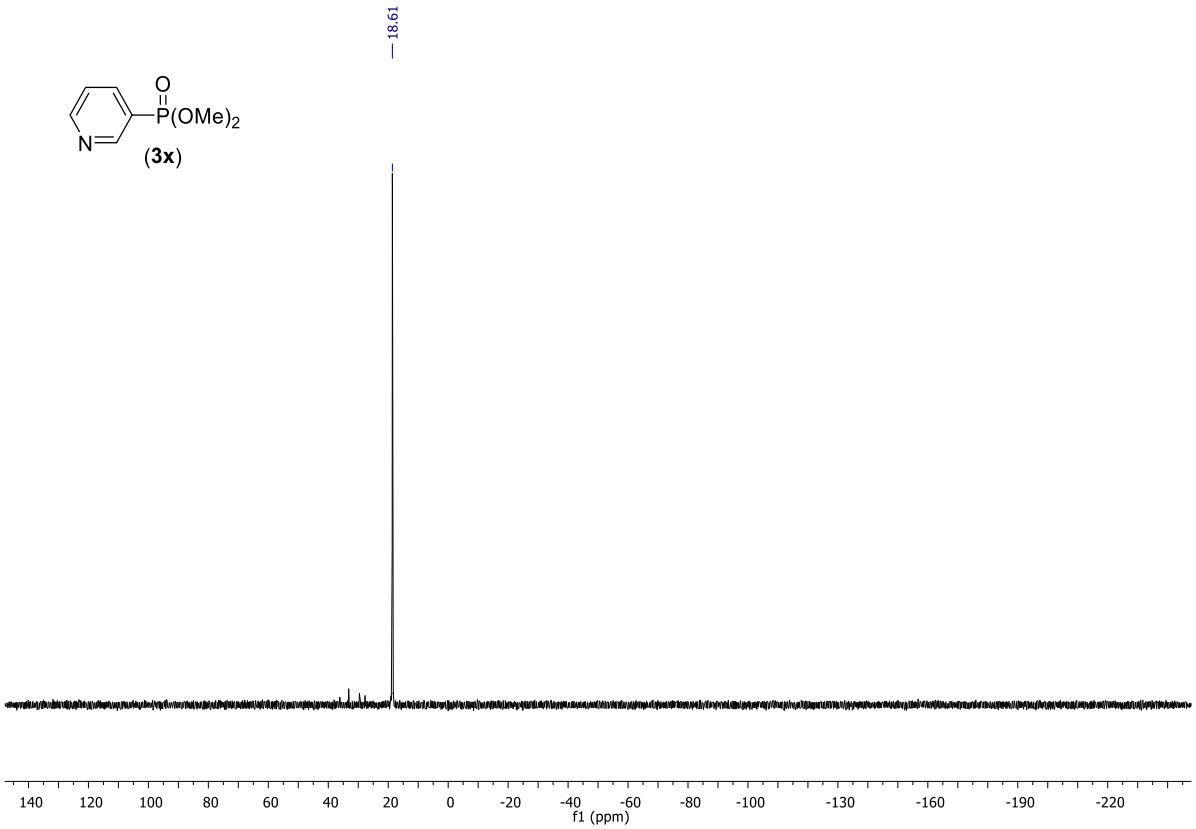

7.99
7.99
7.99
7.98
7.97
7.97
7.97
7.44
7.43
7.42
7.42
7.41
7.41
7.31
7.31
7.30
7.29
7.29
7.26
3.75
3.72
O
P(OMe)2
S
(3w)
1.00
1.03
1.03
6.59
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

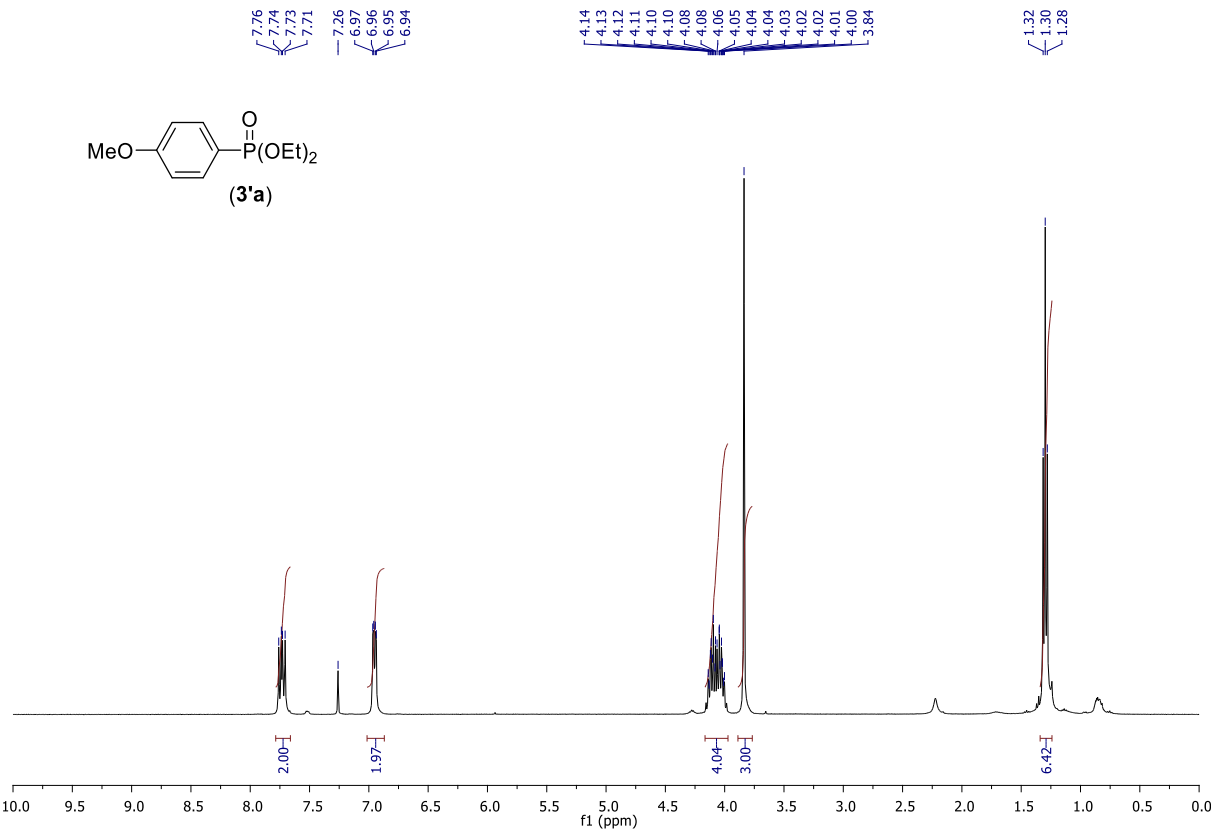

135.9
135.7
129.0
129.0
128.9
127.4
127.2
127.0
77.4
77.1
76.7
52.7
52.6
O
P(OMe)2
S
(3w)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

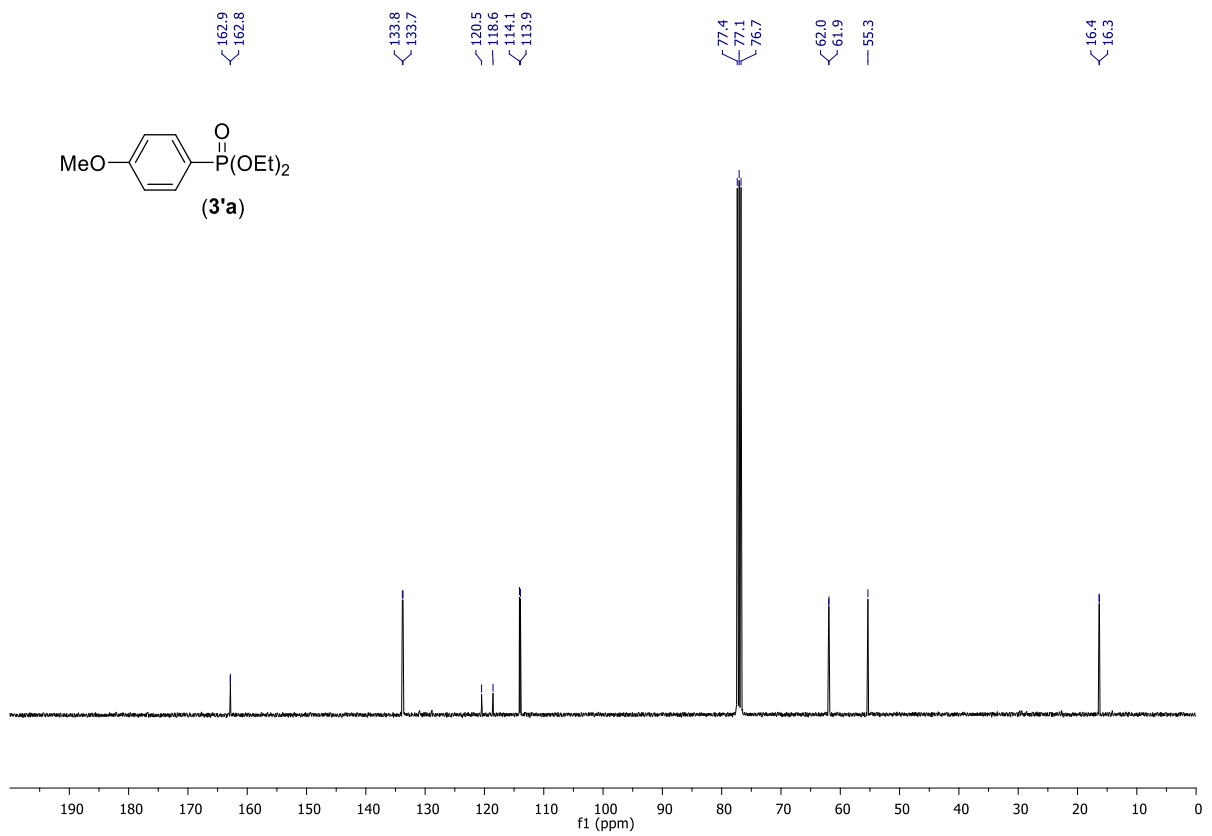

16.02
O
P(OMe)2
S
(3w)
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)

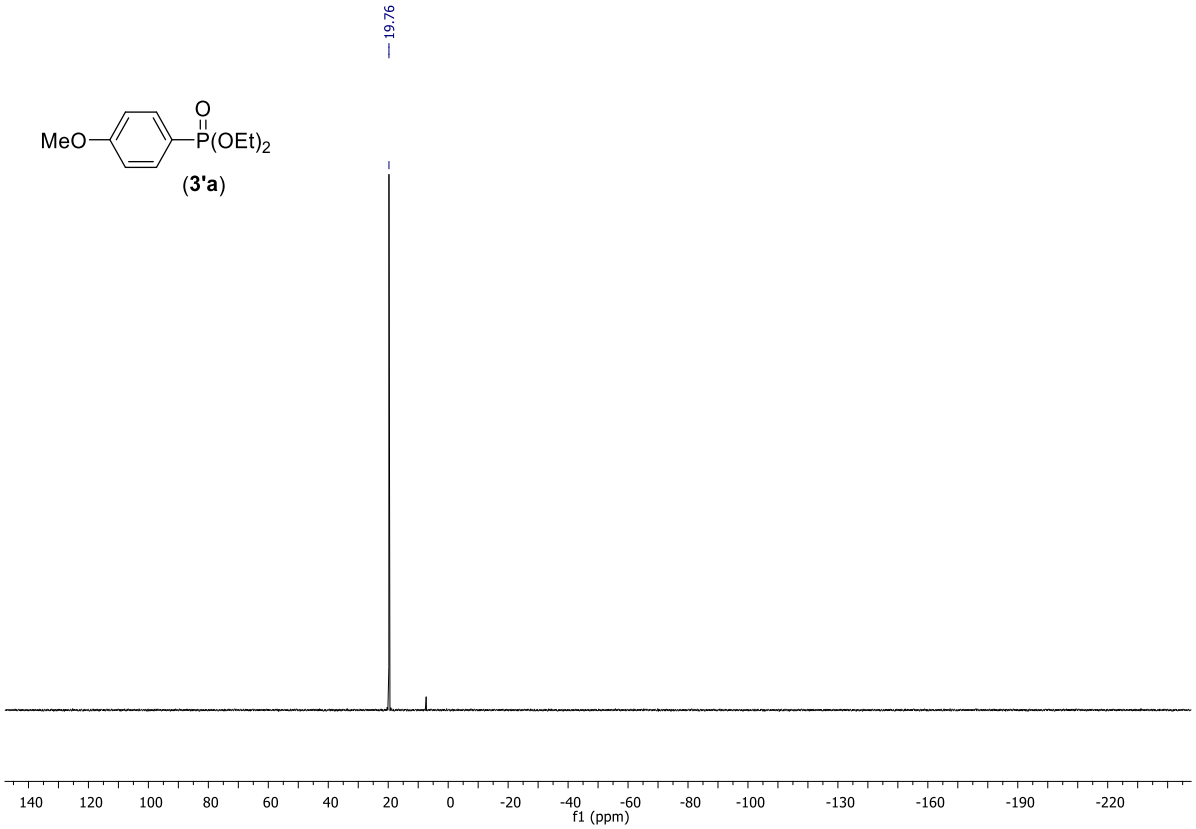

8.96
8.78
8.11
8.09
8.07
8.05
7.40
7.26
3.80
3.77
O
P(OMe)2
N
(3x)
0.95
0.97
0.98
0.97
6.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

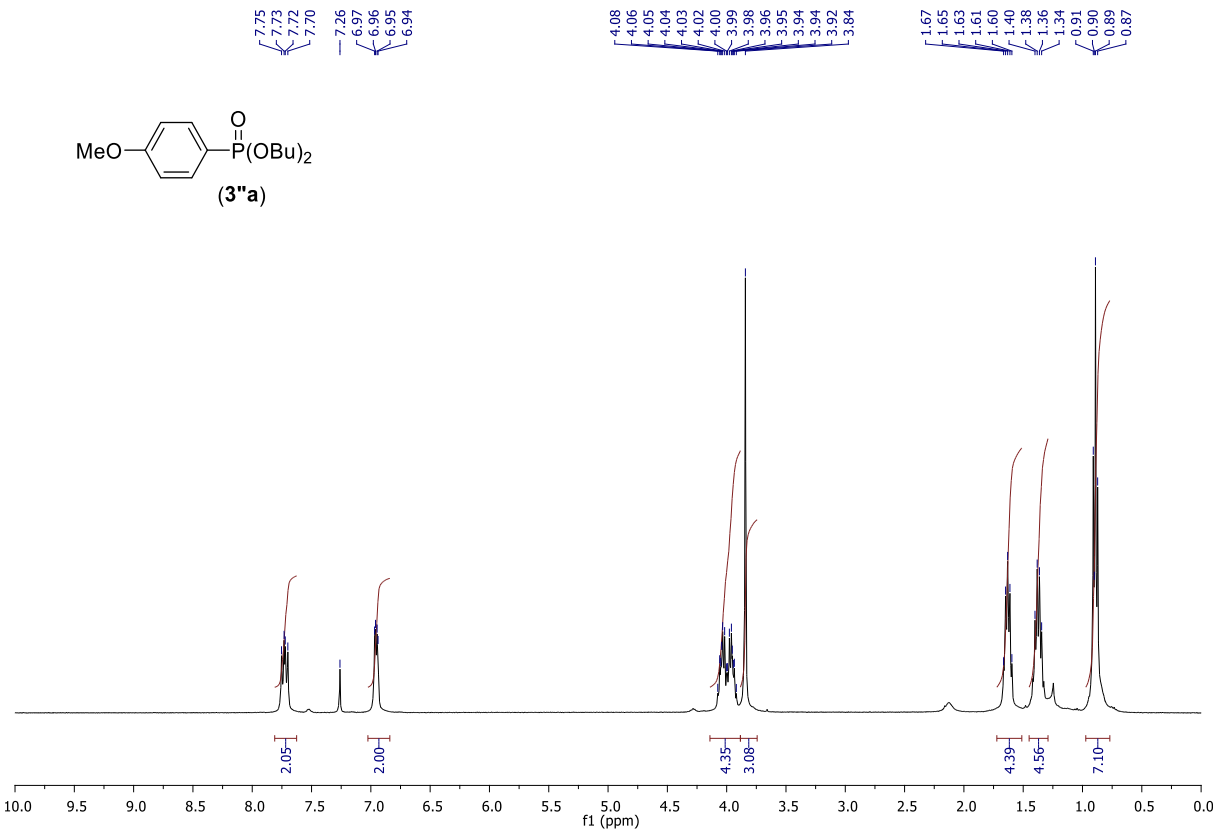

153.2
152.4
152.2
139.6
139.6
124.7
123.5
122.8
53.0
52.9
O
P(OMe)2
N
(3x)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

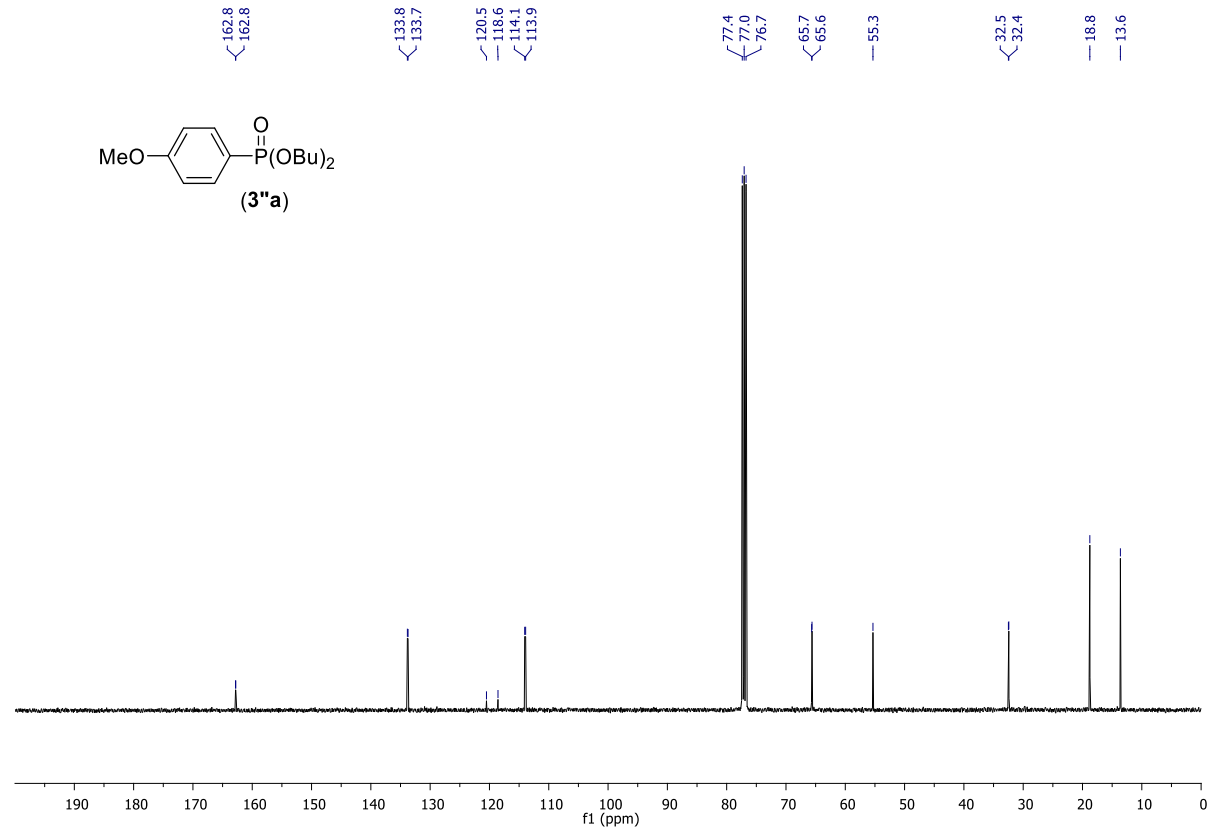

18.61
O
P(OMe)2
N
(3x)
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)

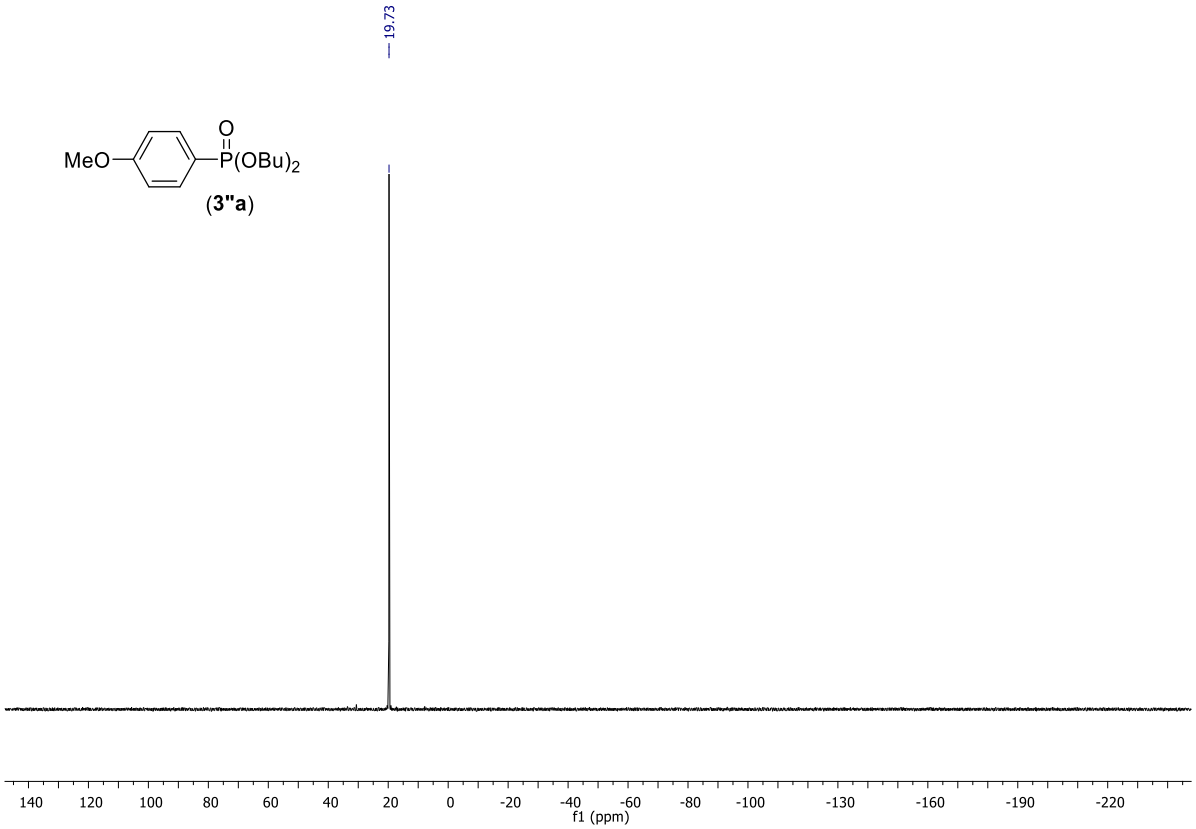

7.76
7.74
7.73
7.71
7.26
6.97
6.96
6.95
6.94
4.14
4.13
4.12
4.11
4.10
4.10
4.08
4.08
4.06
4.05
4.04
4.04
4.03
4.02
4.02
4.01
4.00
3.84
1.32
1.30
1.28
MeO
O
P(OEt)2
(3'a)
2.00
1.97
4.04
3.00
6.42
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

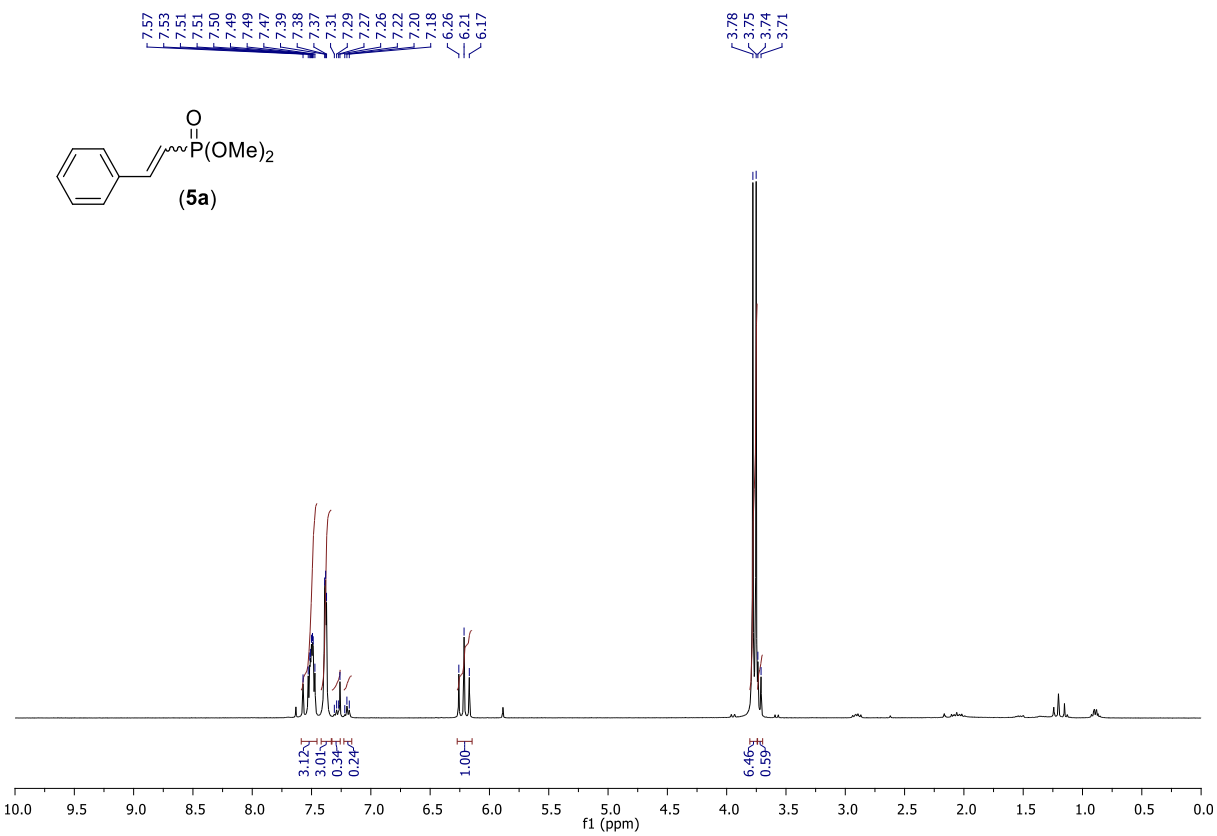

162.9
162.8
133.8
133.7
120.5
118.6
114.1
113.9
77.4
77.1
76.7
62.0
61.9
55.3
16.4
16.3
MeO
O
P(OEt)2
(3'a)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

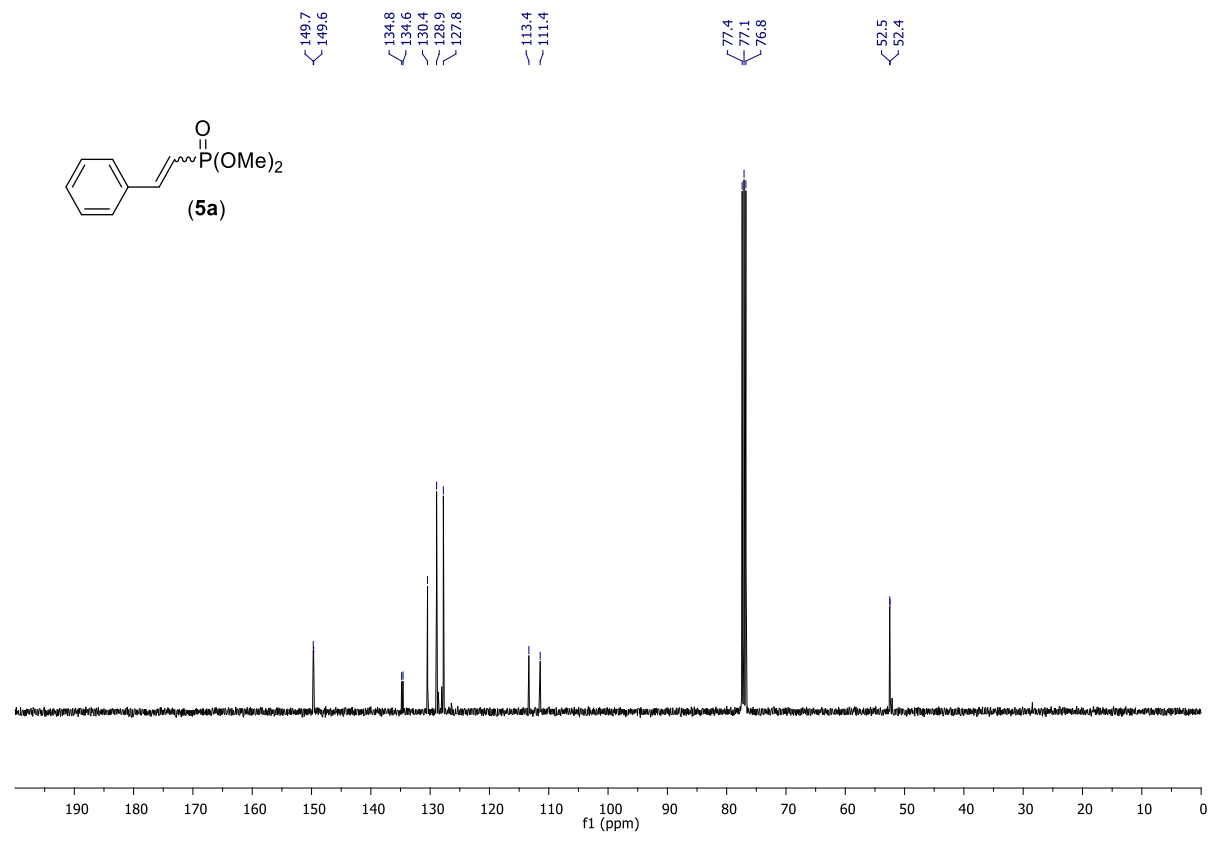

19.76
MeO
O
P(OEt)2
(3'a)
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)

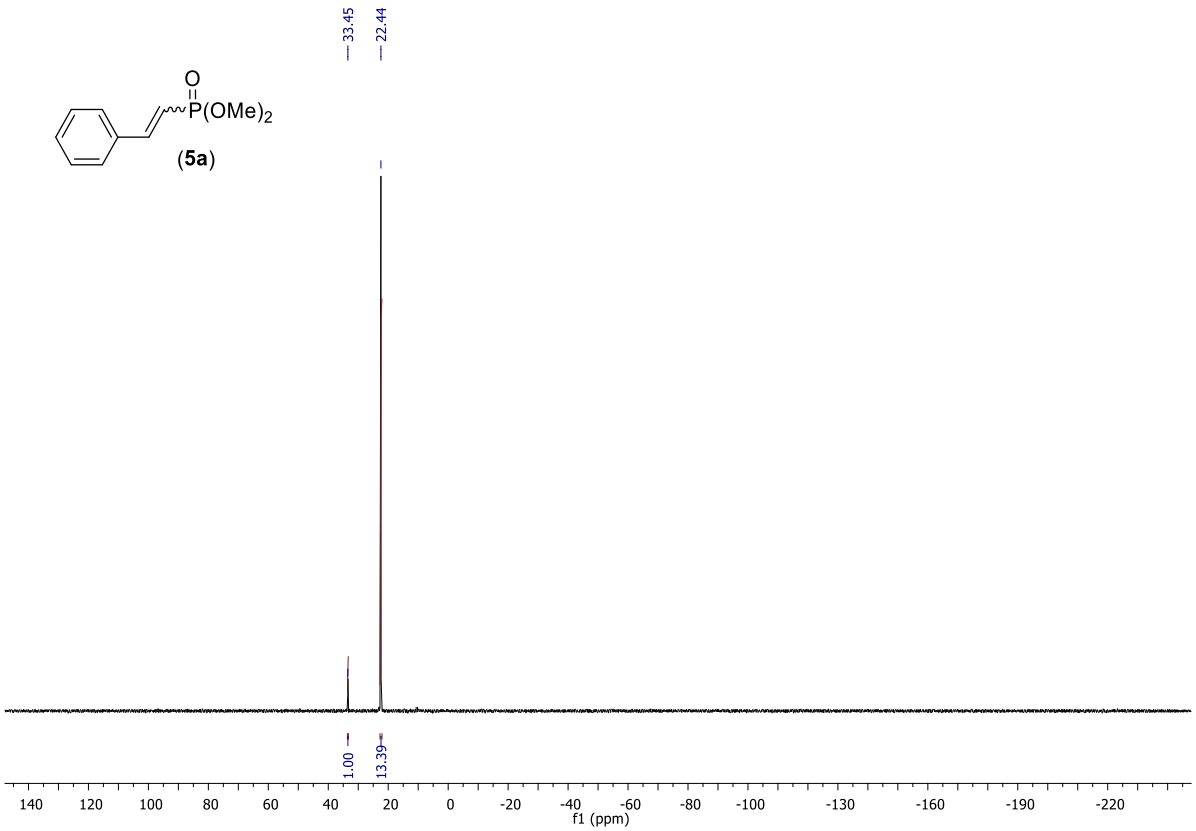

7.75
7.73
7.72
7.70
7.26
6.97
6.96
6.95
6.94
4.08
4.06
4.05
4.04
4.03
4.02
4.00
3.99
3.98
3.96
3.95
3.94
3.94
3.92
3.84
1.67
1.65
1.63
1.61
1.60
1.40
1.38
1.36
1.34
0.91
0.90
0.89
0.87
MeO
O
P(OBu)2
(3"a)
2.05
2.00
4.35
3.08
4.39
4.56
7.10
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

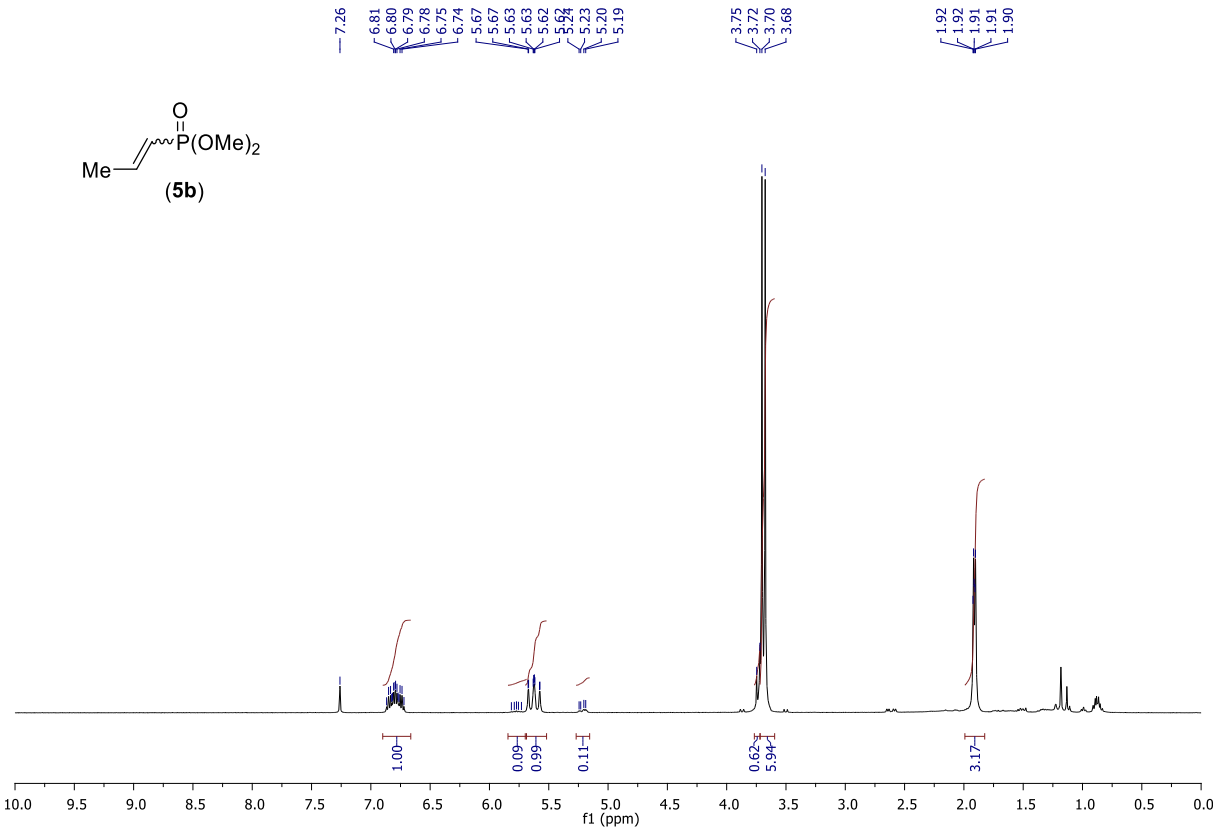

162.8
162.8
133.8
133.7
120.5
118.6
114.1
113.9
77.4
77.0
76.7
65.7
65.6
55.3
32.5
32.4
18.8
13.6
MeO
O
P(OBu)2
(3"a)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

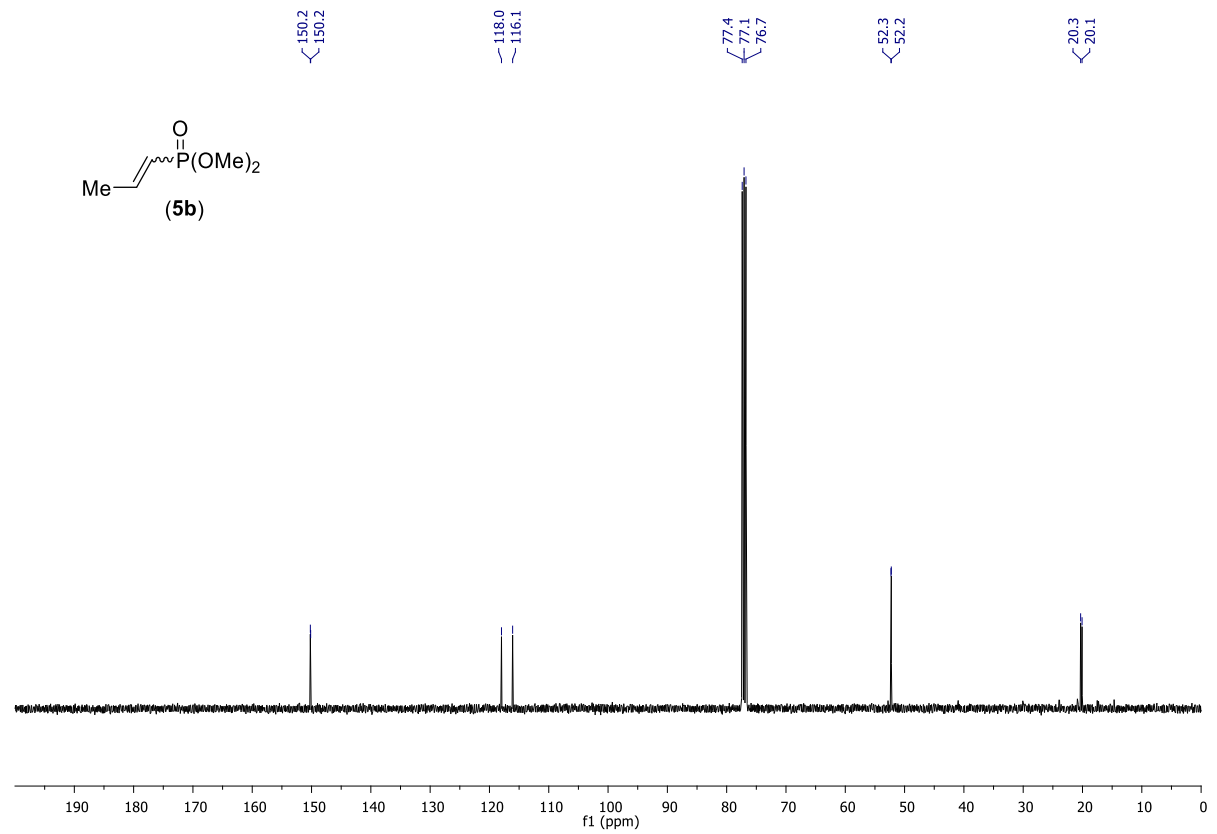

19.73
MeO
O
P(OBu)2
(3"a)
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)

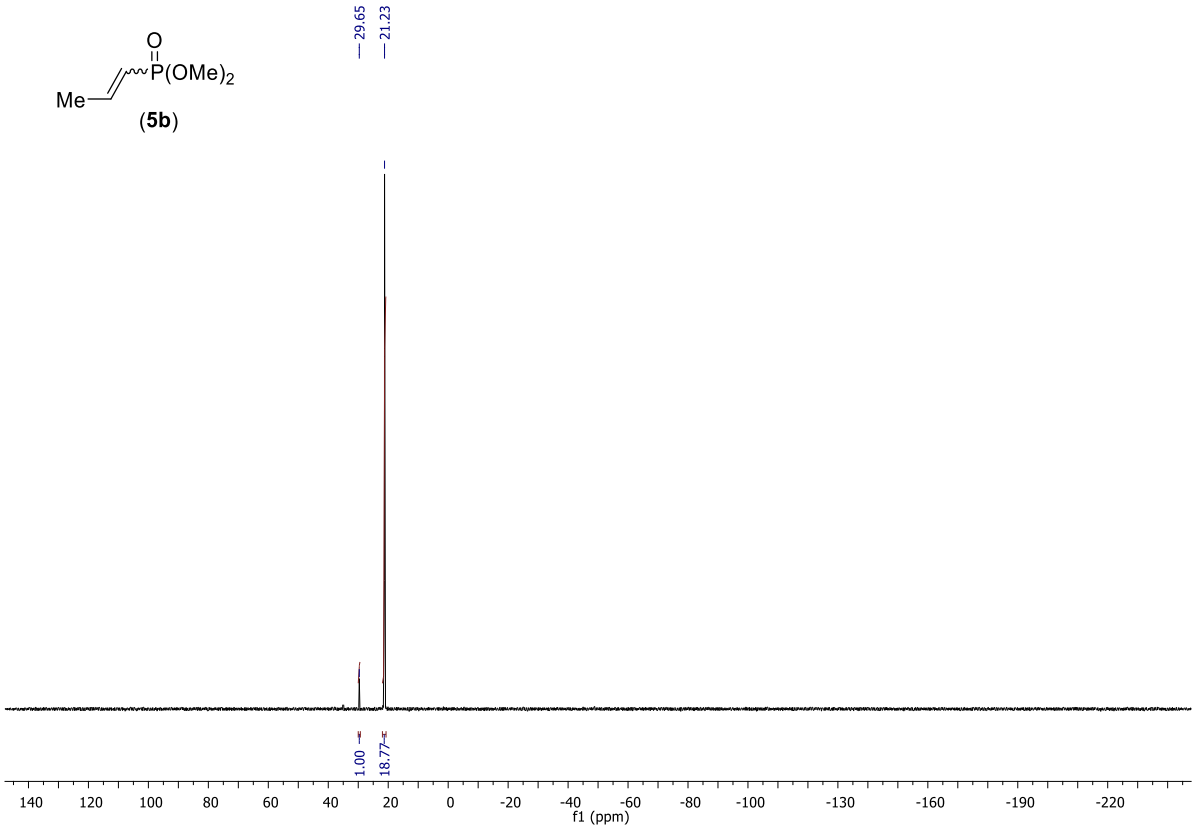

7.57
7.53
7.51
7.51
7.50
7.49
7.49
7.47
7.39
7.38
7.37
7.31
7.29
7.27
7.26
7.22
7.20
7.18
6.26
6.21
6.17
3.78
3.75
3.74
3.71
O
P(OMe)2
(5a)
3.12
3.01
0.34
0.24
1.00
6.46
0.59
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


149.7
149.6
134.8
134.6
130.4
128.9
127.8
113.4
111.4
77.4
77.1
76.8
52.5
52.4
O
P(OMe)2
(5a)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


33.45
22.44
O
P(OMe)2
(5a)
1.00
13.39
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)


7.26
6.81
6.80
6.79
6.78
6.75
6.74
5.67
5.63
5.63
5.62
5.23
5.20
5.19
3.75
3.72
3.70
3.68
1.92
1.92
1.91
1.91
1.90
O
P(OMe)2
Me
(5b)
1.00
0.09
0.99
0.11
0.62
5.94
3.17
10.0 9.5 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 0.0
f1 (ppm)


150.2
150.2
118.0
116.1
77.4
77.1
76.7
52.3
52.2
20.3
20.1
O
P(OMe)2
Me
(5b)
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


O
P(OMe)2
Me
(5b)
29.65
21.23
1.00
18.77
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220
f1 (ppm)