Supporting Information

Iron-Catalyzed Cross Coupling of P-H/C-O Bonds: Efficient Synthesis of *α*-Alkoxyphosphorus Compounds

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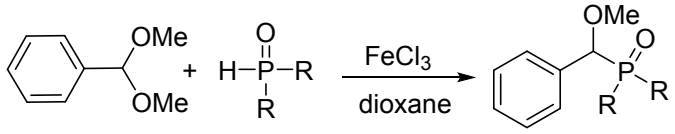
General information

All reactions were carried out in oven-dried Schlenk tubes under N_2 atmosphere. Dry solvents were obtained by purification according to standard methods. Reagents were used as received unless otherwise noted. ^{1}H NMR, ^{13}C NMR and ^{31}P NMR data were obtained on a Bruker-400 spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C, and 162 MHz for ^{31}P NMR spectroscopy). Data are report as follows: Chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q= quartet, m = multiplet), Coupling constants (J) are reported in hertz. Mass spectra were measured on a Shimadzu GCMS-QP2010 Plus spectrometer (EI).

General procedure for the synthesis of phosphine oxides

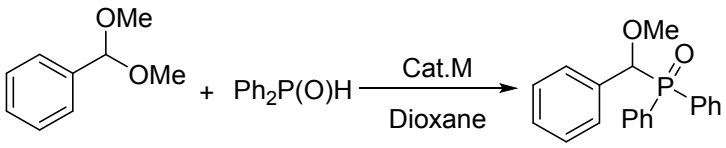


Procedure for secondary phosphine oxide: under N_2 atmosphere, 0.24 mmol (dimethoxymethyl)benzene, 0.2 mmol diphenylphosphine oxide, 10 mol% $FeCl_3$ and 0.2 mL dioxane were charged into a 25 mL schlenk tube, and the mixture was stirred at 80

°C for 10 h. After removal of the volatiles, the residues were passed through a short silica chromatography (particle size 37–54 μm, petroleum ether/ethyl acetate as eluent) to afford analytically pure product **3**.

Procedure for H-phosphonate: under N_2 atmosphere, 0.60 mmol (dimethoxymethyl)benzene, 0.4 mmol H-phosphonate, 10 mol% $FeCl_3$ and 0.2 mL 1,4-dioxane were charged into a 25 mL schlenk tube, and the mixture was stirred at 120 °C for 10 h. After removal of the volatiles, the residues were passed through a short silica chromatography (particle size 37–54 μm, petroleum ether/ethyl acetate as eluent) to afford analytically pure product **3**.

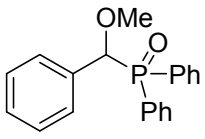
Control experiments on the effect of possible impurity copper:



Run	Cat.	Amount	Temp. (°C)	Time (h)	GC Yield (%)
1	$CuCl_2$	1 mol%	80	12	15
2	$CuCl_2$	0.5 mol%	80	12	trace
3	CuCl	1 mol%	80	12	trace
4	Cu_2O	0.5 mol%	80	12	trace

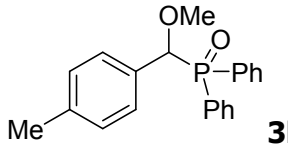
These results clearly indicate that it was Fe, *not the possible impurity Cu*, catalyzed the reactions.

Characterization data of 3



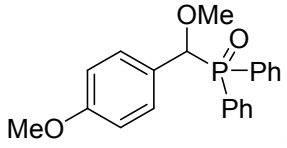
3a[1]

Following the general procedure (dioxane, 80 °C, 10 h), **3a** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (t, J = 8.8 Hz, 2H), 7.75 (t, J = 8.8 Hz, 2H), 7.40-7.53 (m, 6H), 7.24 (b, 3H), 7.17 (b, 2H), 5.03 (d, J = 11.2 Hz, 1H), 3.35 (s, 3H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.58; ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.43 (d, $J_{P\text{-}C}$ = 1.3 Hz), 132.59 (d, $J_{P\text{-}C}$ = 8.8 Hz), 131.94 (d, $J_{P\text{-}C}$ = 97.2 Hz), 131.96 (q, $J_{P\text{-}C}$ = 3.1 Hz), 131.92 (d, $J_{P\text{-}C}$ = 2.9 Hz), 131.72 (d, $J_{P\text{-}C}$ = 8.7 Hz), 129.05 (d, $J_{P\text{-}C}$ = 97.1 Hz),128.42 (d, $J_{P\text{-}C}$ = 11.5 Hz), 128.26 (d, $J_{P\text{-}C}$ = 2.7 Hz), 128.13 (d, $J_{P\text{-}C}$ = 2.0 Hz), 128.04, 127.96 (d, $J_{P\text{-}C}$ = 7.5 Hz),83.55 (d, $J_{P\text{-}C}$ = 87.5 Hz), 58.64 (d, $J_{P\text{-}C}$ = 12.6 Hz).



3b

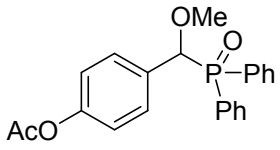
Following the general procedure (dioxane, 80 °C, 10 h), **3b** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (t, J = 8.4 Hz, 2H), 7.77 (t, J = 8.4 Hz, 2H), 7.41-7.53 (m, 6H), 7.04 (b, 4H), 4.99 (d, J = 10.8 Hz, 1H), 3.34 (s, 3H), 2.30 (s, 3H). ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.47; ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.10 (d, $J_{P\text{-}C}$ = 2.2 Hz), 132.63 (d, $J_{P\text{-}C}$ = 8.9 Hz), 132.12 (d, $J_{P\text{-}C}$ = 94.5 Hz), 131.90 (d, $J_{P\text{-}C}$ = 2.8 Hz), 131.85 (d, $J_{P\text{-}C}$ = 2.4 Hz), 131.69 (d, $J_{P\text{-}C}$ = 8.8 Hz), 130.26, 129.18 (d, $J_{P\text{-}C}$ = 96.5 Hz), 128.89 (d, $J_{P\text{-}C}$ = 1.6 Hz), 128.40 (d, $J_{P\text{-}C}$ = 11.4 Hz), 128.02, 127.95 (d, $J_{P\text{-}C}$ = 8.7 Hz), 83.48 (d, $J_{P\text{-}C}$ = 88.3 Hz), 58.46 (d, $J_{P\text{-}C}$ = 12.7 Hz), 21.22. HRMS Calcd. for $C_{21}H_{21}O_2P$ (M+) 336.1279, found 336.1273, M.P. 118.1-118.3 °C.



3c

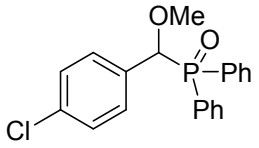
Following the general procedure (THF, 80 °C, 10 h), **3c** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84-7.89 (m, 2H), 7.74-7.78 (m, 2H), 7.39-7.54 (m, 6H), 7.07 (dd, $J_{P\text{-}H}$ = 2.0 Hz, $J_{H\text{-}H}$ = 8.8 Hz, 2H), 6.78 (d, J = 8.8 Hz, 2H), 4.97 (d, J = 10.4 Hz, 1H), 3.77 (s, 3H), 3.33 (s, 3H). ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.51; ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.68 (d, $J_{P\text{-}C}$ = 2.5 Hz), 132.60 (d, $J_{P\text{-}C}$ = 8.8 Hz), 132.04 (d, $J_{P\text{-}C}$ = 96.7 Hz), 131.92 (d, $J_{P\text{-}C}$ = 2.8 Hz), 131.85 (d, $J_{P\text{-}C}$ = 2.7 Hz), 131.67 (d, $J_{P\text{-}C}$ = 8.7 Hz), 129.36

(d, J_{P-C} = 4.5 Hz), 129.22 (d, J_{P-C} = 96.4 Hz), 128.40 (d, J_{P-C} = 11.4 Hz), 127.99 (d, J_{P-C} = 11.6 Hz), 125.22 (d, J_{P-C} = 1.7 Hz), 113.64 (d, J_{P-C} = 2.0 Hz), 83.10 (d, J_{P-C} = 89.4 Hz), 58.32 (d, J_{P-C} = 12.8 Hz), 55.22. HRMS Calcd. for $C_{21}H_{21}O_3P$ (M+) 352.1228, found 352.1224, M.P. 178.7-178.9 °C.



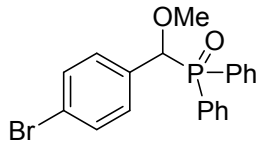
3d

Following the general procedure (THF, 80 °C, 10 h), **3d** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86-7.91 (m, 2H), 7.70-7.75 (m, 2H), 7.38-7.56 (m, 6H), 7.17 (dd, J_{P-H} = 2.0 Hz, J_{H-H} = 8.4 Hz, 2H), 6.97 (d, J = 8.4 Hz, 2H), 5.03 (d, J = 11.2 Hz, 1H), 3.36 (s, 3H), 2.27 (s, 3H). ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.80; ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.32 (d, J_{P-C} = 0.5 Hz), 150.65 (d, J_{P-C} = 3.2 Hz), 132.56 (d, J_{P-C} = 8.8 Hz), 132.07 (d, J_{P-C} = 3.0 Hz), 132.03 (d, J_{P-C} = 2.7 Hz), 131.69 (d, J_{P-C} = 8.8 Hz), 131.63 (d, J_{P-C} = 97.5 Hz), 130.97 (d, J_{P-C} = 1.5 Hz), 128.96 (d, J_{P-C} = 4.2 Hz), 128.68 (d, J_{P-C} = 97.4 Hz), 128.49 (d, J_{P-C} = 11.5 Hz), 128.05 (d, J_{P-C} = 11.7 Hz), 121.28 (d, J_{P-C} = 2.3 Hz), 83.03 (d, J_{P-C} = 87.6 Hz), 58.76 (d, J_{P-C} = 12.5 Hz), 21.16. HRMS Calcd. for $C_{22}H_{21}O_4P$ (M+) 380.1177, found 380.1170, M.P. 157.1-158.0 °C.



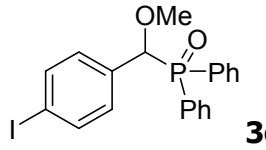
3e

Following the general procedure (THF, 80 °C, 10 h), **3e** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89 (t, J = 8.4 Hz, 2H), 7.74 (t, J = 8.4 Hz, 2H), 7.39-7.55 (m, 6H), 7.21 (d, J = 7.6 Hz, 2H), 7.10 (d, J = 7.6 Hz, 2H), 5.00 (d, J = 11.2 Hz, 1H), 3.33 (s, 3H). ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.41; ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.17 (d, J_{P-C} = 3.3 Hz), 132.54 (d, J_{P-C} = 8.8 Hz), 132.13 (d, J_{P-C} = 3.3 Hz), 132.08 (d, J_{P-C} = 2.7 Hz), 131.63 (d, J_{P-C} = 8.8 Hz), 130.62 (d, J_{P-C} = 96.6 Hz), 129.23 (d, J_{P-C} = 4.2 Hz), 128.62 (d, J_{P-C} = 96.5 Hz), 128.53 (d, J_{P-C} = 11.6 Hz), 128.35 (d, J_{P-C} = 2.2 Hz), 128.08 (d, J_{P-C} = 11.8 Hz), 82.97 (d, J_{P-C} = 87.1 Hz), 58.72 (d, J_{P-C} = 12.5 Hz). HRMS Calcd. for $C_{20}H_{18}ClO_2P$ (M+) 356.0733, found 356.0726, M.P. 184.8-186.8 °C.



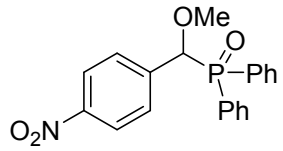
3f

Following the general procedure (dioxane, 80 °C, 10 h), **3f** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79-7.84 (m, 2H), 7.64-7.69 (m, 2H), 7.29-7.51 (m, 8H), 6.96 (dd, $J_{P\text{-}H}$ = 1.6 Hz, $J_{H\text{-}H}$ = 8.4 Hz, 2H), 4.90 (d, J = 11.6 Hz, 1H), 3.27 (s, 3H). ^{31}P NMR (162 MHz, $CDCl_3$) δ 28.84; ^{13}C NMR (100 MHz, $CDCl_3$) δ 131.67 (d, $J_{P\text{-}C}$ = 1.3 Hz), 131.53 (d, $J_{P\text{-}C}$ = 8.8 Hz), 131.08 (d, $J_{P\text{-}C}$ = 2.8 Hz), 131.03 (d, $J_{P\text{-}C}$ = 2.7 Hz), 130.70 (d, $J_{P\text{-}C}$ = 97.3 Hz), 130.62 (d, $J_{P\text{-}C}$ = 8.9 Hz), 130.27 (d, $J_{P\text{-}C}$ = 2.2 Hz), 128.52 (d, $J_{P\text{-}C}$ = 4.2 Hz), 127.68 (d, $J_{P\text{-}C}$ = 97.0 Hz), 127.49 (d, $J_{P\text{-}C}$ = 11.6 Hz), 127.05 (d, $J_{P\text{-}C}$ = 11.8 Hz), 121.40 (d, $J_{P\text{-}C}$ = 3.5 Hz), 82.13 (d, $J_{P\text{-}C}$ = 86.8 Hz), 57.73 (d, $J_{P\text{-}C}$ = 12.3 Hz). HRMS Calcd. for $C_{20}H_{18}BrO_2P$ (M+) 400.0228, found 400.0209, M.P. 193.5-194.0 °C.



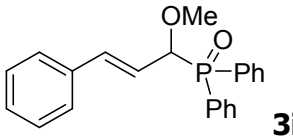
3g

Following the general procedure (THF, 80 °C, 10 h), **3g** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86-7.91 (m, 2H), 7.71-7.76 (m, 2H), 7.39-7.58 (m, 8H), 6.90 (dd, $J_{P\text{-}H}$ = 1.6 Hz, $J_{H\text{-}H}$ = 8.4 Hz, 2H), 4.96 (d, J = 3.0 Hz, 1H), 3.33 (s, 3H). ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.13; ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.23 (d, $J_{P\text{-}C}$ = 2.2 Hz), 133.37 (d, $J_{P\text{-}C}$ = 1.7 Hz), 132.55 (d, $J_{P\text{-}C}$ = 8.8 Hz), 132.12 (d, $J_{P\text{-}C}$ = 2.8 Hz), 132.06 (d, $J_{P\text{-}C}$ = 2.8 Hz), 131.70 (d, $J_{P\text{-}C}$ = 97.6 Hz), 131.63 (d, $J_{P\text{-}C}$ = 8.8 Hz), 129.75 (d, $J_{P\text{-}C}$ = 4.2 Hz), 128.64 (d, $J_{P\text{-}C}$ = 97.2 Hz), 128.51 (d, $J_{P\text{-}C}$ = 11.5 Hz), 128.07 (d, $J_{P\text{-}C}$ = 11.7 Hz), 94.23 (d, $J_{P\text{-}C}$ = 3.6 Hz), 83.21 (d, $J_{P\text{-}C}$ = 86.6 Hz), 58.77 (d, $J_{P\text{-}C}$ = 12.3 Hz). HRMS Calcd. for $C_{20}H_{18}IO_2P$ (M+) 448.0089, found 448.0070, M.P. 197.5-197.8 °C.



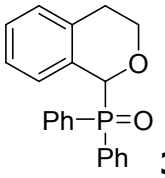
3h

Following the general procedure (dioxane, 80 °C, 10 h), **3h** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (d, J = 8.4 Hz, 2H), 7.91-7.96 (m, 2H), 7.71-7.76 (m, 2H), 7.51-7.61 (m, 4H), 7.37-7.45 (m, 4H), 5.15 (d, J = 13.2 Hz, 1H), 3.40 (s, 3H). ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.30; ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.75 (d, $J_{P\text{-}C}$ = 3.2 Hz), 141.53 (d, $J_{P\text{-}C}$ = 1.3 Hz), 132.43 (d, $J_{P\text{-}C}$ = 8.7 Hz), 132.41 (d, $J_{P\text{-}C}$ = 3.4 Hz), 132.37 (d, $J_{P\text{-}C}$ = 4.5 Hz), 131.59 (d, $J_{P\text{-}C}$ = 8.9 Hz), 131.21 (d, $J_{P\text{-}C}$ = 98.8 Hz), 128.67 (d, $J_{P\text{-}C}$ = 11.7 Hz), 128.42 (d, $J_{P\text{-}C}$ = 3.9 Hz), 128.19 (d, $J_{P\text{-}C}$ = 11.8 Hz), 127.98 (d, $J_{P\text{-}C}$ = 97.5 Hz), 123.22 (d, $J_{P\text{-}C}$ = 2.3 Hz), 83.16 (d, $J_{P\text{-}C}$ = 83.7 Hz), 59.29 (d, $J_{P\text{-}C}$ = 11.7 Hz). HRMS Calcd. for $C_{20}H_{18}NO_4P$ (M+) 367.0973, found 367.0960, M.P. 209.0-210.5 °C.



3i[1]

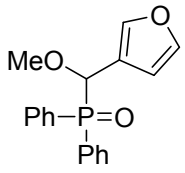
Following the general procedure (dioxane, 80 °C, 10 h), **3i** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.89-7.95 (m, 4H), 7.46-7.58 (m, 6H), 7.23-7.33 (m, 5H), 6.60-6.65 (m, 1H), 6.19-6.26 (m, 1H), 4.65-4.71 (m, 1H), 3.42 (s, 3H). ^{31}P NMR (162 MHz, $CDCl_3$) δ 28.28; ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.10 (d, $J_{P\text{-}C}$ = 2.7 Hz), 135.17 (d, $J_{P\text{-}C}$ = 10.5 Hz), 132.31 (d, $J_{P\text{-}C}$ = 8.9 Hz), 132.07 (d, $J_{P\text{-}C}$ = 2.8 Hz), 132.00 (d, $J_{P\text{-}C}$ = 2.7 Hz), 131.73 (d, $J_{P\text{-}C}$ = 96.5 Hz), 131.68 (d, $J_{P\text{-}C}$ = 8.8 Hz), 129.53 (d, $J_{P\text{-}C}$ = 96.5 Hz), 128.52, 128.46 (d, $J_{P\text{-}C}$ = 11.8 Hz), 128.23 (d, $J_{P\text{-}C}$ = 11.7 Hz), 128.04 (s), 126.73 (d, $J_{P\text{-}C}$ = 1.4 Hz), 82.46 (d, $J_{P\text{-}C}$ = 89.2 Hz), 58.74 (d, $J_{P\text{-}C}$ = 11.4 Hz).



3j

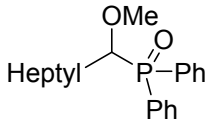
Following the general procedure (dioxane, 80 °C, 10 h), **3j** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95-8.00 (m, 2H), 7.56-7.65 (m, 4H), 7.48-7.53 (m, 2H), 7.41-7.45 (m, 1H), 7.26-7.31 (m, 2H), 7.12-7.20 (m, 2H), 6.97 (d, J = 7.2 Hz, 1H), 5.72 (d, J = 8.8 Hz, 1H), 4.14-4.19 (m, 1H), 3.61-3.67 (m, 1H), 2.46-2.48 (m, 2H). ^{31}P NMR (162 MHz, $CDCl_3$) δ 31.23; ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.44 (d, $J_{P\text{-}C}$ = 4.8 Hz), 132.37 (d, $J_{P\text{-}C}$ = 8.5 Hz), 132.24 (d, $J_{P\text{-}C}$ = 8.9 Hz), 131.93 (d, $J_{P\text{-}C}$ = 2.8 Hz), 131.77 (d,

$J_{P\text{-}C}$ = 2.7 Hz), 131.68 (d, $J_{P\text{-}C}$ = 96.9 Hz), 129.86 (d, $J_{P\text{-}C}$ = 3.8 Hz), 129.72 (d, $J_{P\text{-}C}$ = 98.0 Hz), 128.42 (d, $J_{P\text{-}C}$ = 2.2 Hz), 128.41 (d, $J_{P\text{-}C}$ = 2.2 Hz), 127.75 (d, $J_{P\text{-}C}$ = 11.6 Hz), 126.87 (d, $J_{P\text{-}C}$ = 2.6 Hz), 126.68 (d, $J_{P\text{-}C}$ = 3.3 Hz), 126.15 (d, $J_{P\text{-}C}$ = 2.6 Hz), 75.35 (d, $J_{P\text{-}C}$ = 87.8 Hz), 64.30 (d, $J_{P\text{-}C}$ = 7.3 Hz), 28.84 (s). HRMS Calcd. for $C_{21}H_{19}O_2P$ (M+) 334.1123, found 334.1112.



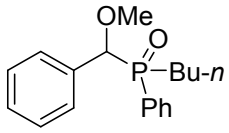
3k

Following the general procedure (dioxane, 80 °C, 10 h), **3k** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84-7.90 (m, 4H), 7.52-7.57 (m, 2H), 7.43-7.49 (m, 4H), 7.30-7.33 (m, 2H), 6.12 (s, 1H), 4.98 (d, J = 10.8 Hz, 1H), 3.34 (s, 3H). ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.13; ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.21 (s), 141.92 (d, $J_{P\text{-}C}$ = 8.3 Hz), 132.45 (d, $J_{P\text{-}C}$ = 8.8 Hz), 131.79 (d, $J_{P\text{-}C}$ = 97.6 Hz), 132.077 (d, $J_{P\text{-}C}$ = 2.8 Hz), 131.95 (d, $J_{P\text{-}C}$ = 2.7 Hz), 131.62 (d, $J_{P\text{-}C}$ = 8.8 Hz), 129.43 (d, $J_{P\text{-}C}$ = 97.1 Hz), 128.43 (d, $J_{P\text{-}C}$ = 115. Hz), 128.14 (d, $J_{P\text{-}C}$ = 11.6 Hz), 118.61 (s), 109.95 (d, $J_{P\text{-}C}$ = 2.6 Hz), 76.13 (s), 58.43 (d, $J_{P\text{-}C}$ = 11.6 Hz).



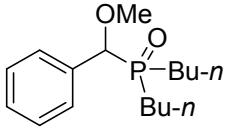
3l

Following the general procedure (THF, 80 °C, 10 h), **3l** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92-7.917 (m, 2H), 7.73-7.78 (m, 2H), 7.37-7.51 (m, 6H), 3.75-3.80 (m, 1H), 3.12 (s, 3H), 1.79-1.89 (m, 1H), 1.44-1.62 (m, 2H), 1.27-1.35 (m, 1H), 1.15-1.21 (m, 8H), 0.78 (t, J = 6.4 Hz, 3H). ^{31}P NMR (162 MHz, $CDCl_3$) δ 28.96; ^{13}C NMR (100 MHz, $CDCl_3$) δ 131.53 (d, $J_{P\text{-}C}$ = 93.9 Hz), 131.13 (d, $J_{P\text{-}C}$ = 8.5 Hz), 130.90 (d, $J_{P\text{-}C}$ = 2.7 Hz), 130.75 (d, $J_{P\text{-}C}$ = 2.7 Hz), 130.49 (d, $J_{P\text{-}C}$ = 8.9 Hz), 128.82 (d, $J_{P\text{-}C}$ = 93.2 Hz), 127.44 (d, $J_{P\text{-}C}$ = 10.8 Hz), 127.33 (d, $J_{P\text{-}C}$ = 10.8 Hz), 81.00 (d, $J_{P\text{-}C}$ = 87.0 Hz), 59.94 (d, $J_{P\text{-}C}$ = 5.2 Hz), 30.70, 29.03 (d, $J_{P\text{-}C}$ = 4.1 Hz), 28.43, 28.08, 25.66 (d, $J_{P\text{-}C}$ = 11.1 Hz), 21.57, 13.05. HRMS Calcd. for $C_{21}H_{29}O_2P$ (M+) 344.1905, found 344.1891, M.P. 79.0-79.6 °C.



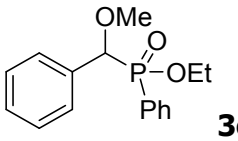
3m

Following the general procedure (dioxane, 80 °C, 10 h), **3m** was isolated as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.74 (t, J = 8.4 Hz, 1H), 7.39-7.47 (m, 3H), 7.11-7.28 (m, 4H), 6.92 (d, J = 5.2 Hz, 1H), 4.47-4.65 (m, 1H), 3.31(s, 1.5 H), 3.24 (s, 1.5 H), 1.79-2.18 (m, 3H), 0.72-1.38 (m, 3H), 0.75-0.86 (m, 3H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 39.84, 37.42; ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.05 (b), 133.68 (b), 131.76 (d, J_{P-C} = 7.6 Hz), 131.73 (b), 131.69 (d, J_{P-C} = 8.0 Hz), 130.89 (d, J_{P-C} = 90.8 Hz), 129.19 (d, J_{P-C} = 90.9 Hz), 128.45 (d, J_{P-C} = 2.3 Hz), 128.27 (d, J_{P-C} = 11.2 Hz), 128.20 (d, J_{P-C} = 3.0 Hz), 128.04 (d, J_{P-C} = 2.2 Hz), 128.01 (d, J_{P-C} = 3.5 Hz), 127.92 (d, J_{P-C} = 11.4 Hz), 127.50 (d, J_{P-C} = 4.1 Hz), 127.36 (d, J_{P-C} = 4.1 Hz), 83.61 (d, J_{P-C} = 82.7 Hz), 85.15 (d, J_{P-C} = 83.1 Hz), 59.05 (d, J_{P-C} = 11.7 Hz), 58.75 (d, J_{P-C} = 12.2 Hz), 25.68 (d, J_{P-C} = 67.3 Hz), 24.25 (d, J_{P-C} = 7.6 Hz), 24.11 (d, J_{P-C} = 7.9 Hz), 23.39 (d, J_{P-C} = 66.7 Hz), 23.14 (d, J_{P-C} = 4.2 Hz), 22.71, 22.66, 13.67, 13.57. HRMS Calcd. for $C_{14}H_{14}O_2P$ (M+) 302.1436, found 302.1429, M.P. 75.7-77.3 °C.



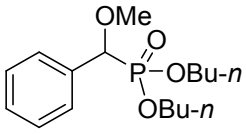
3n

Following the general procedure (dioxane, 80 °C, 10 h), **3n** was isolated as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.39 (mb, 5H), 4.55 (b, 1H), 3.43 (s, 3H), 1.26-1.94 (mb, 12H), 0.95 (b, 3H); 0.84 (b, 3H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 48.82; ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.06, 128.60 (d, J_{P-C} = 1.9 Hz), 128.15 (d, J_{P-C} = 2.4 Hz), 127.06 (d, J_{P-C} = 3.6 Hz), 81.44 (d, J_{P-C} = 79.4 Hz), 58.85 (d, J_{P-C} = 11.9 Hz), 25.21 (d, J_{P-C} = 63.7 Hz), 24.36 (d, J_{P-C} = 7.1 Hz), 24.22 (d, J_{P-C} = 7.2 Hz), 24.12 (d, J_{P-C} = 63.0 Hz), 23.35 (d, J_{P-C} = 4.4 Hz), 23.14 (d, J_{P-C} = 4.1 Hz), 13.65, 13.52. HRMS Calcd. for $C_{16}H_{27}O_2P$ (M+) 282.1749, found 282.1743.



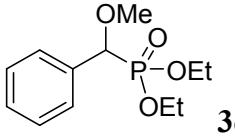
3o

Following the general procedure (THF, 120 °C, 10 h), **3o** was isolated as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63-7.70 (m, 2H), 7.53 (t, J = 7.6 Hz, 1H), 7.39-7.42 (m, 2H), 7.27-7.30 (bm, 4H), 7.21 (b, 1H), 4.57-4.64 (m, 1H), 3.93-4.15 (m, 2H), 3.33 (d, J = 5.2 Hz, 3H), 1.28 (t, J = 7.6 Hz, 3H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 35.40, 34.82; ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.14 (d, J_{P-C} = 1.7 Hz), 134.01, 132.76 (d, J_{P-C} = 2.1 Hz), 132.67 (d, J_{P-C} = 2.1 Hz), 132.42 (d, J_{P-C} = 2.5 Hz), 128.72 (d, J_{P-C} = 105.0 Hz), 128.50 (d, J_{P-C} = 105.3 Hz), 128.25 (d, J_{P-C} = 3.0 Hz), 128.17 (b), 128.15 (b), 128.13 (b), 128.07 (b), 128.03 (d, J_{P-C} = 2.1 Hz), 127.95 (d, J_{P-C} = 4.9 Hz), 127.77 (b),82.57 (d, J_{P-C} = 118.5 Hz), 82.64 (d, J_{P-C} = 118.7 Hz), 61.65 (d, J_{P-C} = 6.9 Hz), 61.49 (d, J_{P-C} = 6.9 Hz), 59.01 (d, J_{P-C} = 12.7 Hz), 58.72 (d, J_{P-C} = 13.1 Hz), 16.54 (d, J_{P-C} = 1.7 Hz), 16.49 (d, J_{P-C} = 1.6 Hz). HRMS Calcd. for $C_{16}H_{19}O_3P$ (M+) 290.1072, found 290.1063.



3p

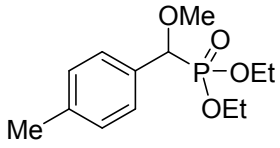
Following the general procedure (dioxane, 120 °C, 10 h), **3p** was isolated as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31-7.44 (m, 5H), 4.52 (d, J = 16.8Hz, 1H), 3.61-3.80 (m, 4H), 3.38 (s, 3H), 1.78-1.91 (m, 3H), 0.84-0.87 (bm, 12H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 18.64; ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.60 (d, J_{P-C} = 1.8 Hz), 128.39 (d, J_{P-C} = 4.6 Hz), 128.35 (d, J_{P-C} = 2.4 Hz), 128.06 (d, J_{P-C} = 5.8 Hz), 80.55 (d, J_{P-C} = 168.4 Hz), 72.79 (d, J_{P-C} = 6.8 Hz), 72.72 (d, J_{P-C} = 6.7 Hz), 58.66 (d, J_{P-C} = 14.9 Hz), 29.25 (d, J_{P-C} = 4.0 Hz), 29.20 (d, J_{P-C} = 4.1 Hz), 18.63, 18.59 (d, J_{P-C} = 12.3 Hz). HRMS Calcd. for $C_{16}H_{27}O_4P$ (M+) 314.1647, found 314.1643.



3q[2]

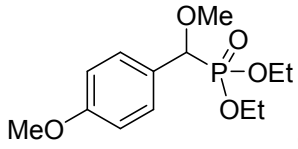
Following the general procedure (dioxane, 120 °C, 10 h), **3q** was isolated as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.44 (m, 5H), 4.50 (d, J = 15.6 Hz, 1H), 3.90-4.08 (m, 4H), 3.37 (s, 3H), 1.22 (dt, J_{P-H} = 13.6 Hz, J_{H-H} = 6.8 Hz, 6H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 19.13; ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.33 (d, J_{P-C} = 1.8 Hz), 127.43 (d, J_{P-C}

= 3.5 Hz), 127.38 (d, J_{P-C} = 2.3 Hz), 126.97 (d, J_{P-C} = 5.8 Hz), 79.40 (d, J_{P-C} = 167.5 Hz), 62.09 (d, J_{P-C} = 6.9 Hz), 62.96 (d, J_{P-C} = 6.8 Hz), 57.66 (d, J_{P-C} = 14.9 Hz), 15.40 (d, J_{P-C} = 6.3 Hz), 15.34 (d, J_{P-C} = 6.3 Hz).



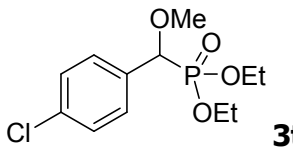
3r[3]

Following the general procedure (dioxane, 120 °C, 10 h), **3r** was isolated as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (d, J = 8.0 Hz, 2H), 7.18 (d, J = 7.2 Hz, 2H), 4.47 (d, J = 15.6 Hz, 1H), 3.89-4.11 (m, 4H), 3.36 (s, 3H), 2.35 (s, 3H), 1.27 (dt, J_{P-H} = 14.8 Hz, J_{H-H} = 6.8 Hz, 6H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 19.33; ^{13}C NMR (100 MHz, $CDCl_3$) δ 138.28 (d, J_{P-C} = 3.1 Hz), 131.25 (d, J_{P-C} = 2.0 Hz), 129.14 (d, J_{P-C} = 2.3 Hz), 127.99 (d, J_{P-C} = 5.8 Hz), 80.29 (d, J_{P-C} = 168.3 Hz), 63.07 (d, J_{P-C} = 6.9 Hz), 62.94 (d, J_{P-C} = 6.8 Hz), 58.52 (d, J_{P-C} = 15.0 Hz), 21.24, 16.45 (d, J_{P-C} = 6.1 Hz), 16.38 (d, J_{P-C} = 6.4 Hz).



3s[3]

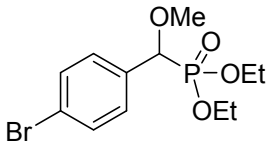
Following the general procedure (dioxane, 120 °C, 10 h), **3s** was isolated as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 (d, J = 8.4 Hz, 2H), 6.90 (d, J = 7.6 Hz, 2H), 4.44 (d, J = 14.8 Hz, 1H), 3.87-4.10 (m, 4H), 3.81 (s, 3H), 3.34 (s, 3H), 1.24 (dt, J_{P-H} = 21.2 Hz, J_{H-H} = 6.8 Hz, 6H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 19.46; ^{13}C NMR (100 MHz, $CDCl_3$) δ 159.79 (d, J_{P-C} = 3.0 Hz), 129.39 (d, J_{P-C} = 6.0 Hz), 126.23 (d, J_{P-C} = 1.8 Hz), 113.87 (d, J_{P-C} = 2.2 Hz), 79.92 (d, J_{P-C} = 169.7 Hz), 63.04 (d, J_{P-C} = 6.9 Hz), 62.91 (d, J_{P-C} = 6.8 Hz), 58.35 (d, J_{P-C} = 15.1 Hz), 55.27, 16.47 (d, J_{P-C} = 5.7 Hz), 16.39 (d, J_{P-C} = 5.7 Hz).



3t[3]

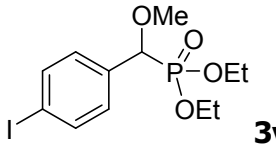
Following the general procedure (dioxane, 120 °C, 10 h), **3t** was isolated as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32-7.37 (m, 4H), 4.45 (d, J = 15.6 Hz, 1H), 3.92-4.11 (m,

4H), 3.36 (s, 3H), 1.23 (dt, $J_{P\text{-}H} = J_{H\text{-}H} = 7.2$ Hz, 6H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 18.42; ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.29 (d, $J_{P\text{-}C} = 3.7$ Hz), 133.12 (d, $J_{P\text{-}C} = 2.0$ Hz), 129.28 (d, $J_{P\text{-}C} = 5.7$ Hz), 128.62 (d, $J_{P\text{-}C} = 2.5$ Hz), 79.83 (d, $J_{P\text{-}C} = 168.1$ Hz), 63.21 (d, $J_{P\text{-}C} = 6.9$ Hz), 63.04 (d, $J_{P\text{-}C} = 6.7$ Hz), 58.80 (d, $J_{P\text{-}C} = 14.5$ Hz), 16.43 (d, $J_{P\text{-}C} = 4.8$ Hz), 16.38 (d, $J_{P\text{-}C} = 4.1$ Hz).



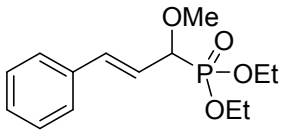
3u

Following the general procedure (dioxane, 120 °C, 10 h), **3u** was isolated as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51 (d, $J = 7.2$ Hz, 2H), 7.31 (d, $J = 8.0$ Hz, 2H), 4.46 (d, $J = 15.6$ Hz, 1H), 3.95-4.13 (m, 4H), 3.38 (s, 3H), 1.25 (dt, $J_{P\text{-}H} = 5.6$ Hz, $J_{H\text{-}H} = 6.8$ Hz, 6H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 18.26; ^{13}C NMR (100 MHz, $CDCl_3$) δ 133.68 (d, $J_{P\text{-}C} = 1.7$ Hz), 131.59 (d, $J_{P\text{-}C} = 2.4$ Hz), 129.60 (d, $J_{P\text{-}C} = 5.7$ Hz), 122.50 (d, $J_{P\text{-}C} = 3.9$ Hz), 79.91 (d, $J_{P\text{-}C} = 168.0$ Hz), 63.24 (d, $J_{P\text{-}C} = 7.0$ Hz), 63.07 (d, $J_{P\text{-}C} = 6.9$ Hz), 58.86 (d, $J_{P\text{-}C} = 14.6$ Hz), 16.44 (d, $J_{P\text{-}C} = 4.4$ Hz), 16.39 (d, $J_{P\text{-}C} = 4.5$ Hz).



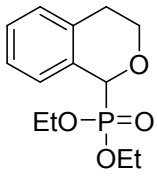
3v

Following the general procedure (dioxane, 120 °C, 10 h), **3v** was isolated as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (d, $J = 8.0$ Hz, 2H), 7.16 (d, $J = 6.4$ Hz, 2H), 4.42 (d, $J = 15.6$ Hz, 1H), 3.95-4.10 (m, 4H), 3.36 (s, 3H), 1.23 (dt, $J_{P\text{-}H} = 6.8$ Hz, $J_{H\text{-}H} = 6.8$ Hz, 6H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 18.19; ^{13}C NMR (100 MHz, $CDCl_3$) δ 137.52 (d, $J_{P\text{-}C} = 2.4$ Hz), 134.38 (d, $J_{P\text{-}C} = 2.0$ Hz), 129.79 (d, $J_{P\text{-}C} = 5.7$ Hz), 94.21 (d, $J_{P\text{-}C} = 4.2$ Hz), 80.00 (d, $J_{P\text{-}C} = 167.7$ Hz), 63.22 (d, $J_{P\text{-}C} = 6.9$ Hz), 63.05 (d, $J_{P\text{-}C} = 6.8$ Hz), 58.86 (d, $J_{P\text{-}C} = 14.4$ Hz), 16.44 (d, $J_{P\text{-}C} = 3.9$ Hz), 16.38 (d, $J_{P\text{-}C} = 4.1$ Hz). HRMS Calcd. for $C_{12}H_{18}IO_4P$ (M+) 383.9987, found 383.9978.



3w

Following the general procedure (dioxane, 120 °C, 10 h), **3w** was isolated as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 (d, J = 7.6 Hz, 2H), 7.34 (t, J = 7.2 Hz, 2H), 7.28 (t, J = 7.2 Hz, 1H), 6.74 (dd, $J_{P\text{-}H}$ = 4.4 Hz, $J_{H\text{-}H}$ = 16.0 Hz, 1H), 6.19-6.26 (m, 1H), 4.14-4.23 (m, 1H), 3.48 (s, 3H), 1.33 (tb, $J_{H\text{-}H}$ = 6.8 Hz, 6H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 19.37; ^{13}C NMR (100 MHz, $CDCl_3$) δ 136.07 (d, $J_{P\text{-}C}$ = 2.6 Hz), 134.62 (d, $J_{P\text{-}C}$ = 13.2 Hz), 128.64, 128.17, 126.73 (d, $J_{P\text{-}C}$ = 1.7 Hz), 122.32 (d, $J_{P\text{-}C}$ = 4.4 Hz), 79.14 (d, $J_{P\text{-}C}$ = 168.7 Hz), 63.11 (d, $J_{P\text{-}C}$ = 6.9 Hz), 62.93 (d, $J_{P\text{-}C}$ = 6.8 Hz), 58.67 (d, $J_{P\text{-}C}$ = 12.9 Hz), 16.57 (d, $J_{P\text{-}C}$ = 1.9 Hz), 16.51 (d, $J_{P\text{-}C}$ = 2.0 Hz). HRMS Calcd. for $C_{14}H_{21}O_4P$ (M+) 284.1177, found 284.1169.



3x

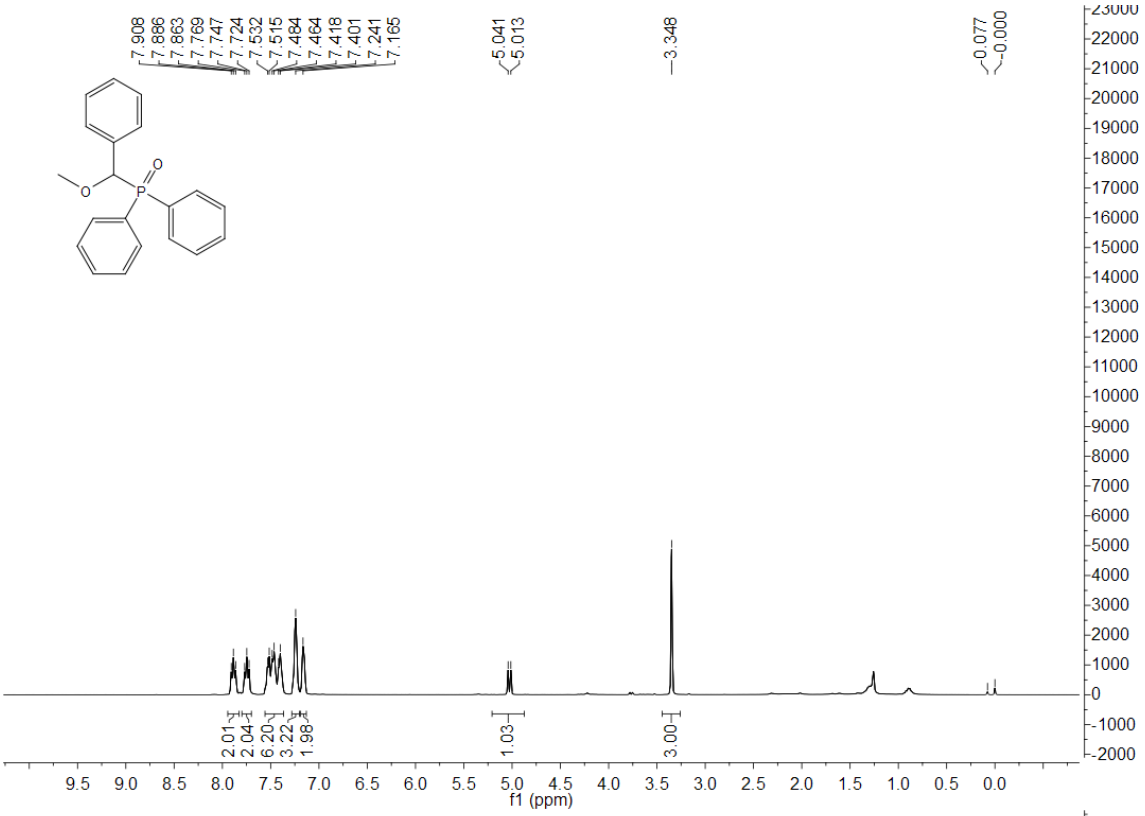
Following the general procedure (dioxane, 120 °C, 10 h), **3x** was isolated as an oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47-7.49 (m, 1H), 7.16-7.20 (m, 2H), 7.01-7.13 (m, 1H), 5.18 (d, J = 14.4 Hz, 1H), 4.29-4.35 (m, 1H), 4.11-4.21 (m, 2H), 3.98-4.07 (m, 1H), 3.84-3.94 (m, 1H), 3.77-3.83 (m, 1H), 2.76-2.95 (m, 2H), 1.33 (t, $J_{H\text{-}H}$ = 7.2 Hz, 3H), 1.13 (t, $J_{H\text{-}H}$ = 6.8 Hz, 3H); ^{31}P NMR (162 MHz, $CDCl_3$) δ 20.29; ^{13}C NMR (100 MHz, $CDCl_3$) δ 134.07 (d, $J_{P\text{-}C}$ = 6.4 Hz), 129.84 (d, $J_{P\text{-}C}$ = 2.0 Hz), 128.96 (d, $J_{P\text{-}C}$ = 2.3 Hz), 127.10 (d, $J_{P\text{-}C}$ = 3.2 Hz), 126.41 (d, $J_{P\text{-}C}$ = 3.7 Hz), 126.16 (d, $J_{P\text{-}C}$ = 3.1 Hz), 72.47 (d, $J_{P\text{-}C}$ = 161.4 Hz), 63.63 (d, $J_{P\text{-}C}$ = 5.9 Hz), 63.36 (d, $J_{P\text{-}C}$ = 7.0 Hz), 62.61 (d, $J_{P\text{-}C}$ = 7.0 Hz), 28.69, 16.45 (d, $J_{P\text{-}C}$ = 5.7 Hz), 16.30 (d, $J_{P\text{-}C}$ = 5.5 Hz). HRMS Calcd. for $C_{13}H_{19}O_4P$ (M+) 270.1021, found 270.1018.

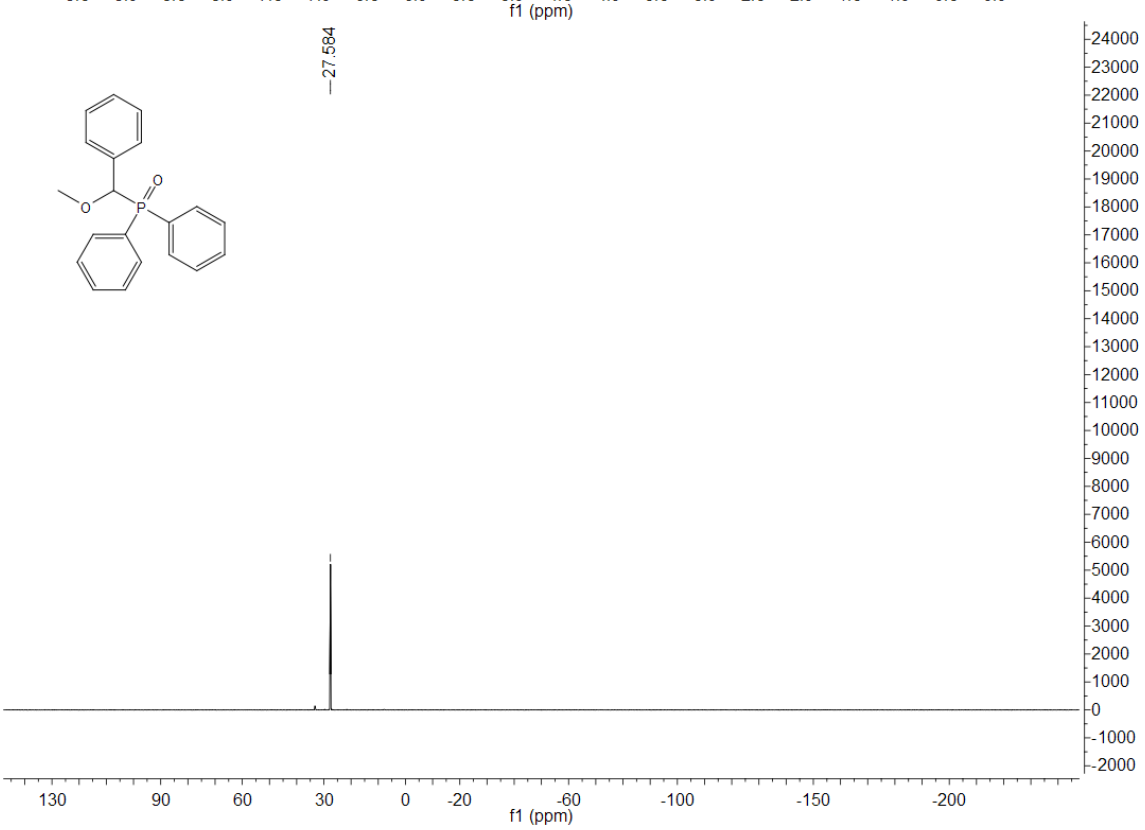
(1) M. Maleki, A. Miller and O. W. Jr. Lever, *Tetrahedron Lett.*, 1981, **22**, 365.

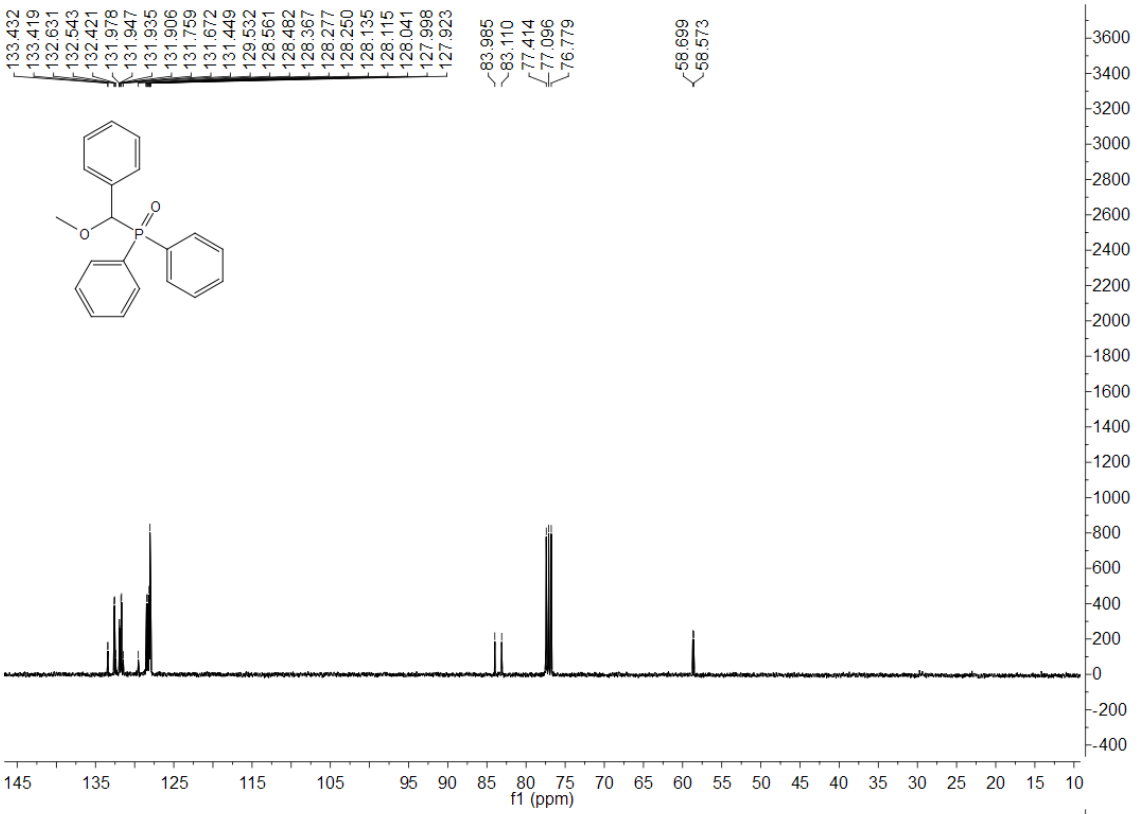
(2) M. E. Jung, J. Cordova and M. Murakami, *Org. Lett.*, 2009, **11**, 3882.

(3) D. Burkhouse and H. Zimmer, *Synthesis*, 1984, **1984**, 330

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



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131.672
131.449
129.532
128.561
128.482
128.367
128.277
128.250
128.135
128.115
128.041
127.998
127.923
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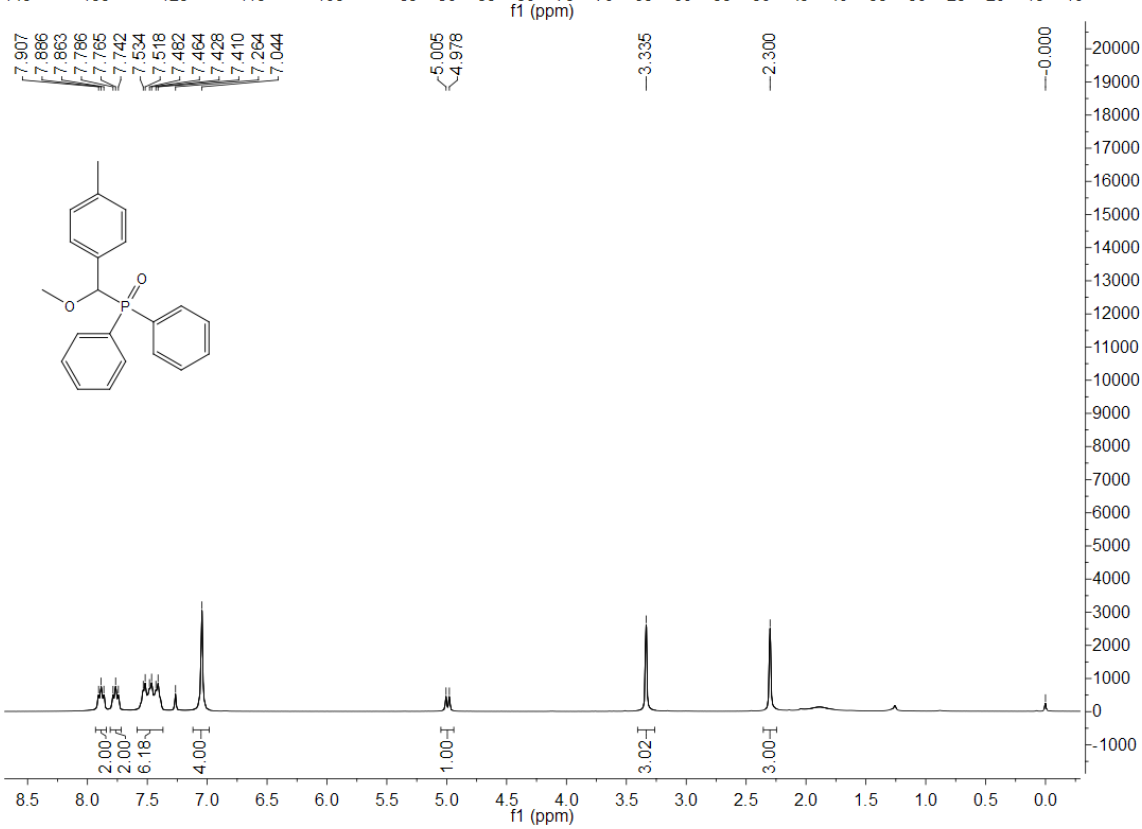
83.110
77.414
77.096
76.779
58.699
58.573
O
O
P
f1 (ppm)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7.742
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7.518
7.482
7.464
7.428
7.410
7.264
7.044
5.005
4.978
3.335
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0.000
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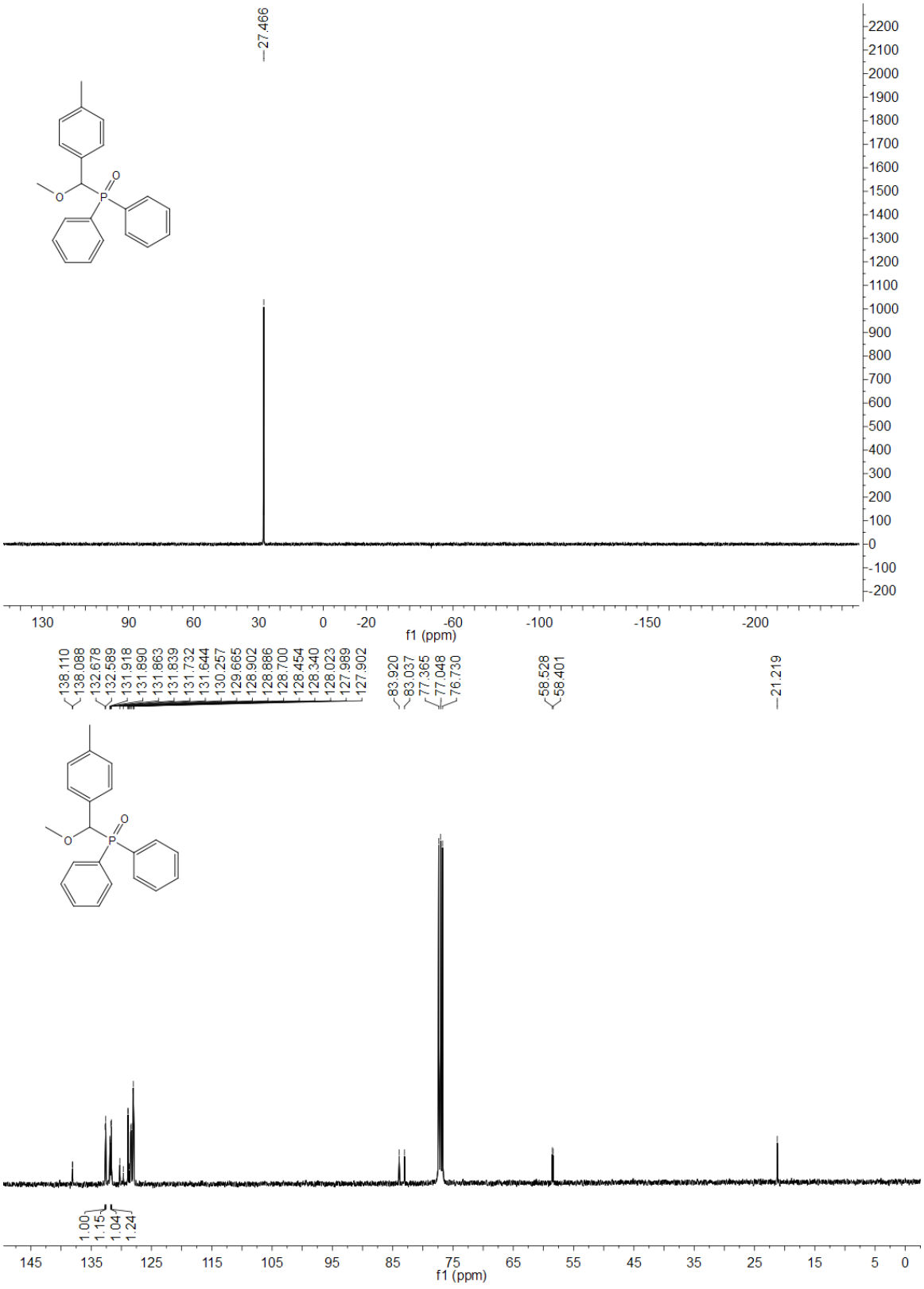
2.00
6.18
4.00
1.00
3.02
3.00
O
O
P
f1 (ppm)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f1 (ppm)
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131.839
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131.644
130.257
129.665
128.902
128.886
128.700
128.454
128.340
128.023
127.989
127.902
83.920
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58.401
21.219
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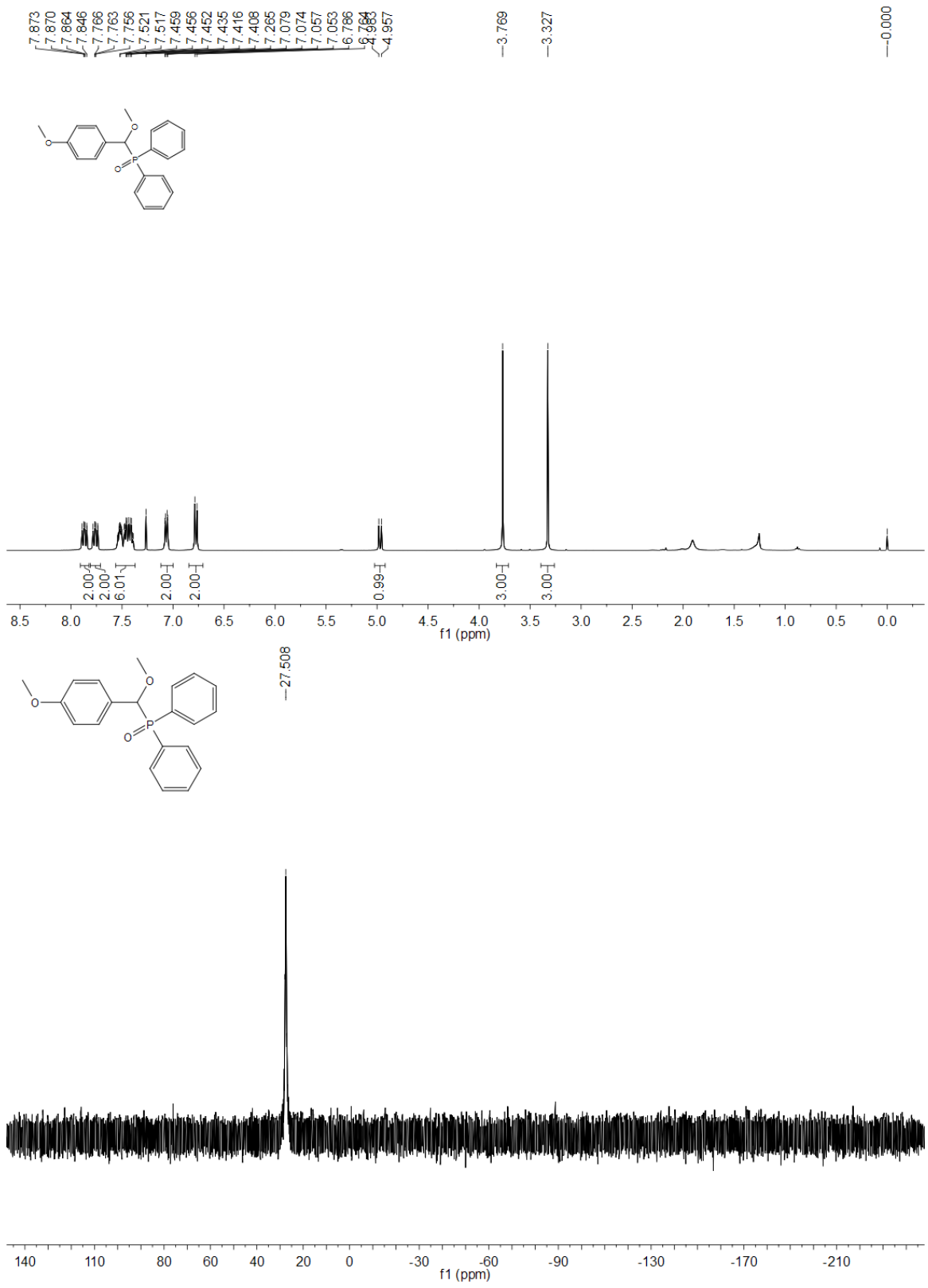
1.15
1.04
1.24
f1 (ppm)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7.864
7.846
7.766
7.763
7.756
7.521
7.517
7.459
7.456
7.452
7.435
7.416
7.408
7.265
7.079
7.074
7.057
7.053
6.786
4.957
3.769
3.327
0.000
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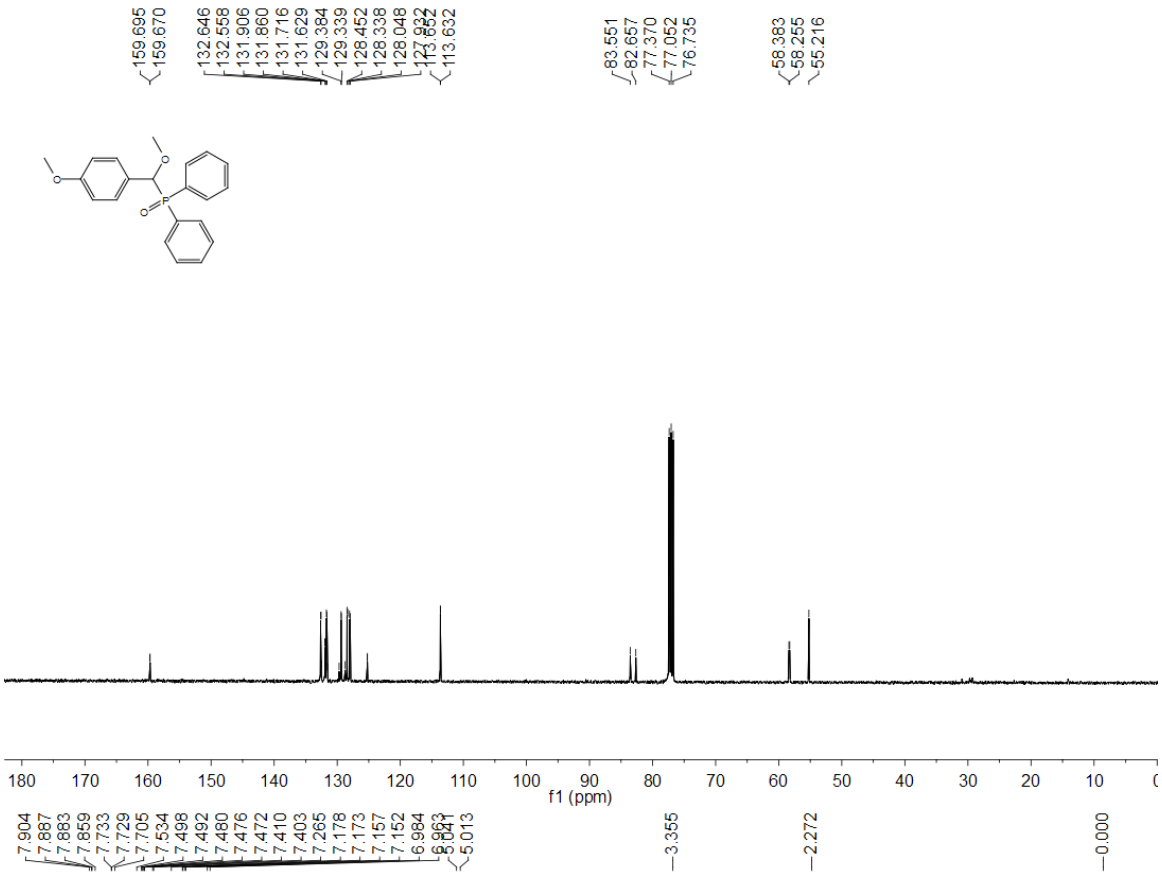
2.00
6.01
2.00
2.00
0.99
3.00
3.00
f1 (ppm)
27.508
f1 (ppm)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131.860
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131.629
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128.452
128.338
128.048
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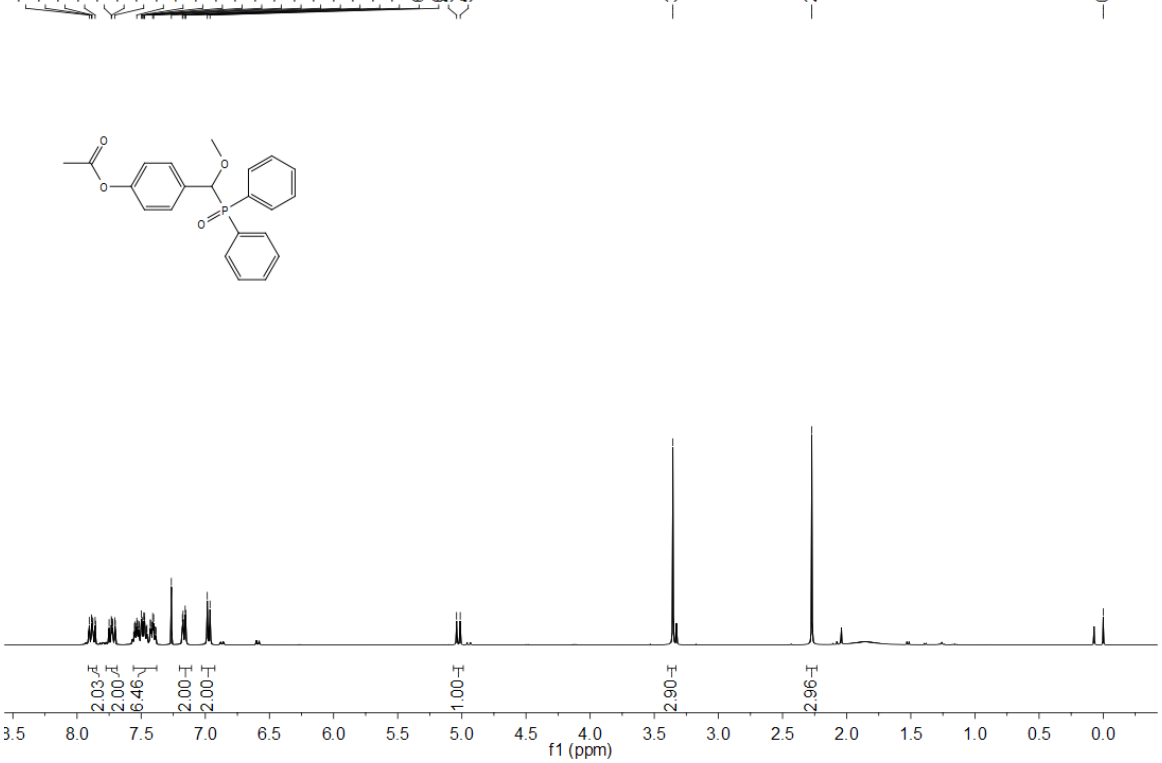
113.632
83.551
82.657
77.370
77.052
76.735
58.383
58.255
55.216
f1 (ppm)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7.883
7.859
7.733
7.729
7.705
7.534
7.498
7.492
7.480
7.476
7.472
7.410
7.403
7.265
7.178
7.173
7.157
7.152
6.984
5.013
3.355
2.272
0.000
2.03
2.00
6.46
2.00
2.00
1.00
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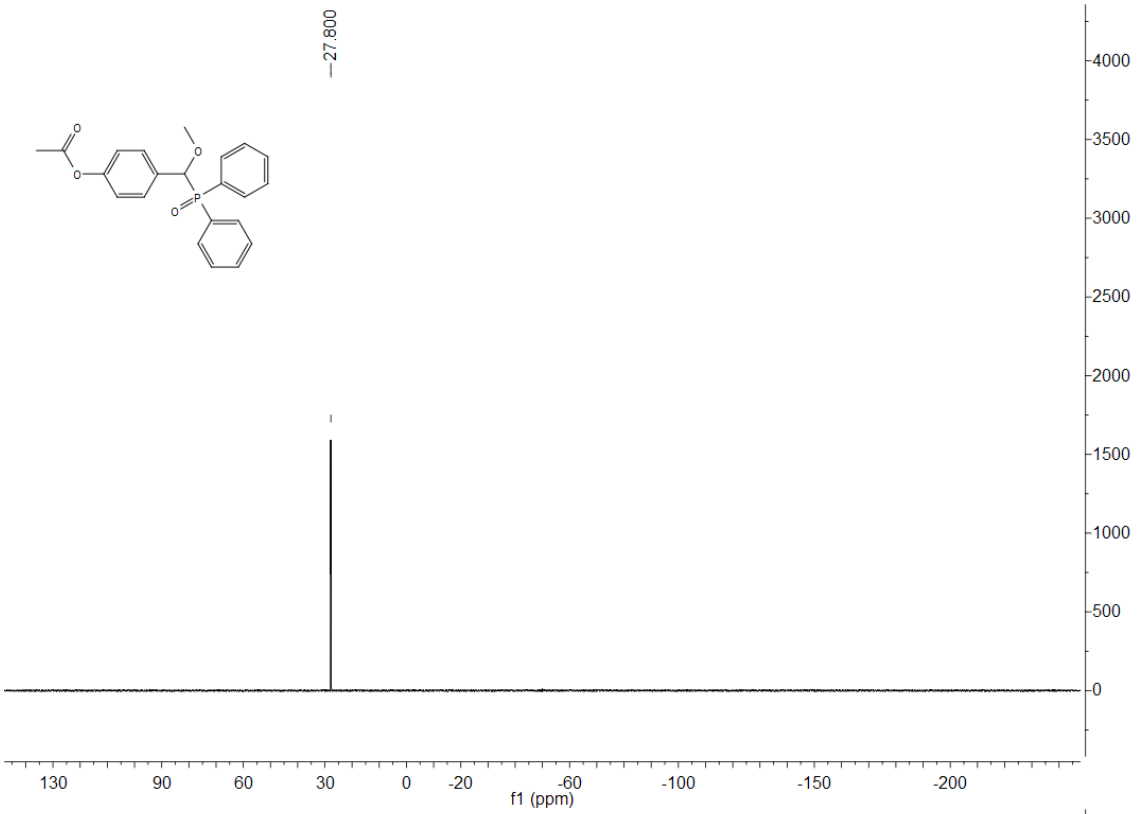
2.96
f1 (ppm)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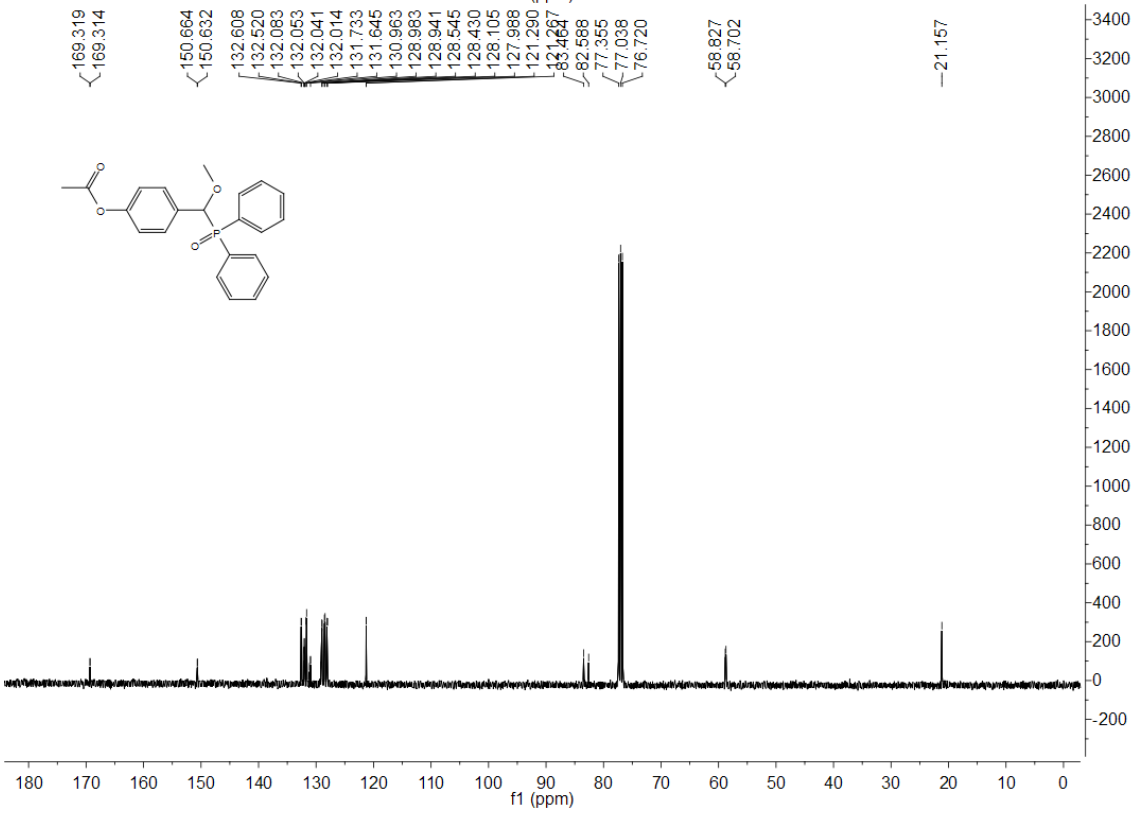
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150.632
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132.083
132.053
132.041
132.014
131.733
131.645
130.963
128.983
128.941
128.545
128.430
128.105
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121.290
83.464
82.588
77.355
77.038
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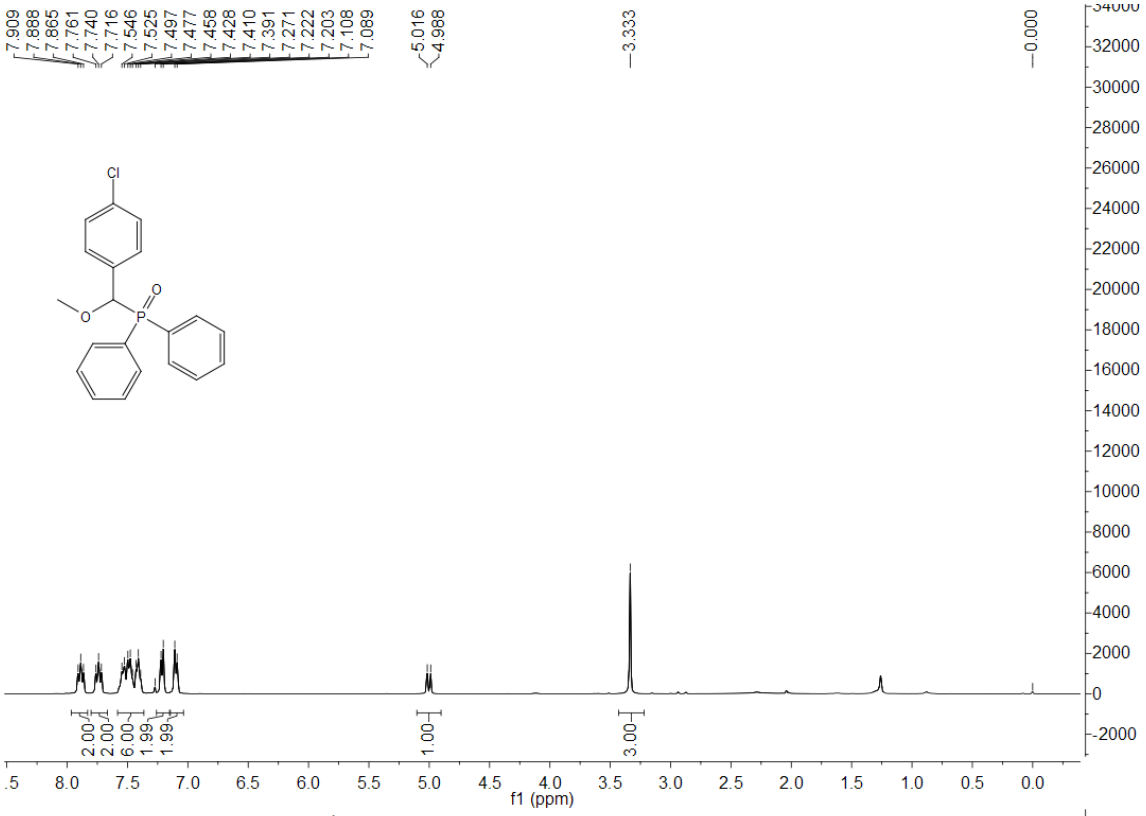
58.827
58.702
21.157
f1 (ppm)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7.888
7.865
7.761
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7.716
7.546
7.525
7.497
7.477
7.458
7.428
7.410
7.391
7.271
7.222
7.203
7.108
7.089
5.016
4.988
3.333
0.000
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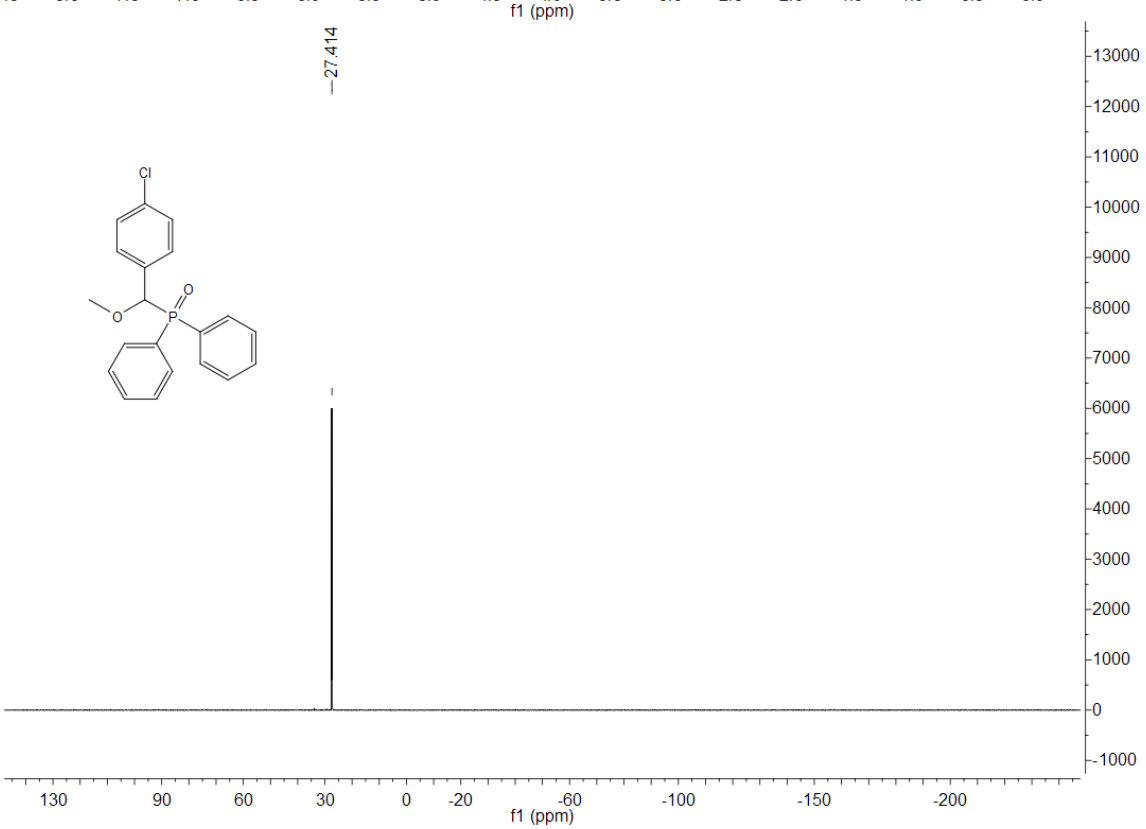
P
O
2.00
2.00
6.00
1.99
1.99
1.00
3.00
f1 (ppm)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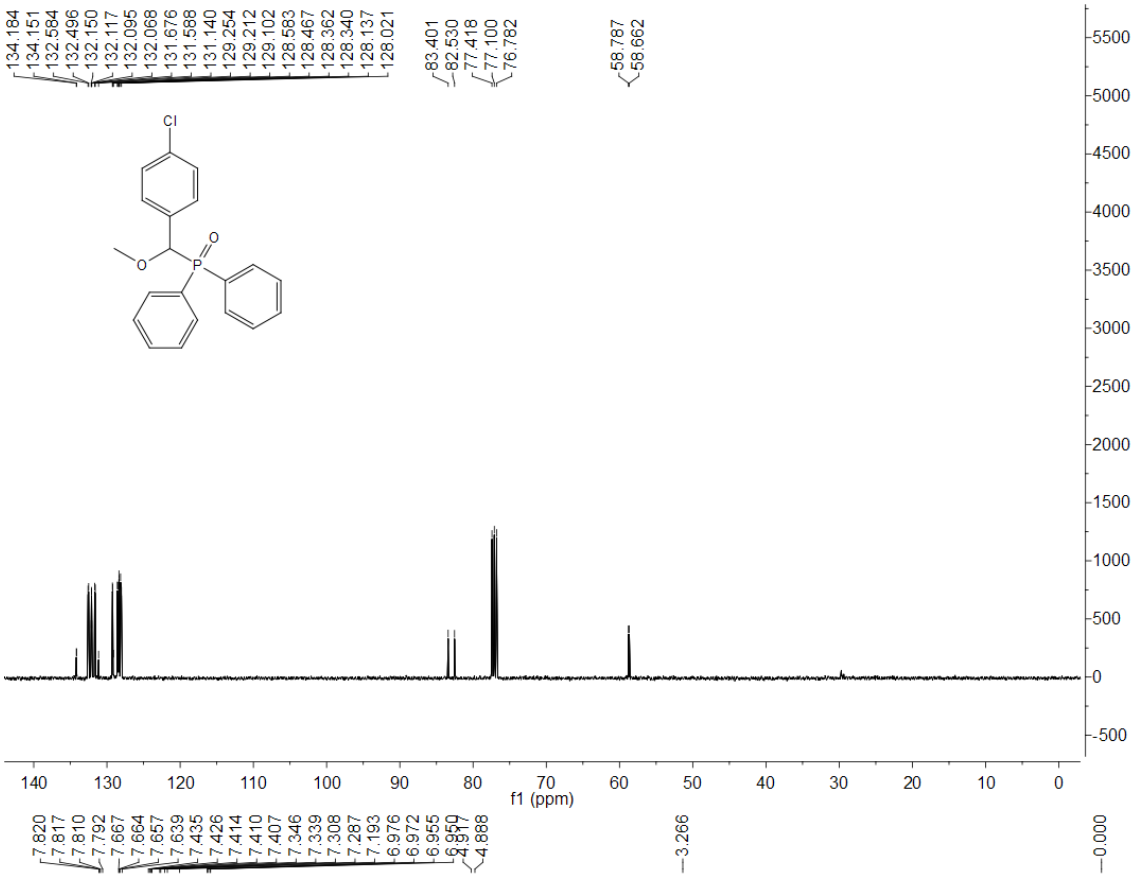
O
P
O
f1 (ppm)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131.140
129.254
129.212
129.102
128.583
128.467
128.362
128.340
128.137
128.021
83.401
82.530
77.418
77.100
76.782
58.787
58.662
Cl
O
O
P
f1 (ppm)

7.820
7.817
7.810
7.792
7.667
7.664
7.657
7.639
7.435
7.426
7.414
7.410
7.407
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7.308
7.287
7.193
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6.972
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4.888
3.266
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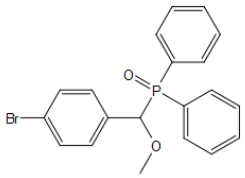
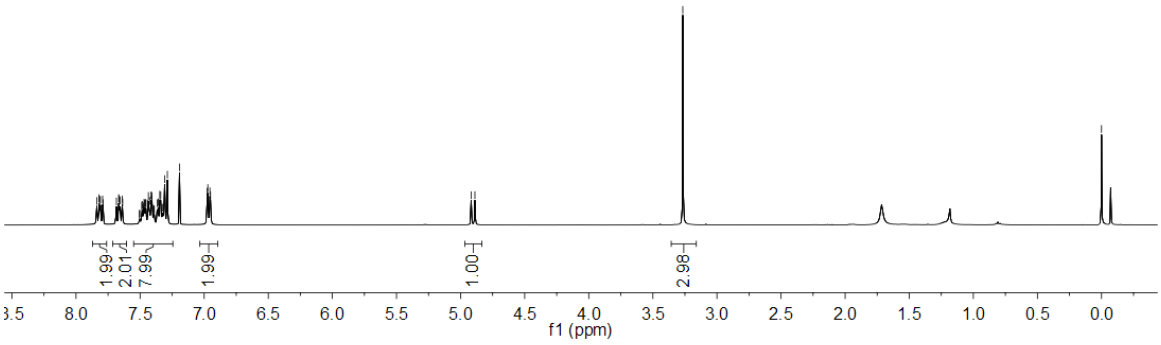

O
P
Br
O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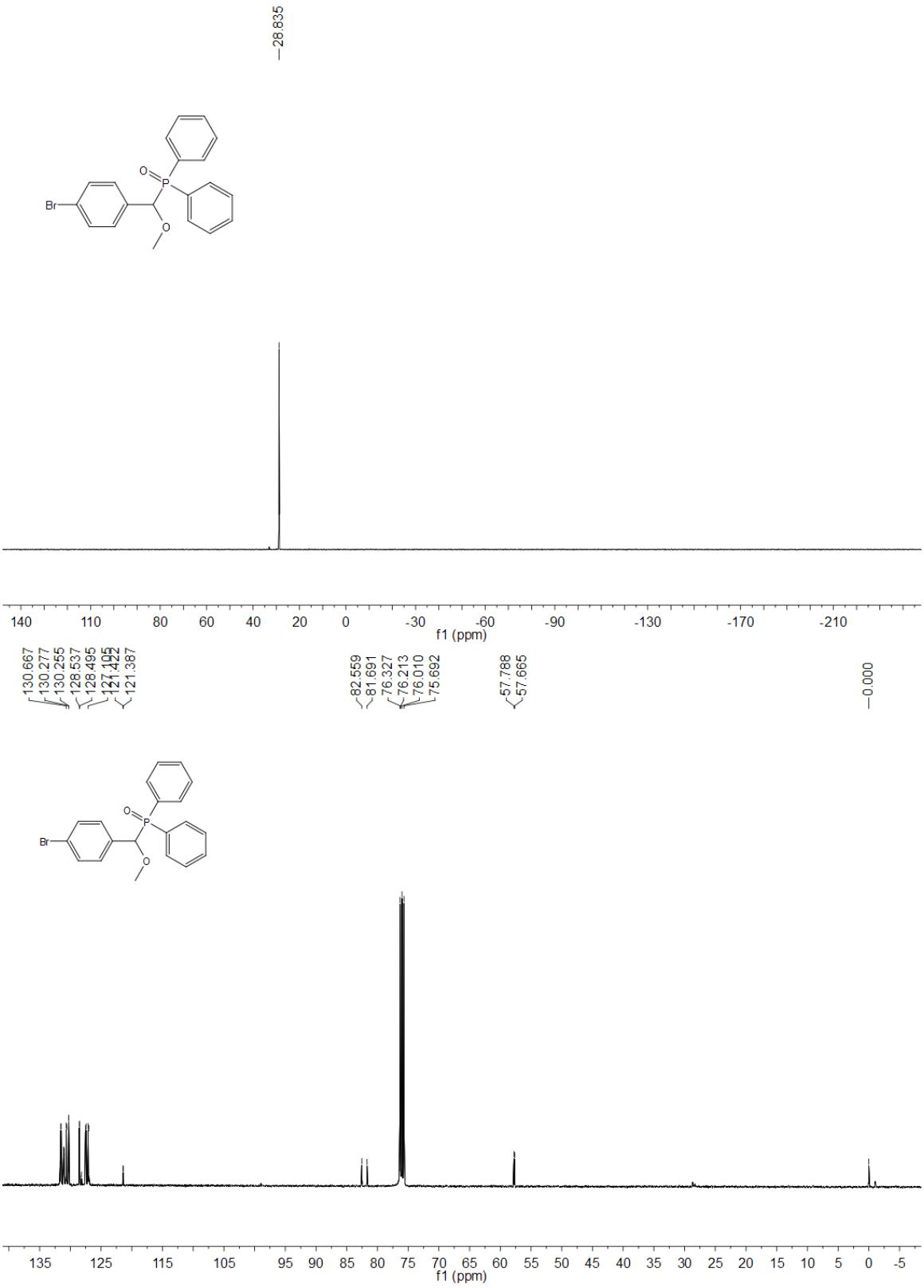
1.99
1.00
2.98
f1 (ppm)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Br
O
P
O
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210
f1 (ppm)
130.667
130.277
130.255
128.537
128.495
121.422
121.387
82.559
81.691
76.327
76.213
76.010
75.692
57.788
57.665
0.000
Br
O
P
O
135
125
115
105
95
90
85
80
75
70
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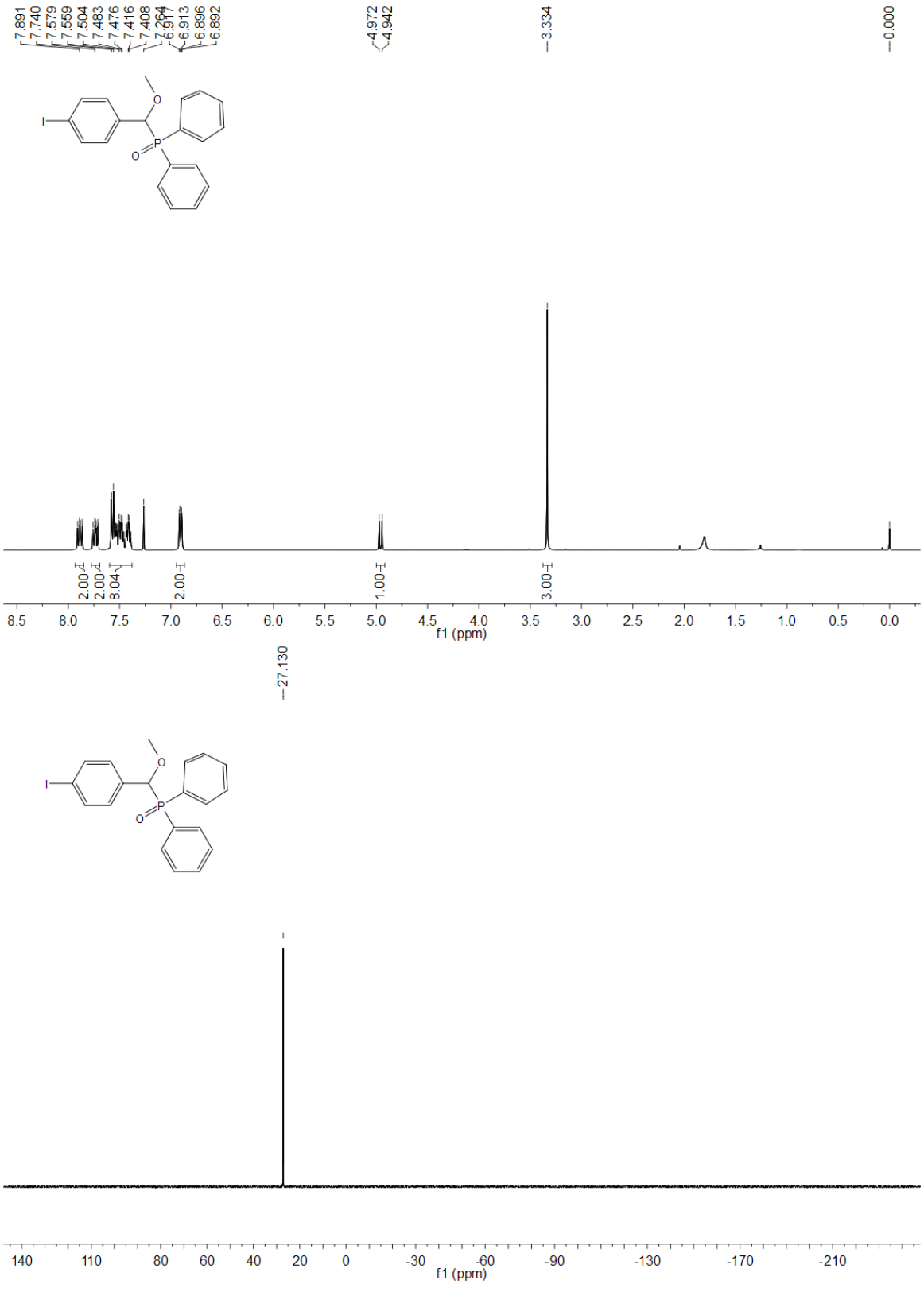
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
f1 (ppm)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7.579
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7.476
7.416
7.408
6.913
6.896
6.892
4.972
4.942
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2.00
2.00
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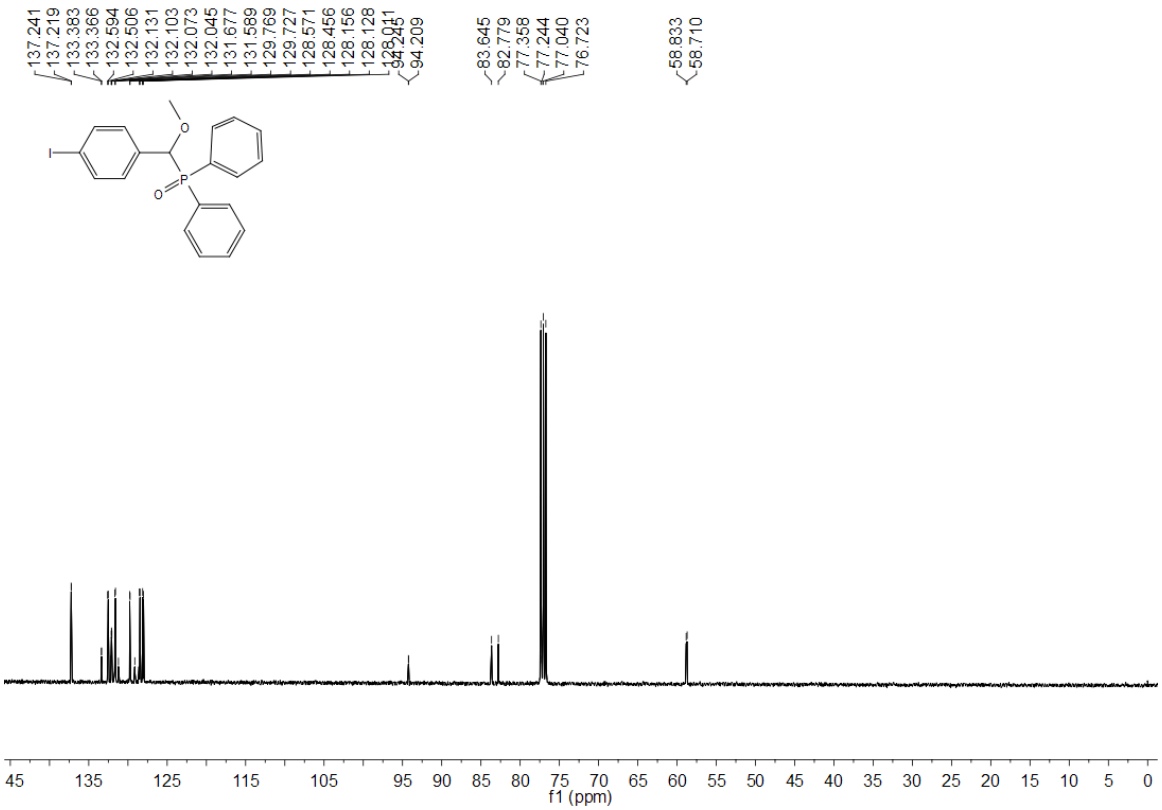
2.00
1.00
3.00
f1 (ppm)
27.130
f1 (ppm)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132.103
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132.045
131.677
131.589
129.769
129.727
128.571
128.456
128.156
128.128
94.245
94.209
83.645
82.779
77.358
77.244
77.040
76.723
58.833
58.710
I
O
P
O
45
135
125
115
105
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
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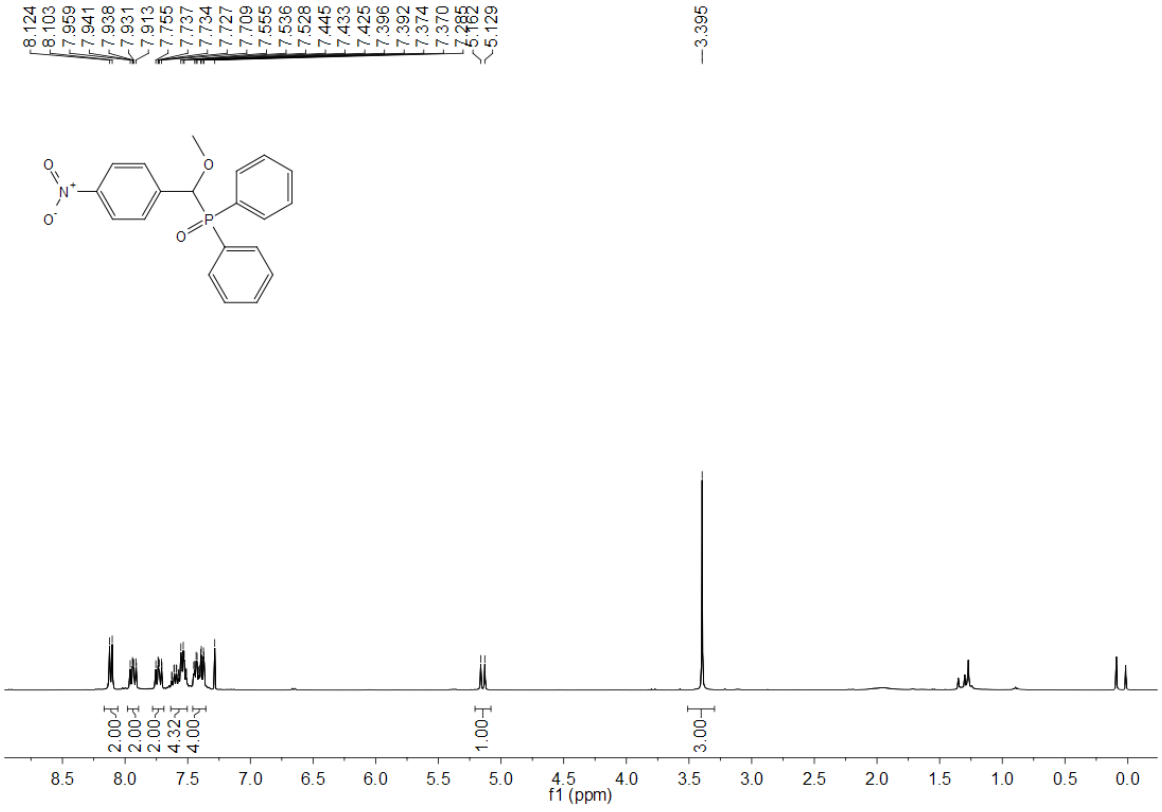
f1 (ppm)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7.555
7.536
7.528
7.445
7.433
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7.392
7.374
7.370
7.285
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7.0
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6.0
5.5
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2.5
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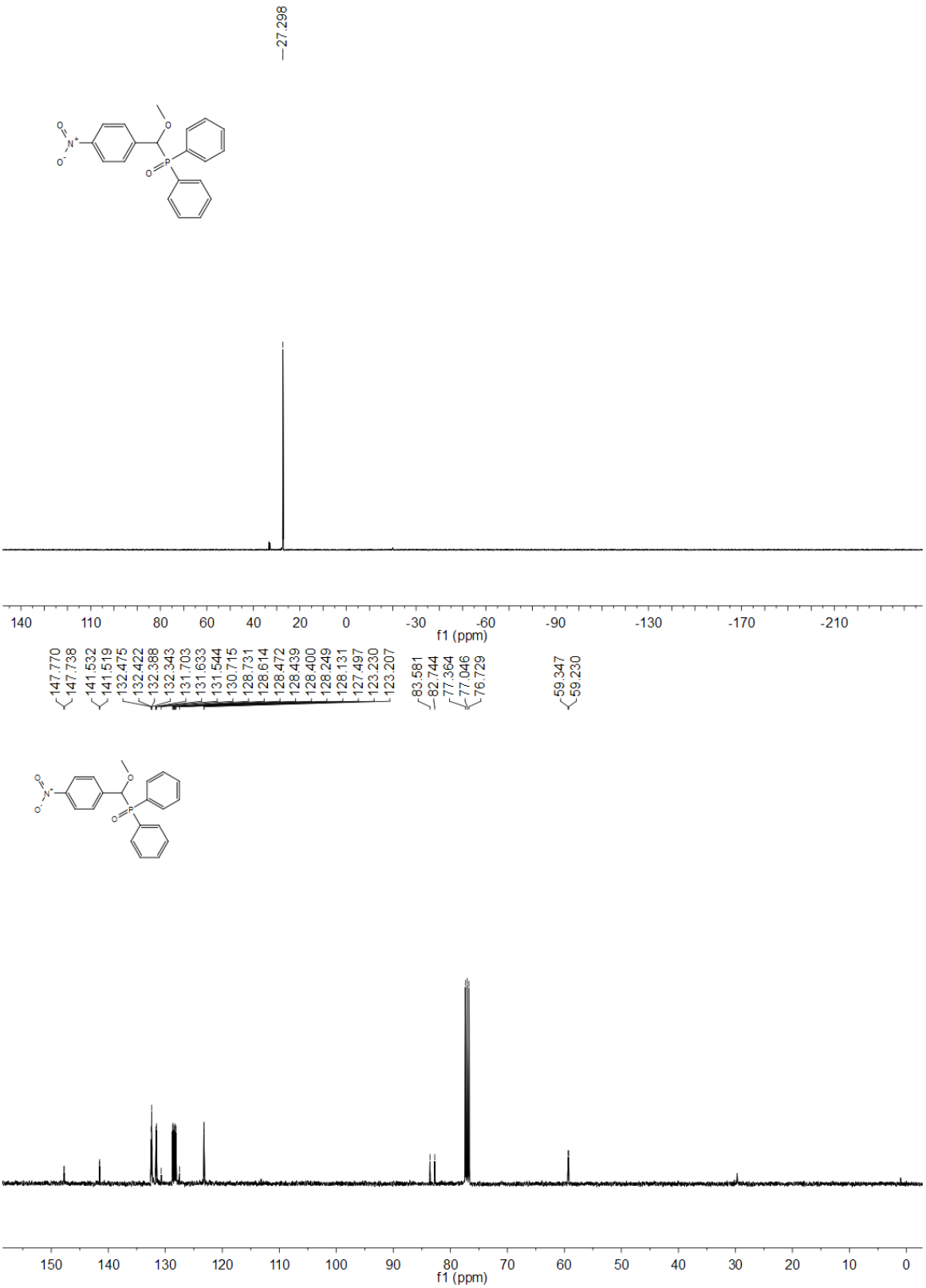
1.0
0.5
0.0
f1 (ppm)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f1 (ppm)
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131.544
130.715
128.731
128.614
128.472
128.439
128.400
128.249
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123.207
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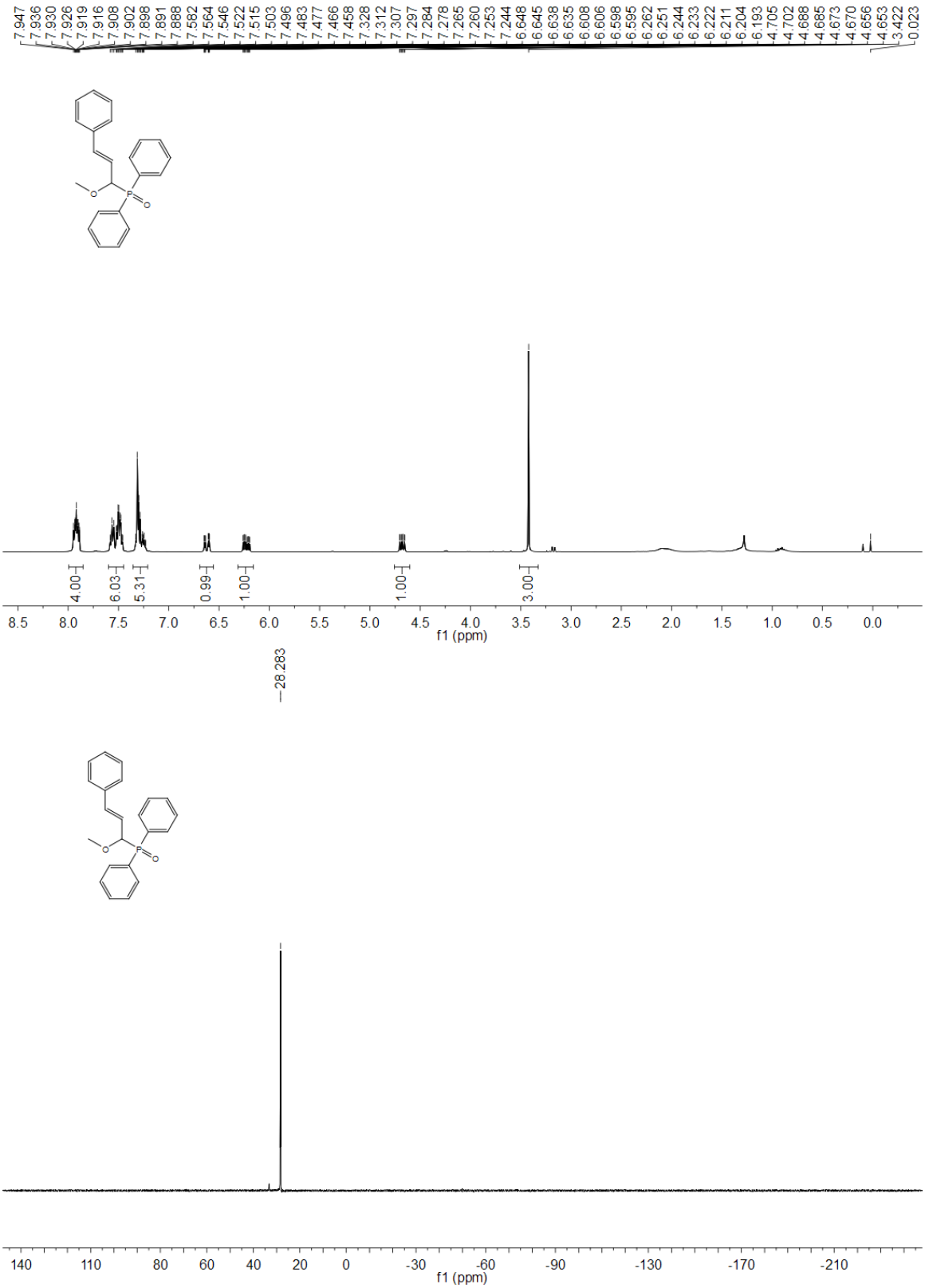
59.230
f1 (ppm)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7.564
7.546
7.522
7.515
7.503
7.496
7.483
7.477
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7.458
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7.307
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7.284
7.278
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7.253
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6.635
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6.598
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6.251
6.244
6.233
6.222
6.211
6.204
6.193
4.705
4.702
4.688
4.685
4.673
4.670
4.656
4.653
3.422
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3.00
f1 (ppm)
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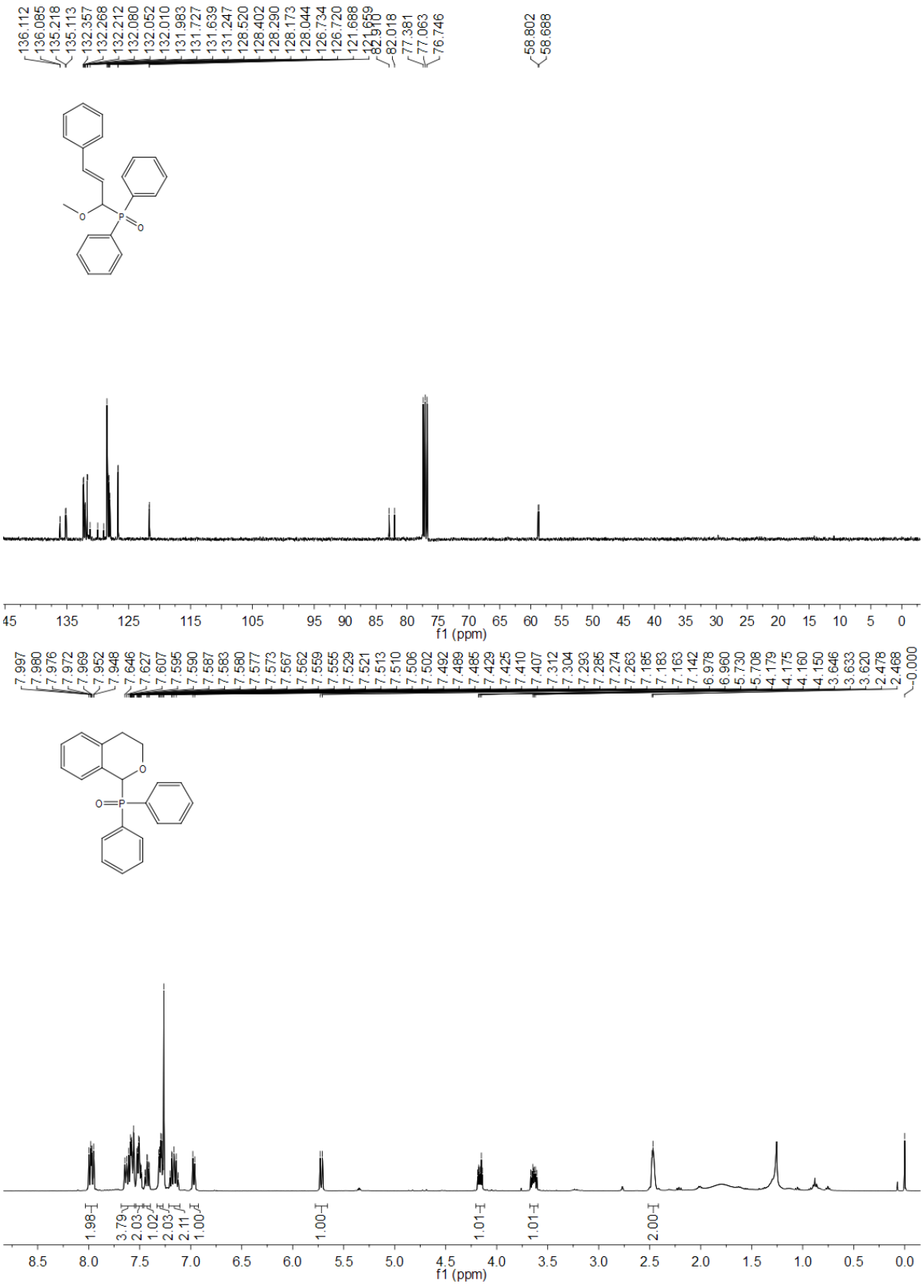
f1 (ppm)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131.247
128.520
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128.290
128.173
128.044
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126.720
121.688
121.659
82.910
82.018
77.381
77.063
76.746
58.802
58.688
O
P
O
45 135 125 115 105 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)
7.997
7.980
7.976
7.972
7.969
7.952
7.948
7.646
7.627
7.607
7.595
7.590
7.587
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3.620
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O
P
1.98
3.79
2.03
1.02
2.03
2.11
1.00
1.00
1.01
1.01
2.00
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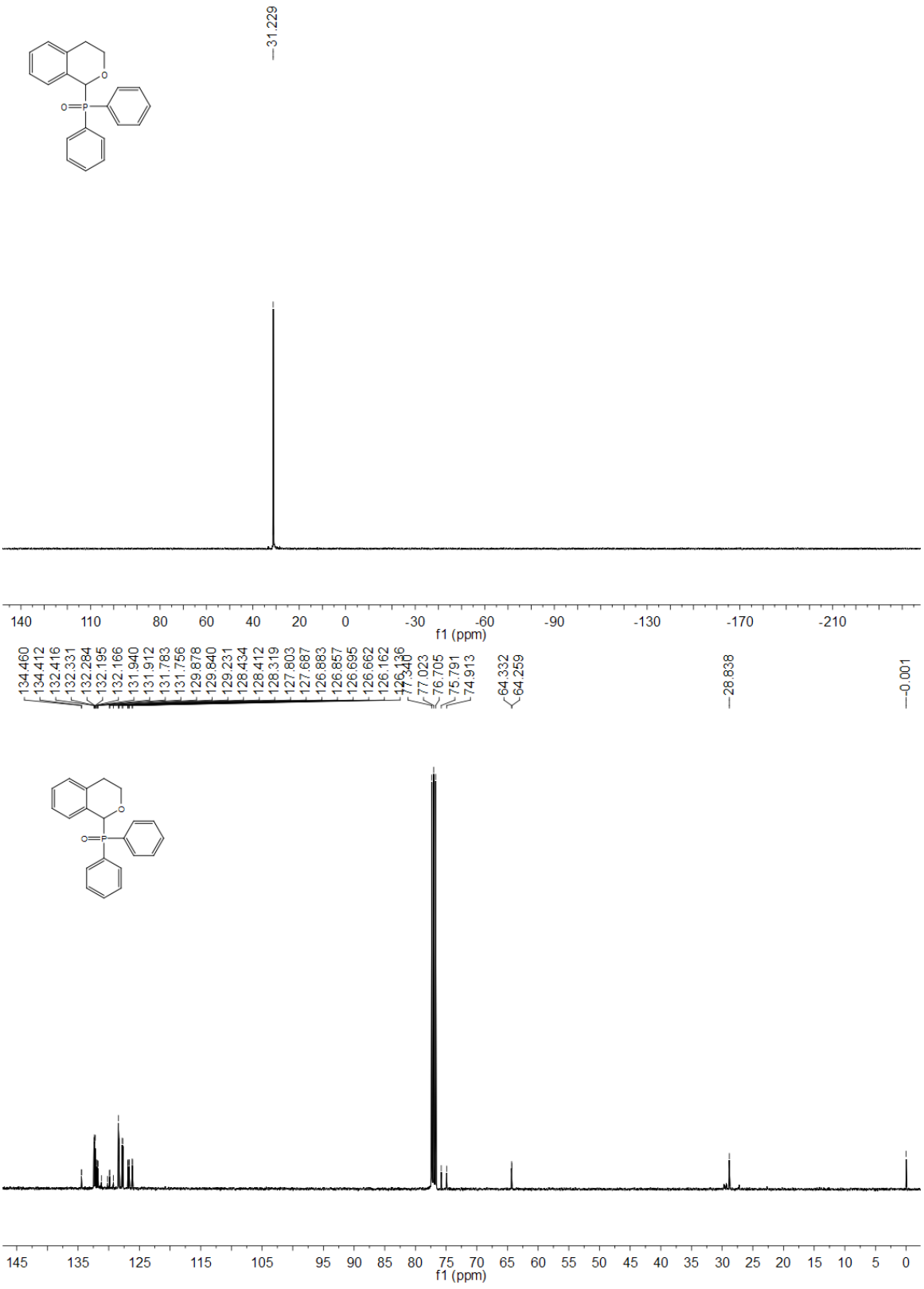
f1 (ppm)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129.878
129.840
129.231
128.434
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128.319
127.803
127.687
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126.695
126.662
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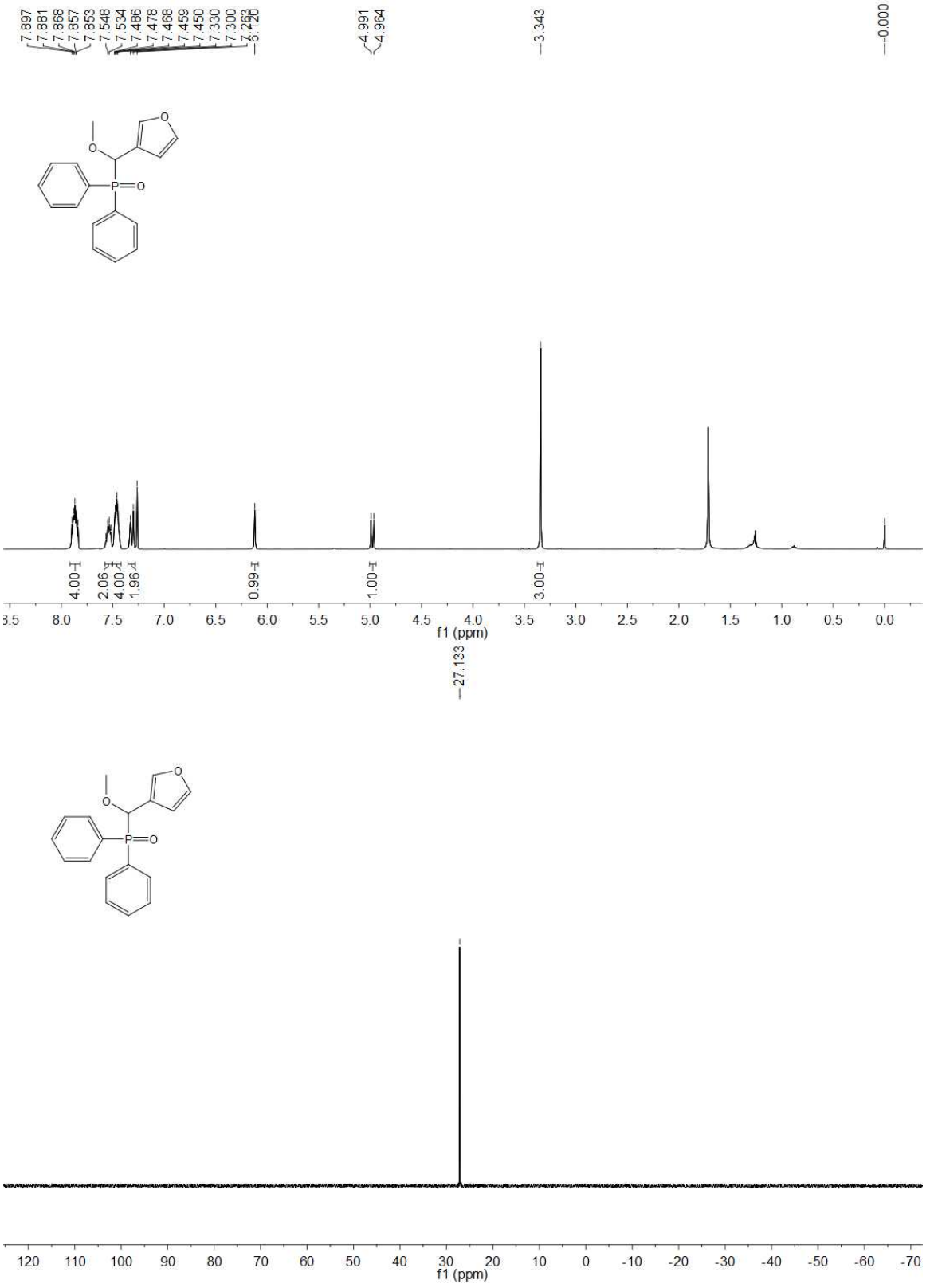
f1 (ppm)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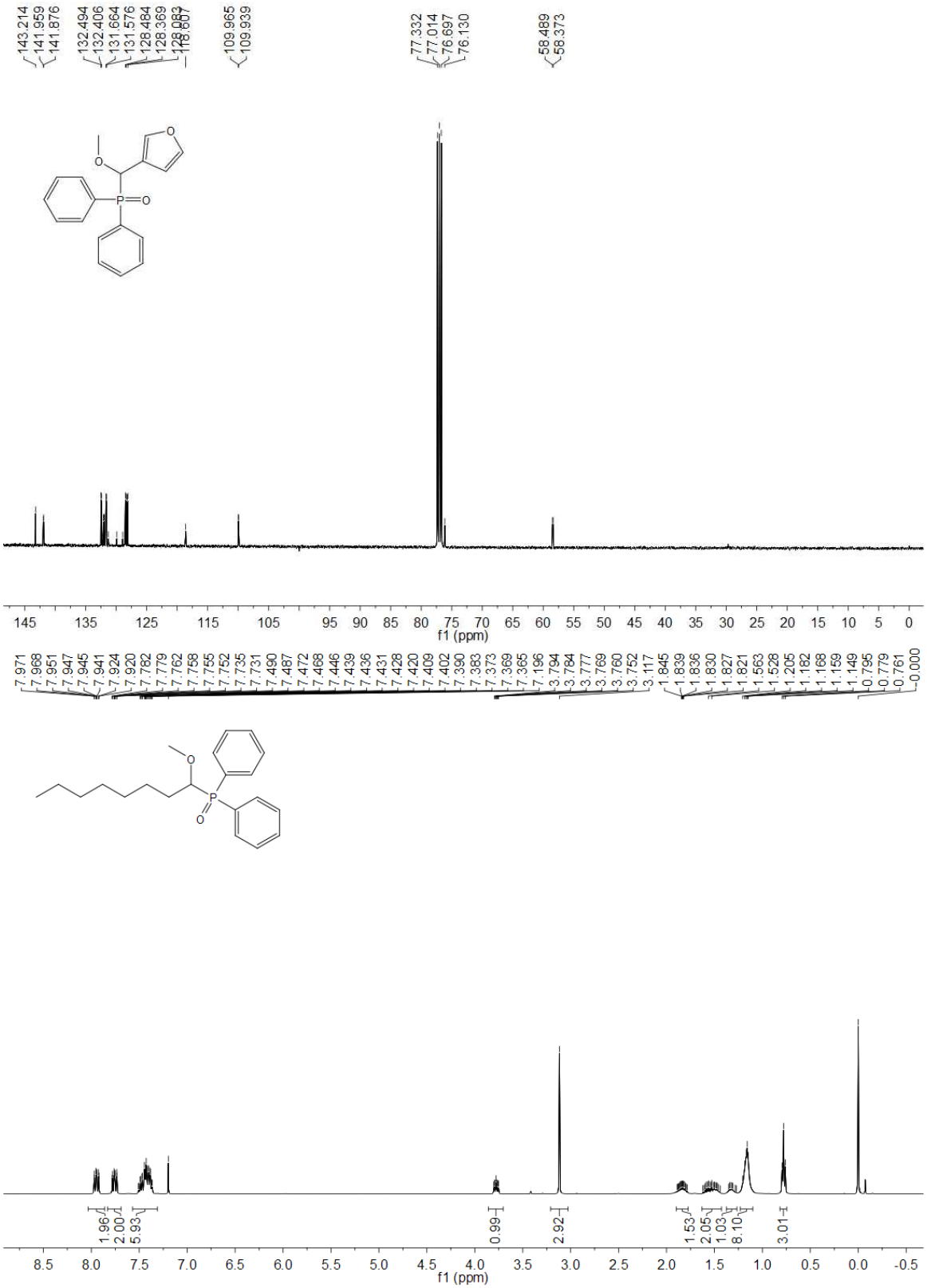
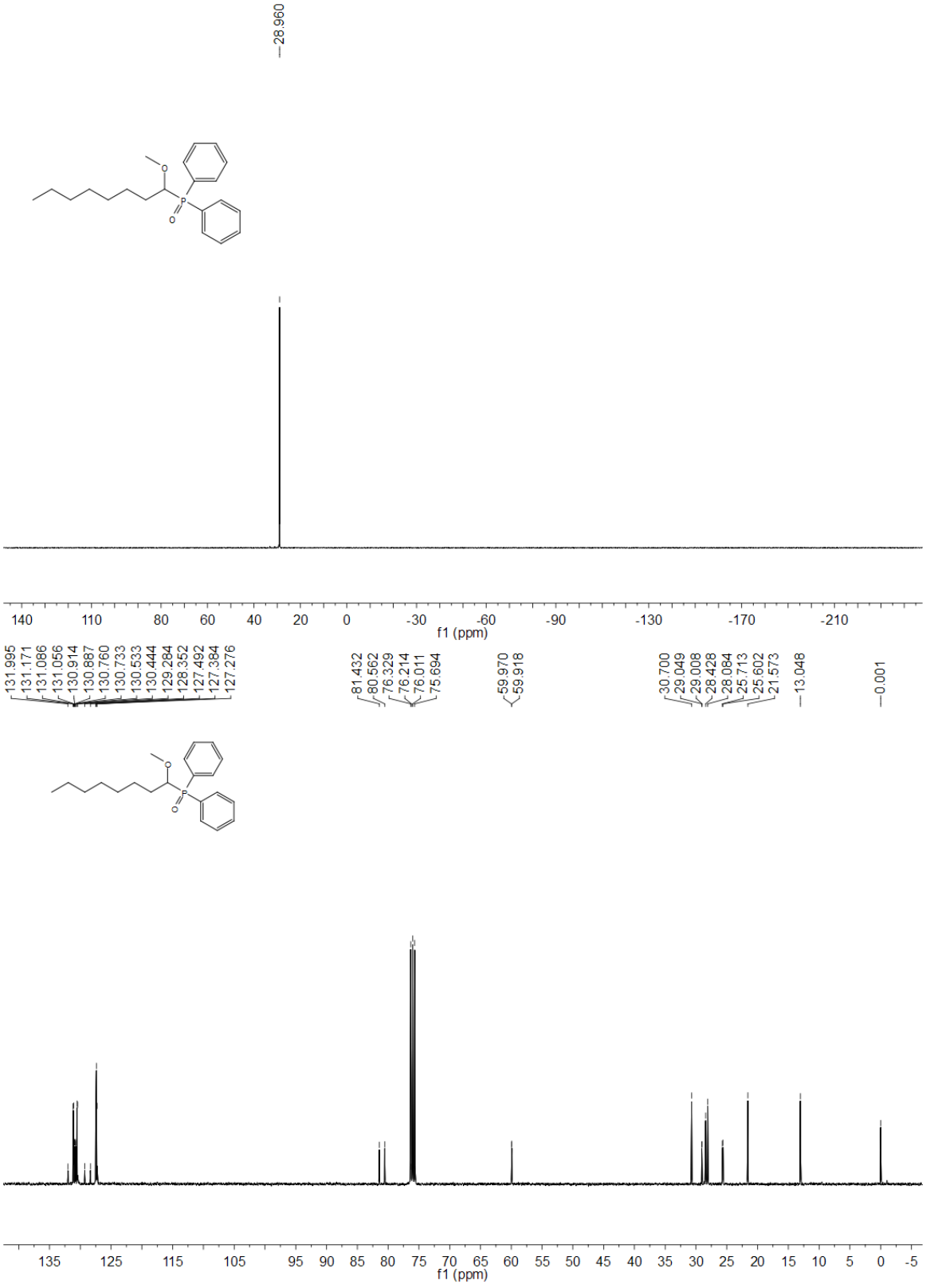
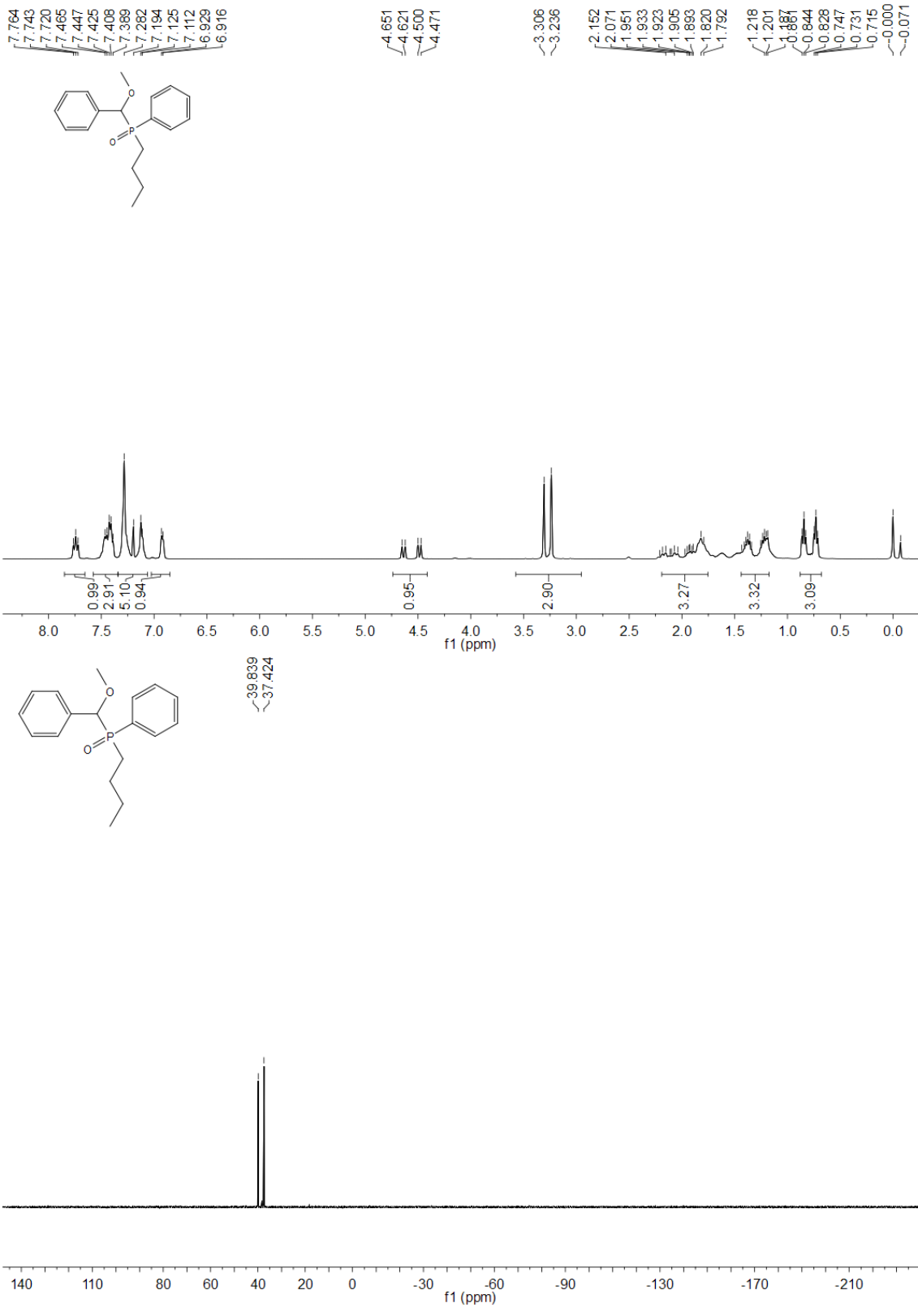
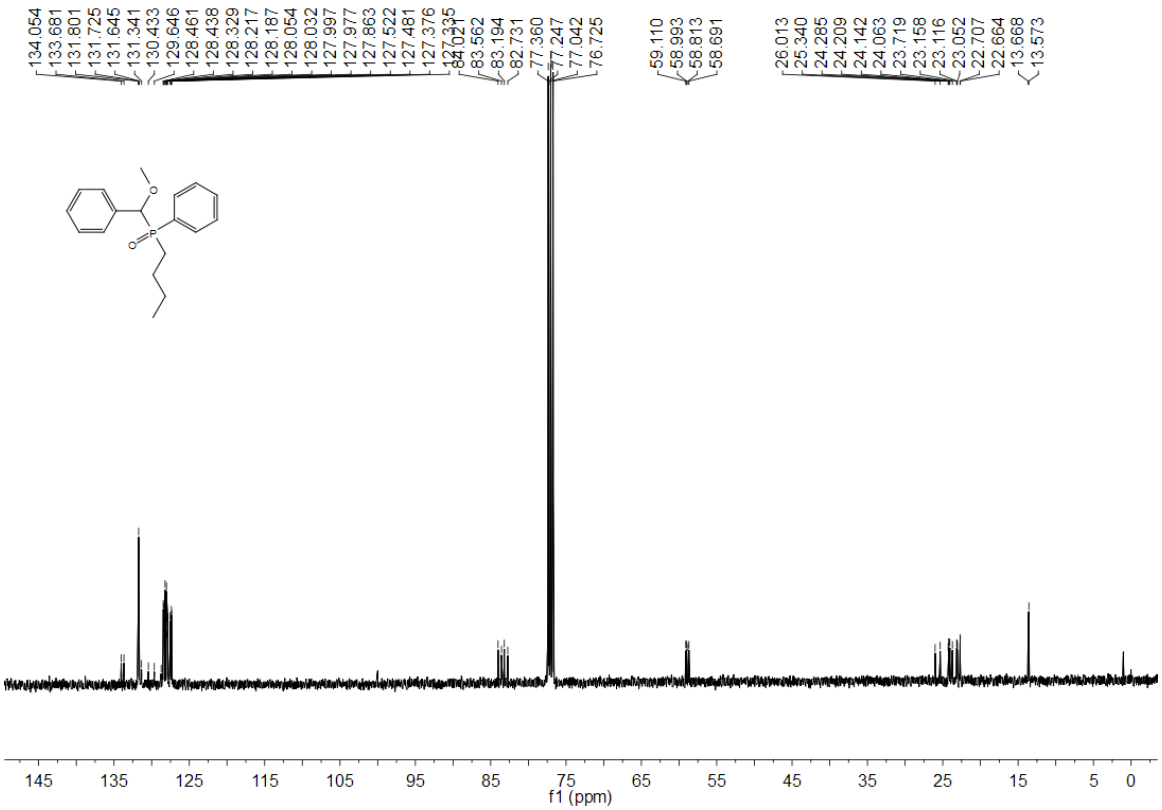
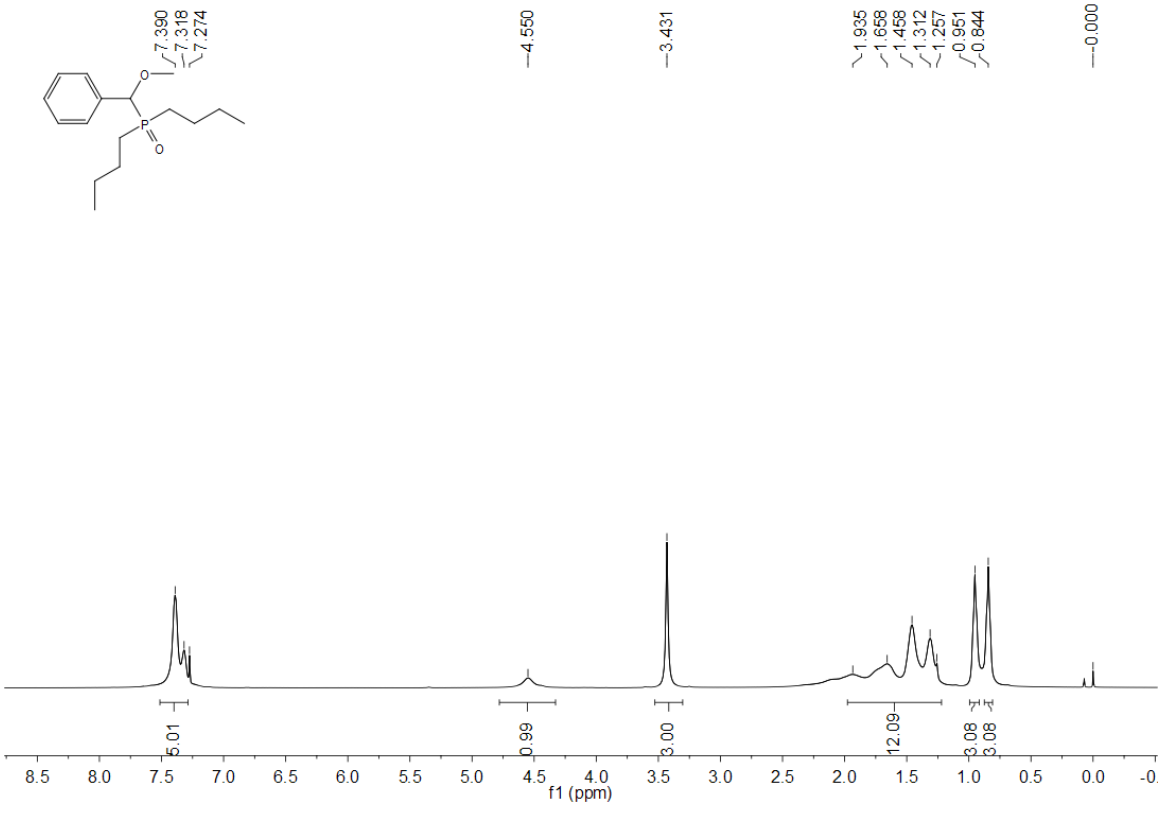
7.881
7.868
7.857
7.853
7.548
7.534
7.486
7.478
7.468
7.459
7.450
7.330
7.300
7.263
6.120
4.991
4.964
3.343
0.000
O
O
P=O
4.00
2.06
4.00
1.96
0.99
1.00
3.00
f1 (ppm)
27.133
O
O
P=O
f1 (ppm)

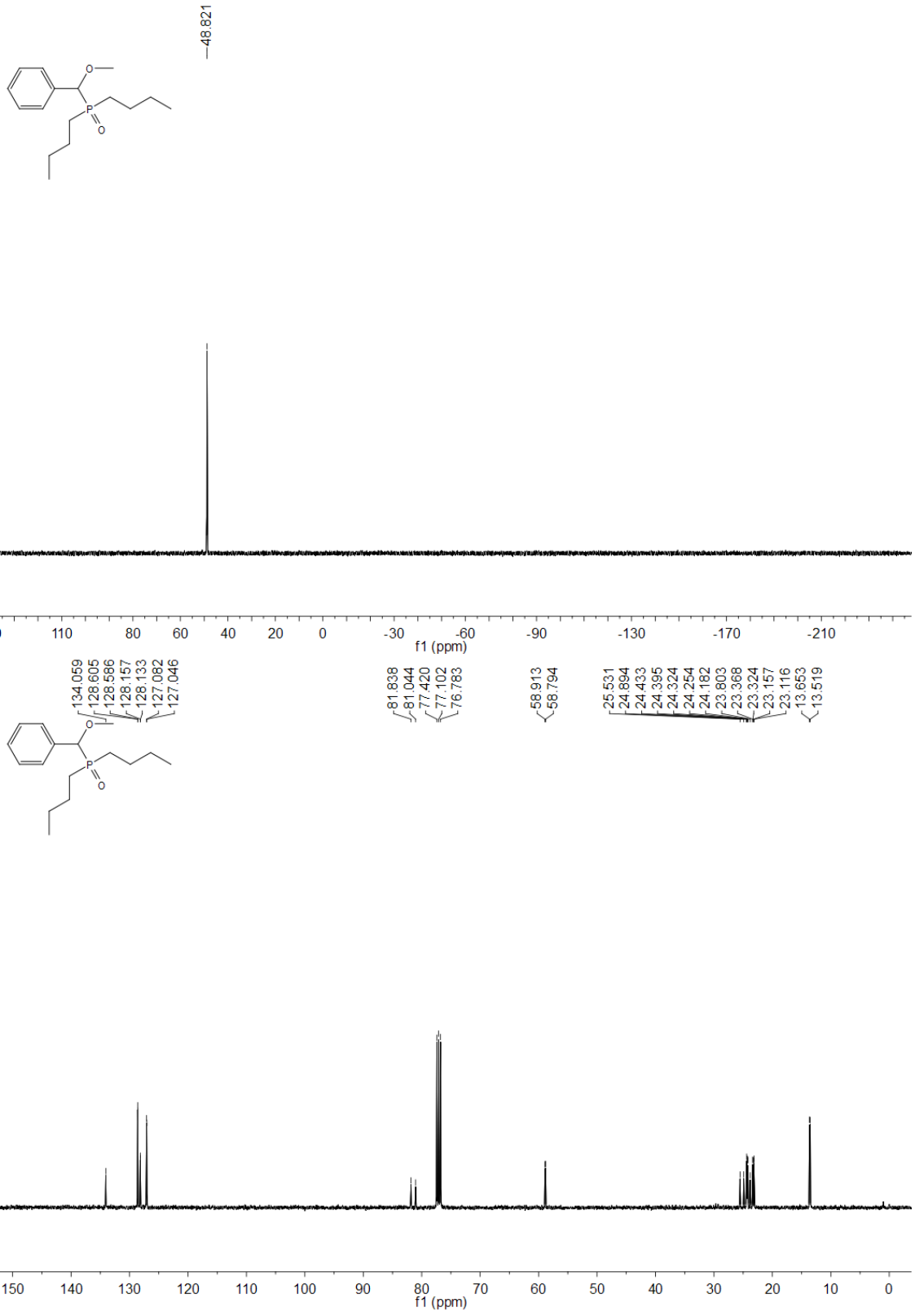

143.214
141.959
141.876
132.494
132.406
131.664
131.576
128.484
128.369
128.083
118.807
109.965
109.939
77.332
77.014
76.697
76.130
58.489
58.373
O
O
P
O
f1 (ppm)
7.971
7.968
7.951
7.947
7.945
7.941
7.924
7.920
7.782
7.779
7.762
7.758
7.755
7.752
7.735
7.731
7.490
7.487
7.472
7.468
7.446
7.439
7.436
7.431
7.428
7.420
7.409
7.402
7.390
7.383
7.373
7.369
7.365
7.196
3.794
3.784
3.777
3.769
3.760
3.752
3.117
1.845
1.839
1.836
1.830
1.827
1.821
1.563
1.528
1.205
1.182
1.168
1.159
1.149
0.795
0.779
0.761
0.000
O
P
O
1.96
2.00
5.93
0.99
2.92
1.53
2.05
1.03
8.10
3.01
f1 (ppm)

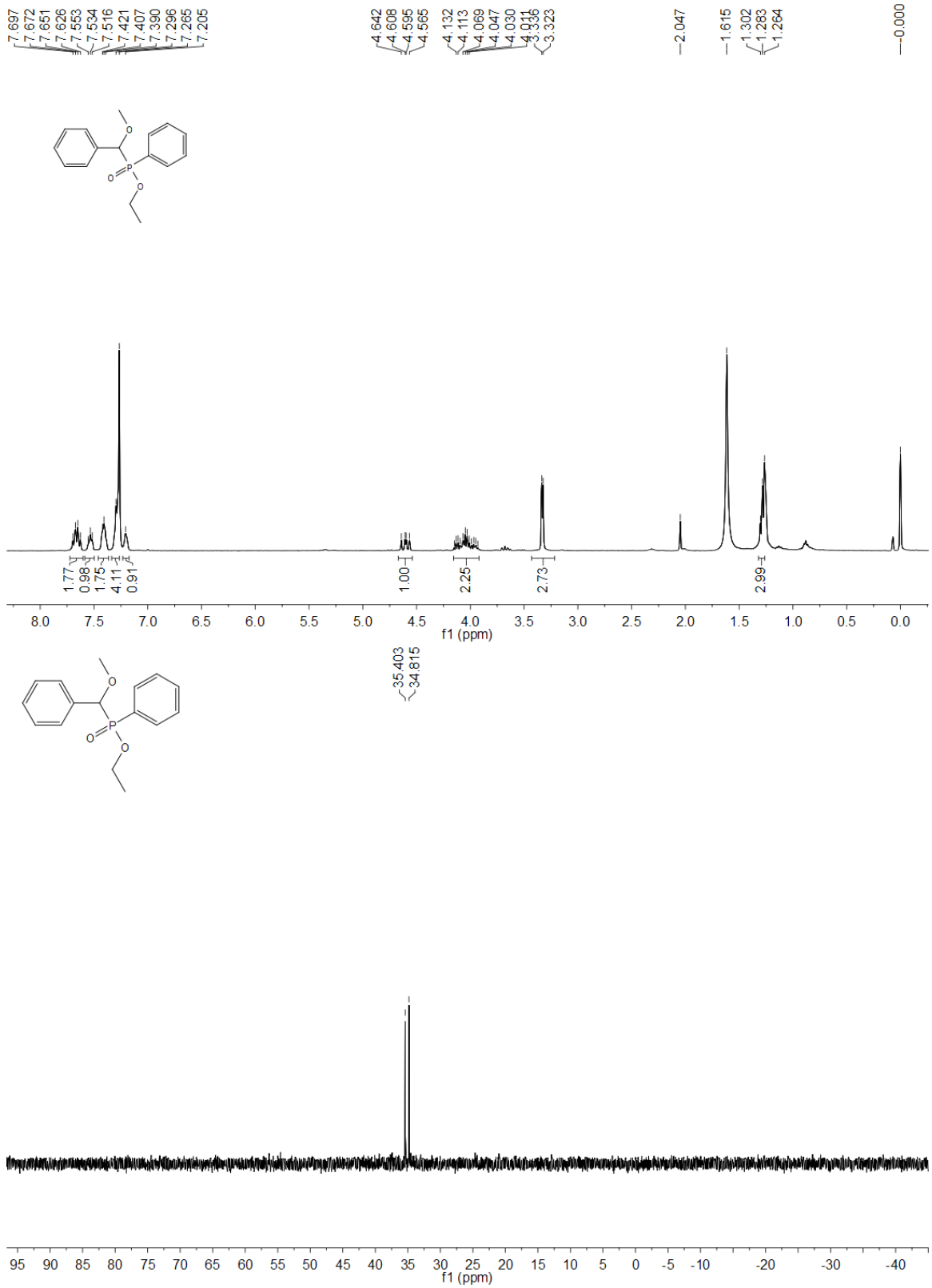

28.960
f1 (ppm)
131.995
131.171
131.086
131.056
130.914
130.887
130.760
130.733
130.533
130.444
129.284
128.352
127.492
127.384
127.276
81.432
80.562
76.329
76.214
76.011
75.694
59.970
59.918
30.700
29.049
29.008
28.428
28.084
25.713
25.602
21.573
13.048
0.001
f1 (ppm)

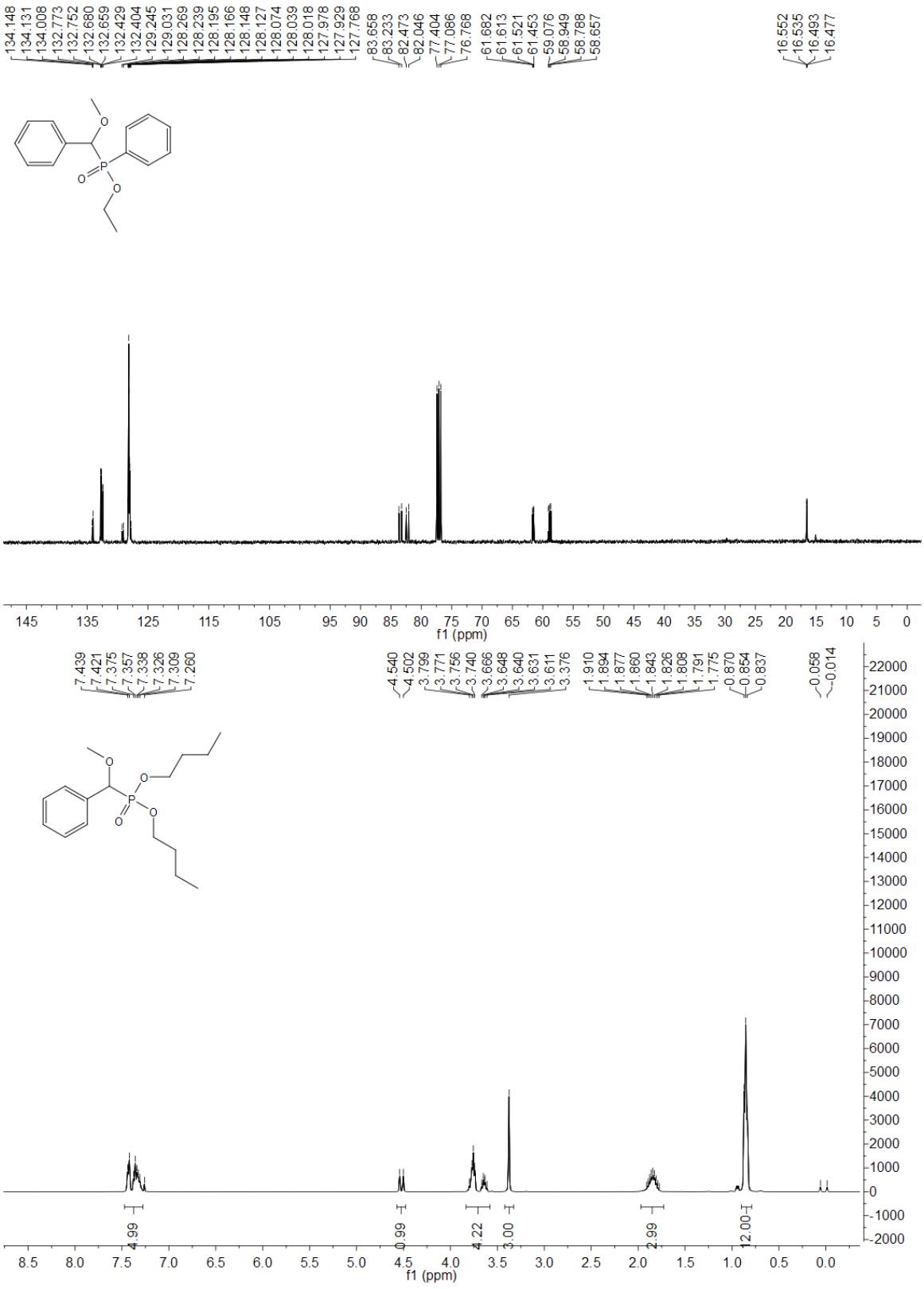

7.764
7.743
7.720
7.465
7.447
7.425
7.408
7.389
7.282
7.194
7.125
7.112
6.929
6.916
4.651
4.621
4.500
4.471
3.306
3.236
2.152
2.071
1.951
1.933
1.923
1.905
1.893
1.820
1.792
1.218
1.201
1.187
0.881
0.844
0.828
0.747
0.731
0.715
-0.000
-0.071
O
O
P
0.99
2.91
5.10
0.94
0.95
2.90
3.27
3.32
3.09
8.0
7.5
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)
39.839
37.424
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210
f1 (ppm)

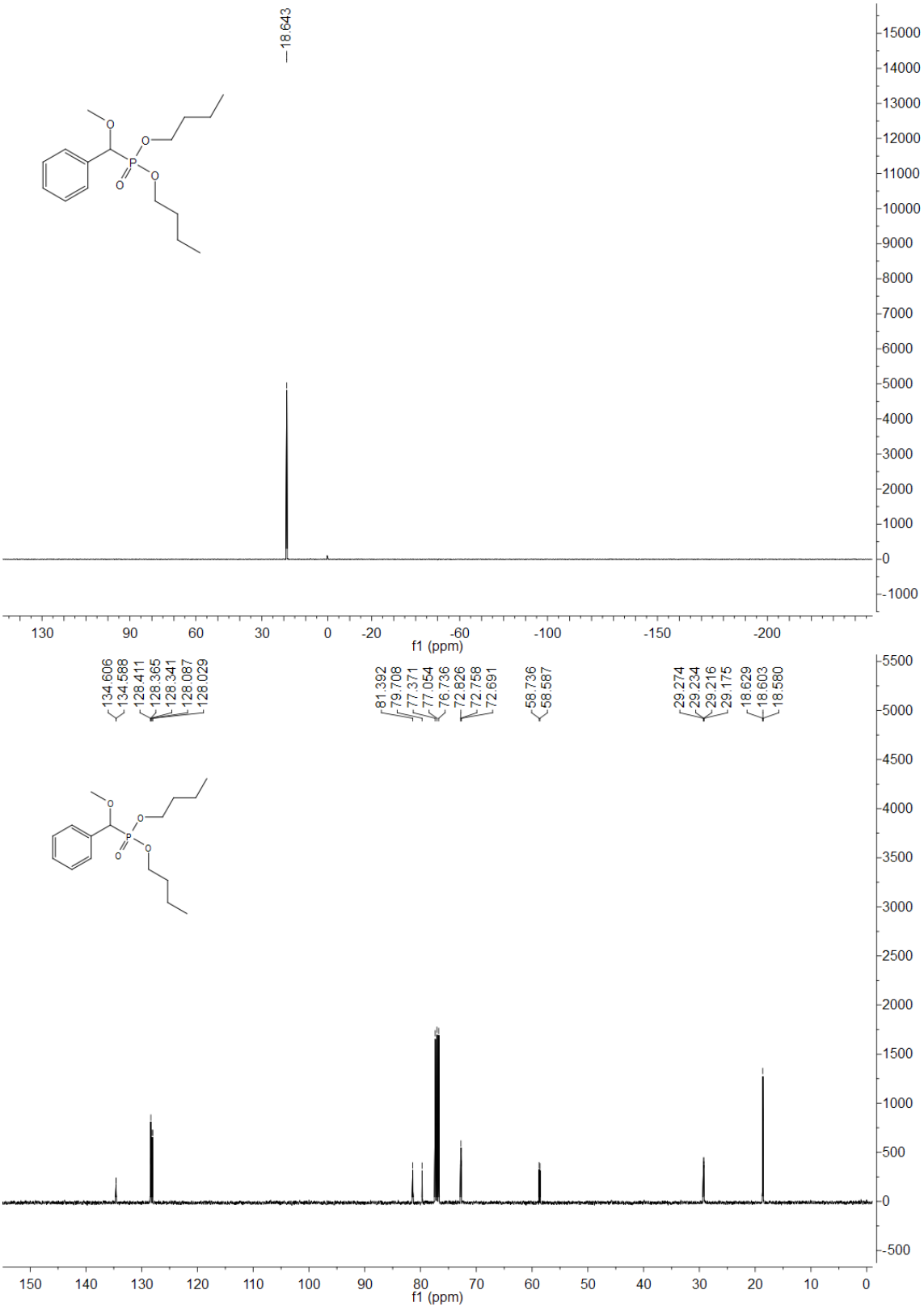

134.054
133.681
131.801
131.725
131.645
131.341
130.433
129.646
128.461
128.438
128.329
128.217
128.187
128.054
128.032
127.997
127.977
127.863
127.522
127.481
127.376
127.335
84.021
83.562
83.194
82.731
77.360
77.247
77.042
76.725
59.110
58.993
58.813
58.691
26.013
25.340
24.285
24.209
24.142
24.063
23.719
23.158
23.116
23.052
22.707
22.664
13.668
13.573
f1 (ppm)

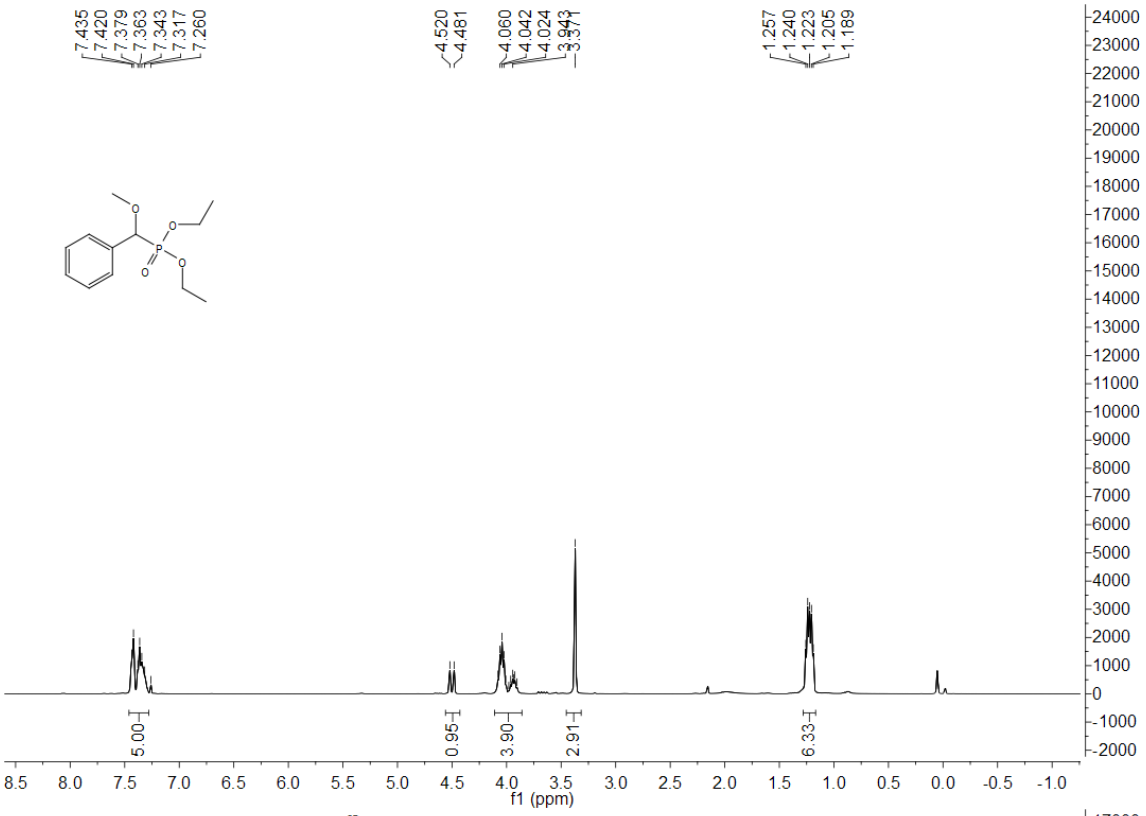

7.390
7.318
7.274
4.550
3.431
1.935
1.658
1.458
1.312
1.257
0.951
0.844
0.000
5.01
0.99
3.00
12.09
3.08
3.08
f1 (ppm)

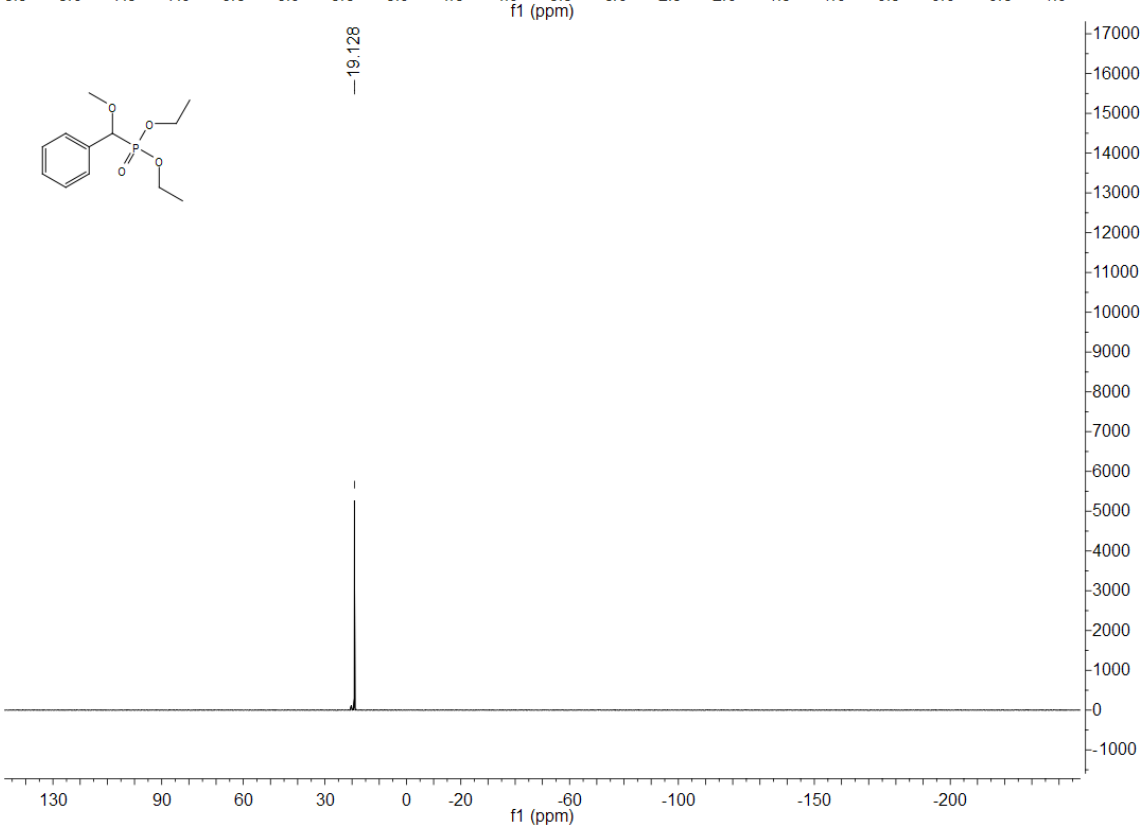

48.821
O
P
O
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210
f1 (ppm)
134.059
128.605
128.586
128.157
128.133
127.082
127.046
81.838
81.044
77.420
77.102
76.783
58.913
58.794
25.531
24.894
24.433
24.395
24.324
24.254
24.182
23.803
23.368
23.324
23.157
23.116
13.653
13.519
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

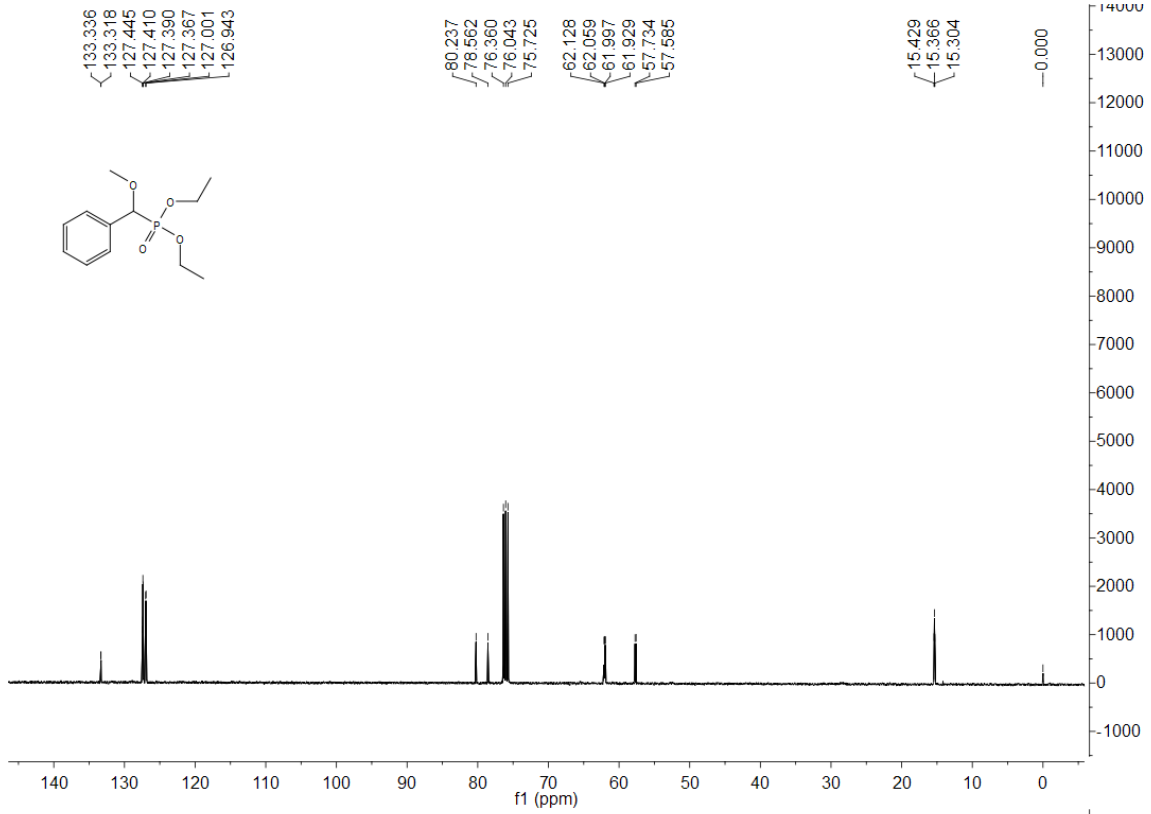

7.697
7.672
7.651
7.626
7.553
7.534
7.516
7.421
7.407
7.390
7.296
7.265
7.205
4.642
4.608
4.595
4.565
4.132
4.113
4.069
4.047
4.030
4.011
3.336
3.323
2.047
1.615
1.302
1.283
1.264
0.000
O
P
O
O
1.77
0.98
1.75
4.11
0.91
1.00
2.25
2.73
2.99
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)
35.403
34.815
95
90
85
80
75
70
65
60
55
50
45
40
35
30
25
20
15
10
5
0
-5
-10
-20
-30
-40
f1 (ppm)

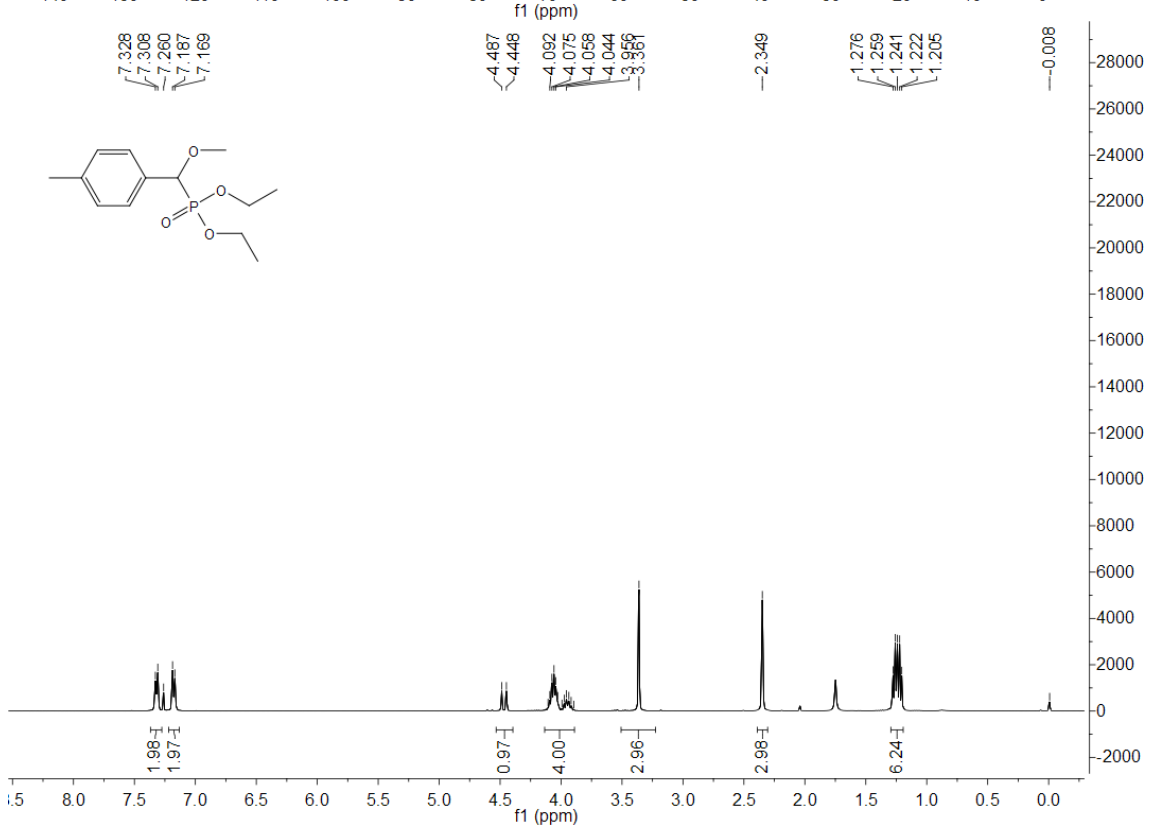

134.148
134.131
134.008
132.773
132.752
132.680
132.659
132.429
132.404
129.245
129.031
128.269
128.239
128.195
128.166
128.148
128.127
128.074
128.039
128.018
127.978
127.929
127.768
83.658
83.233
82.473
82.046
77.404
77.086
76.768
61.682
61.613
61.521
61.453
59.076
58.949
58.788
58.657
16.552
16.535
16.493
16.477
O
O
P
O
O
145 135 125 115 105 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10 5 0
f1 (ppm)
7.439
7.421
7.375
7.357
7.338
7.326
7.309
7.260
4.540
4.502
3.799
3.771
3.756
3.740
3.666
3.648
3.640
3.631
3.611
3.376
1.910
1.894
1.877
1.860
1.843
1.826
1.808
1.791
1.775
0.870
0.854
0.837
0.058
-0.014
O
O
P
O
O
22000
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
4.99
0.99
4.22
3.00
2.99
12.00
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)

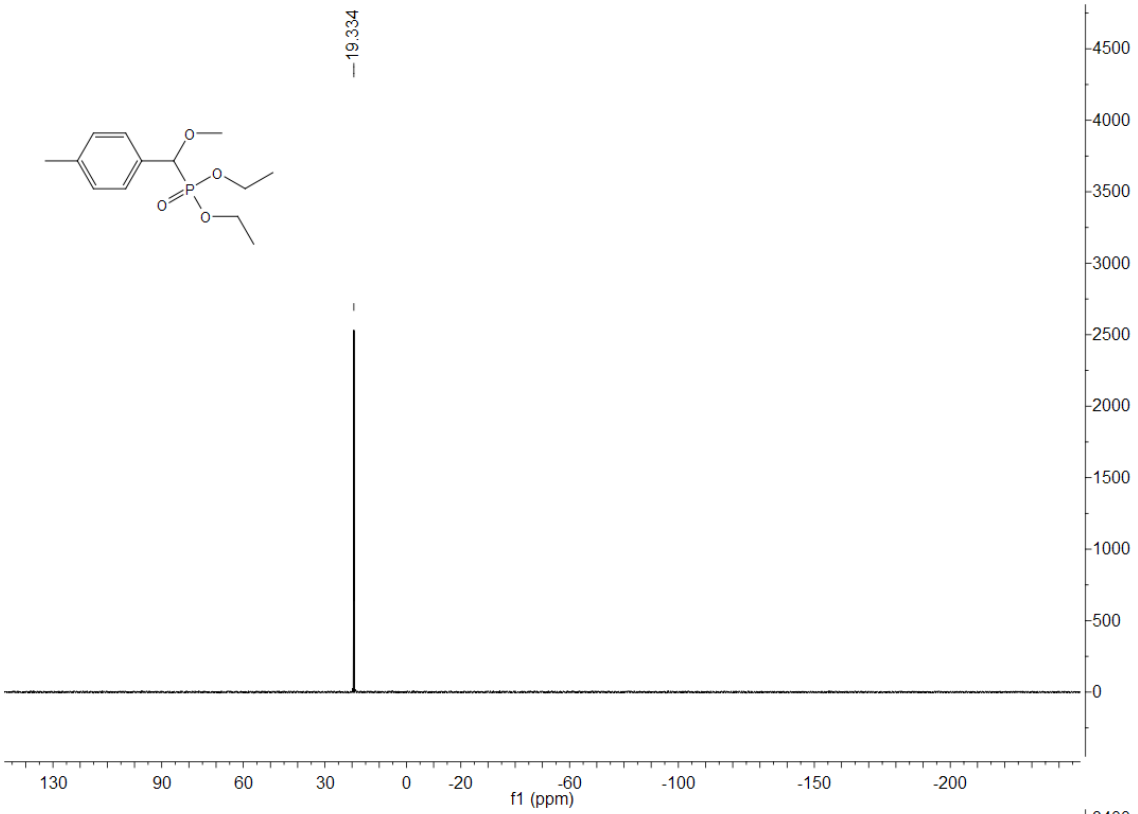

18.643
f1 (ppm)
134.606
134.588
128.411
128.365
128.341
128.087
128.029
81.392
79.708
77.371
77.054
76.736
72.826
72.758
72.691
58.736
58.567
29.274
29.234
29.216
29.175
18.629
18.603
18.580
f1 (ppm)

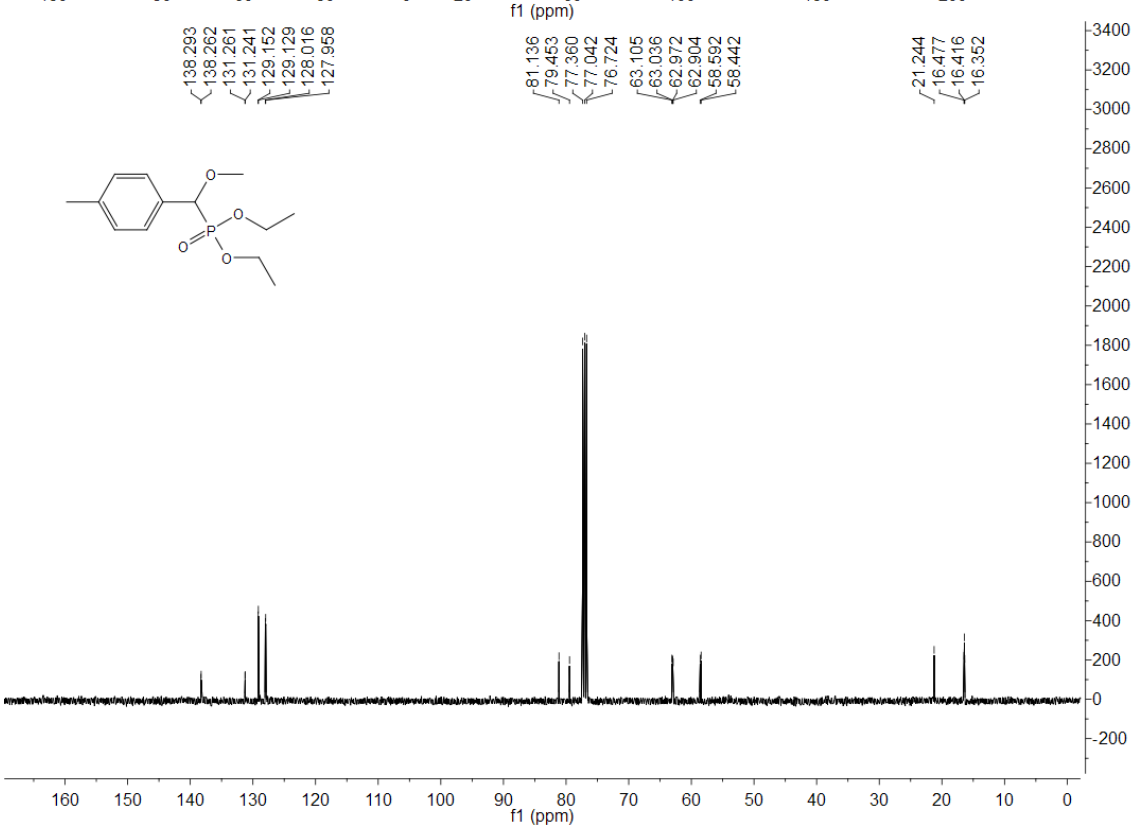

7.435
7.420
7.379
7.363
7.343
7.317
7.260
4.520
4.481
4.060
4.042
4.024
3.943
3.371
1.257
1.240
1.223
1.205
1.189
5.00
0.95
3.90
2.91
6.33
f1 (ppm)

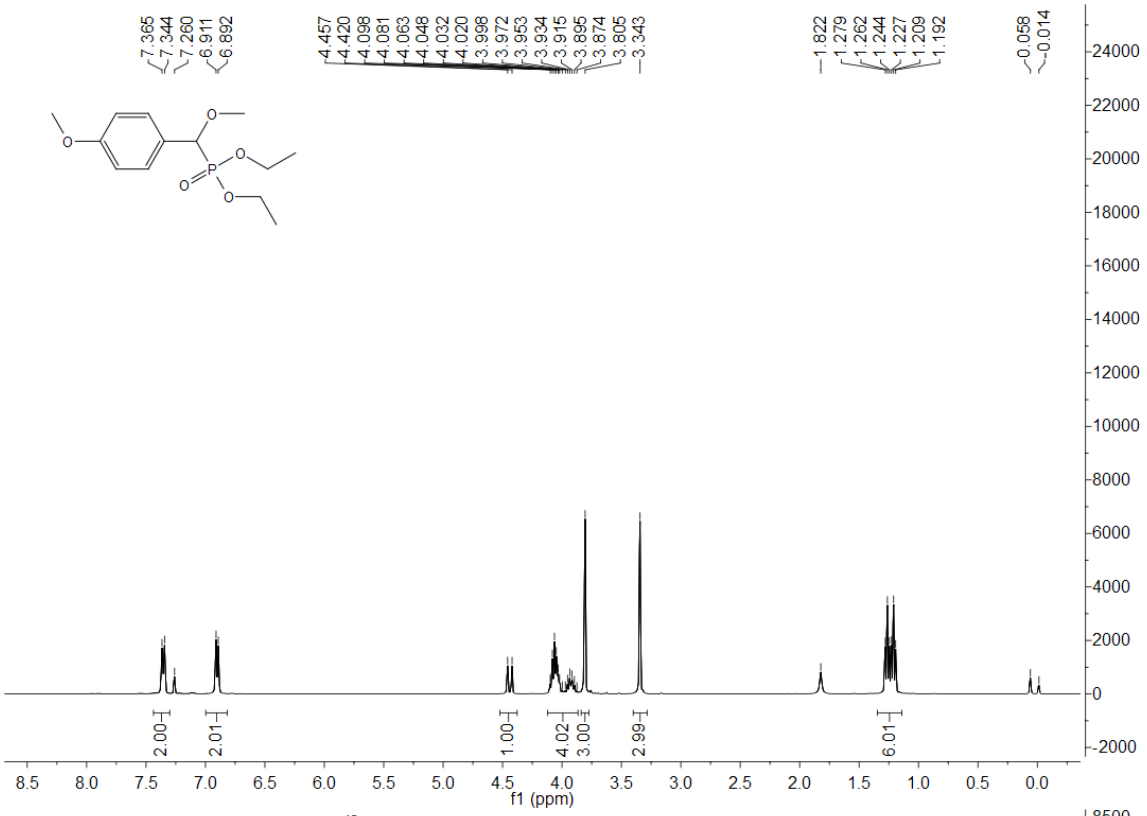

19.128
f1 (ppm)

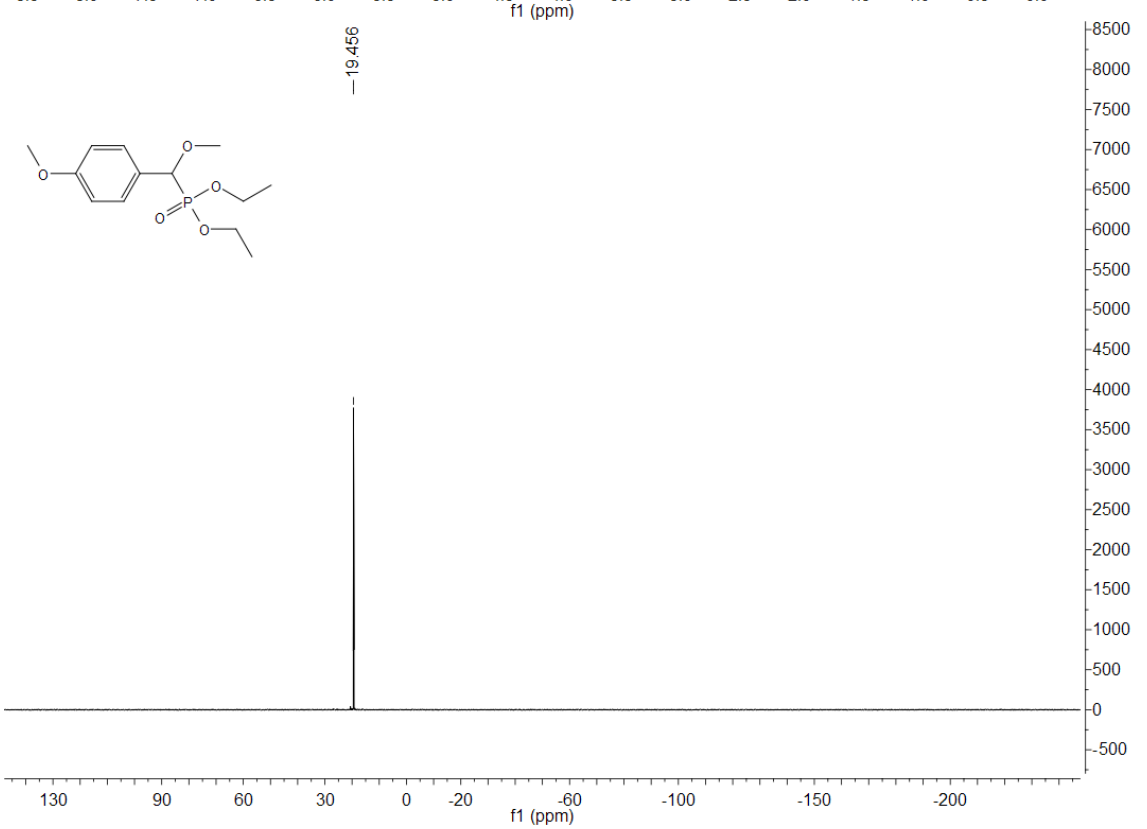

133.336
133.318
127.445
127.410
127.390
127.367
127.001
126.943
80.237
78.562
76.360
76.043
75.725
62.128
62.059
61.997
61.929
57.734
57.585
15.429
15.366
15.304
0.000
f1 (ppm)

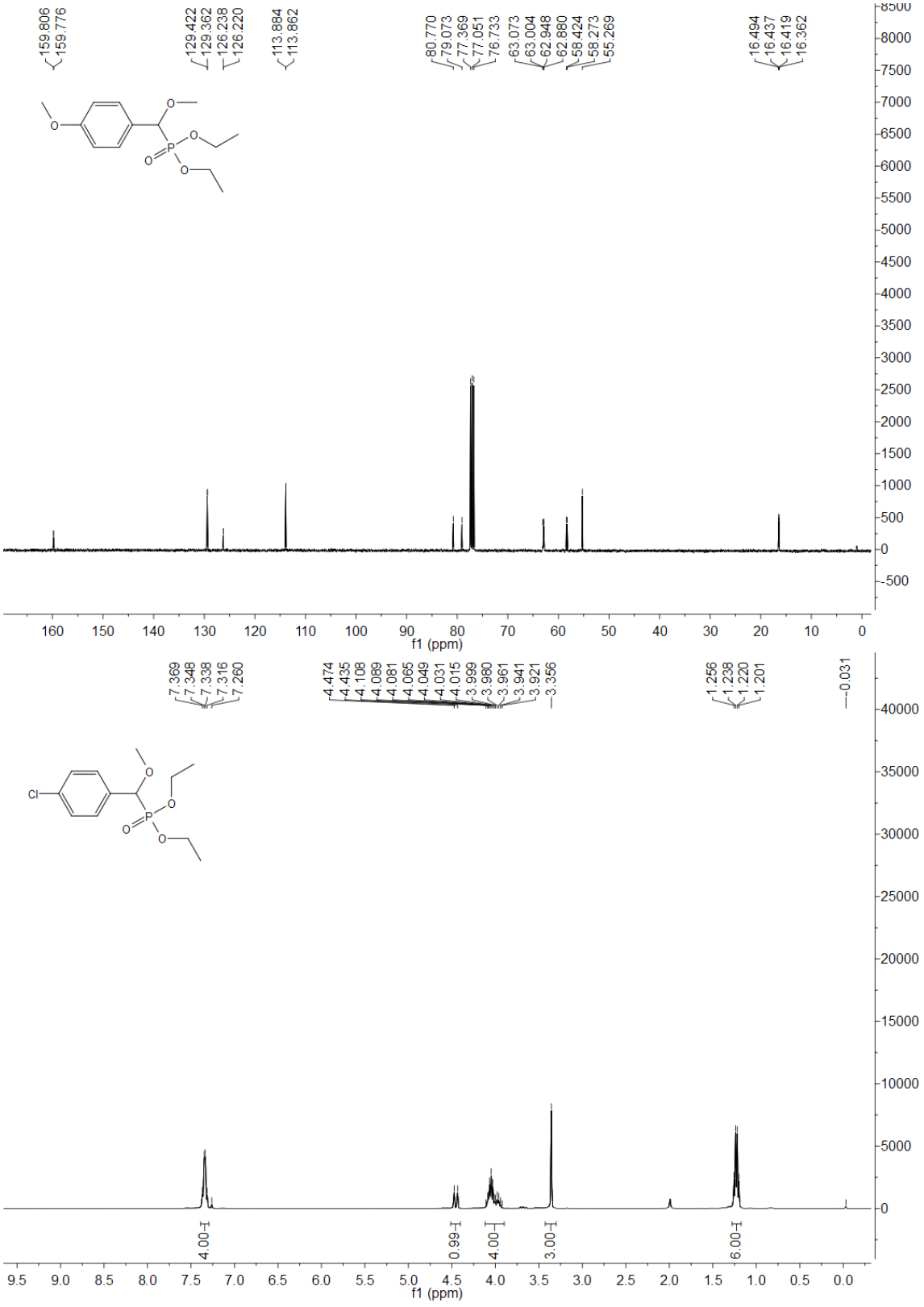

7.328
7.308
7.260
7.187
7.169
4.487
4.448
4.092
4.075
4.058
4.044
3.956
3.381
2.349
1.276
1.259
1.241
1.222
1.205
0.008
1.98
1.97
0.97
4.00
2.96
2.98
6.24
f1 (ppm)

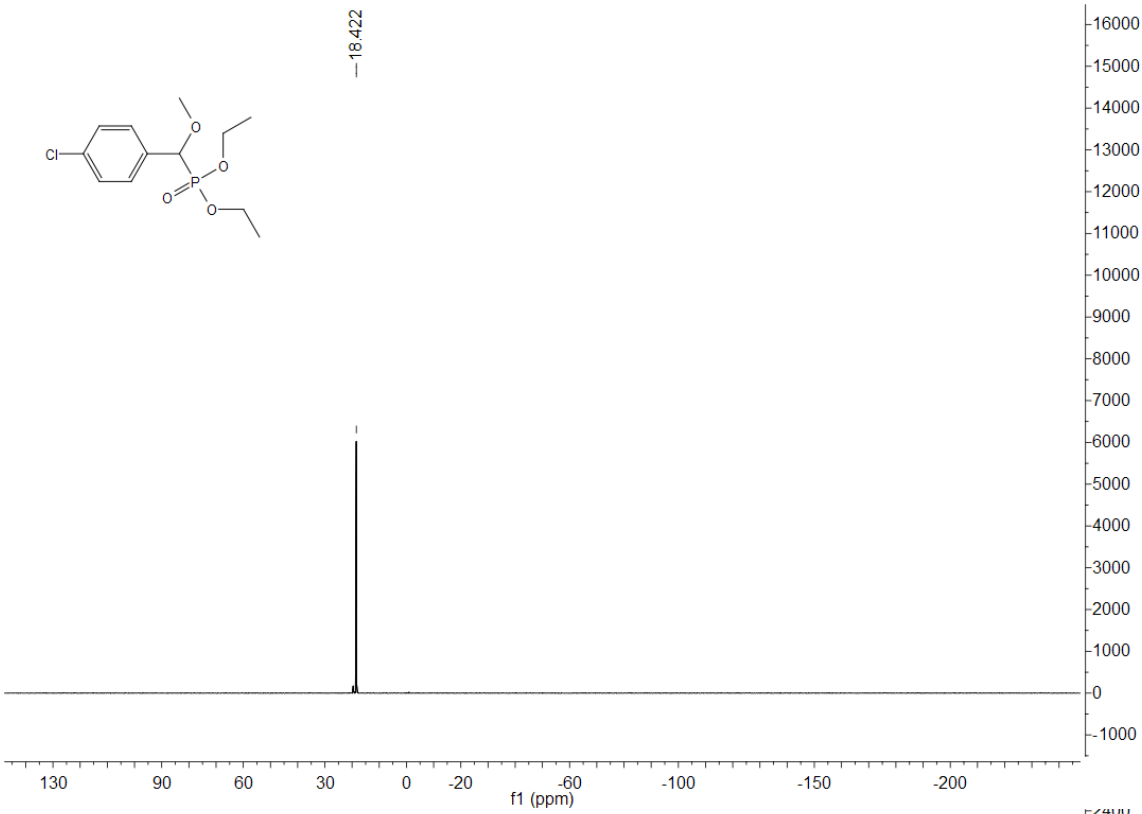

19.334
4500
4000
3500
3000
2500
2000
1500
1000
500
0
130
90
60
30
0
-20
-60
-100
-150
-200
f1 (ppm)

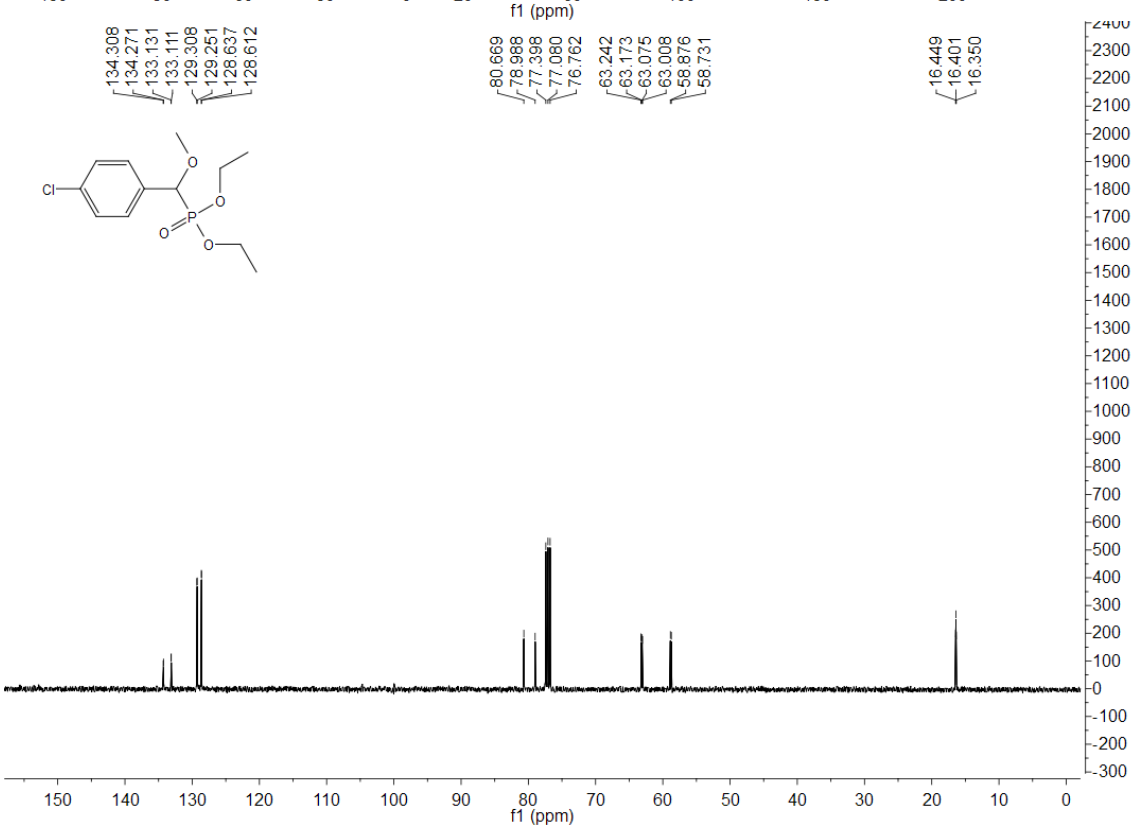

138.293
138.262
131.261
131.241
129.152
129.129
128.016
127.958
81.136
79.453
77.360
77.042
76.724
63.105
63.036
62.972
62.904
58.592
58.442
21.244
16.477
16.416
16.352
3400
3200
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

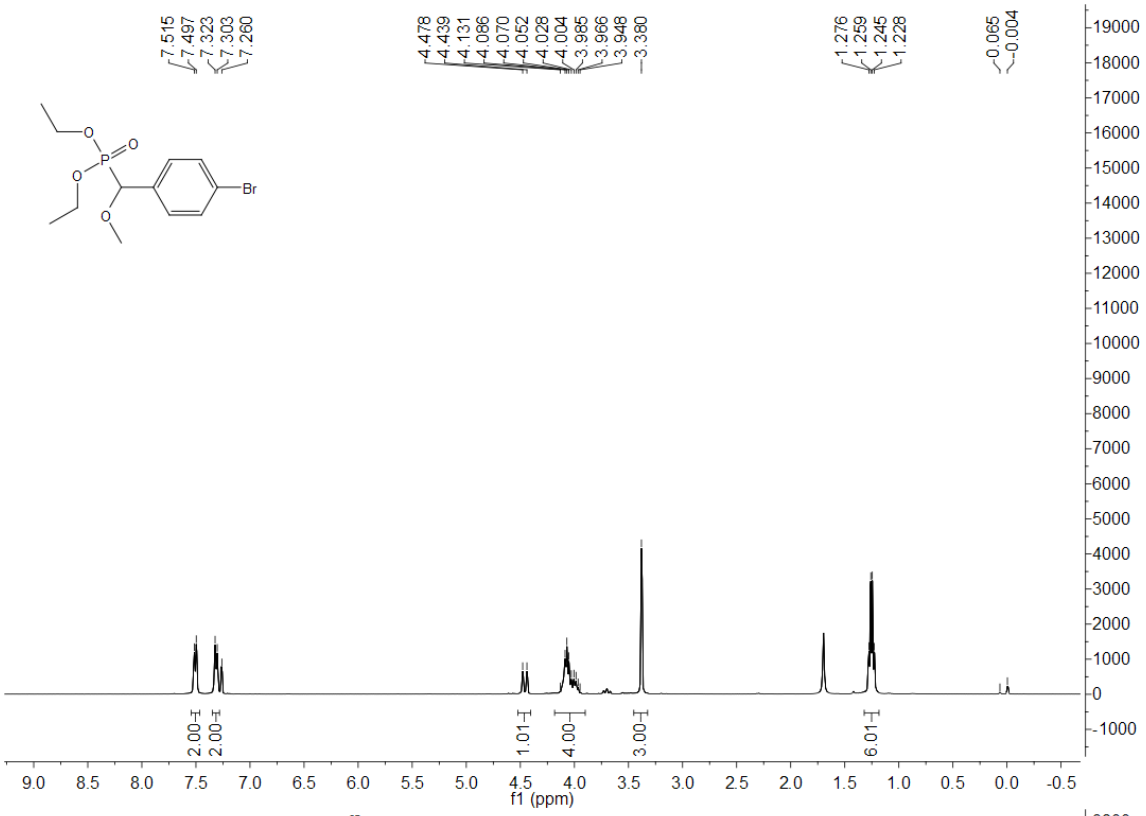

7.365
7.344
7.260
6.911
6.892
4.457
4.420
4.098
4.081
4.063
4.048
4.032
4.020
3.998
3.972
3.953
3.934
3.915
3.895
3.874
3.805
3.343
1.822
1.279
1.262
1.244
1.227
1.209
1.192
0.058
-0.014
2.00
2.01
1.00
4.02
3.00
2.99
6.01
f1 (ppm)

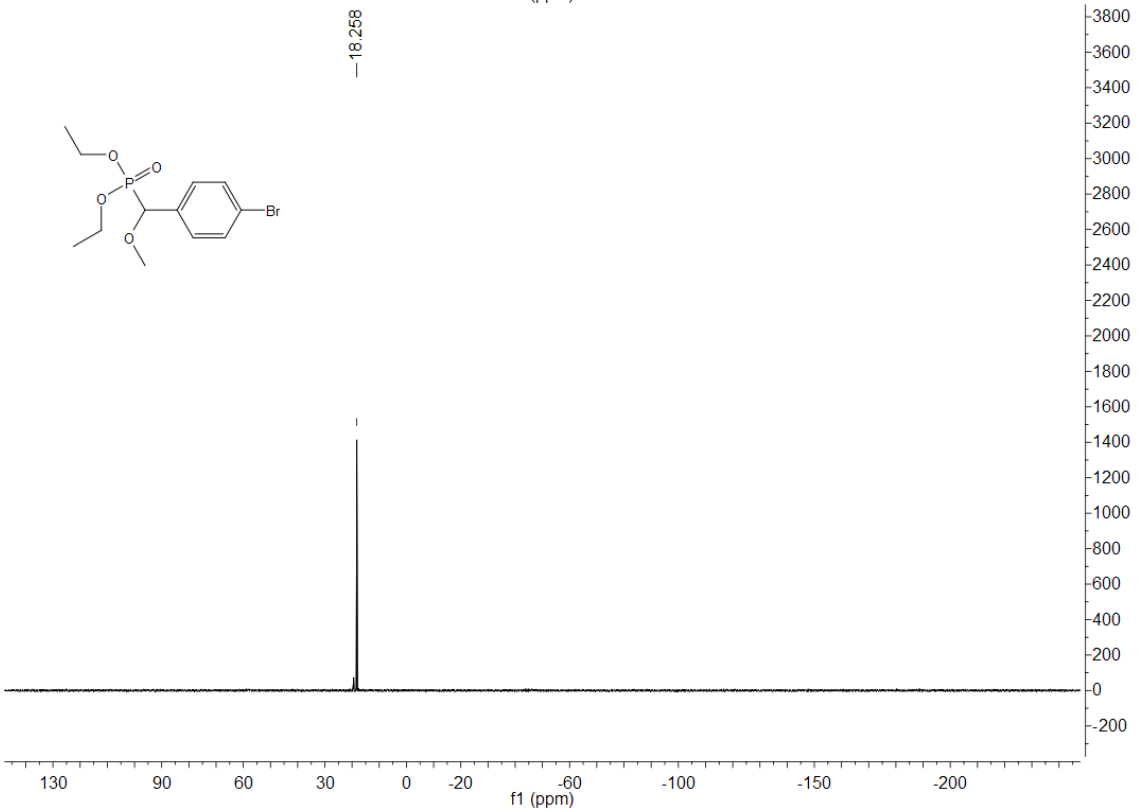

19.456
f1 (ppm)

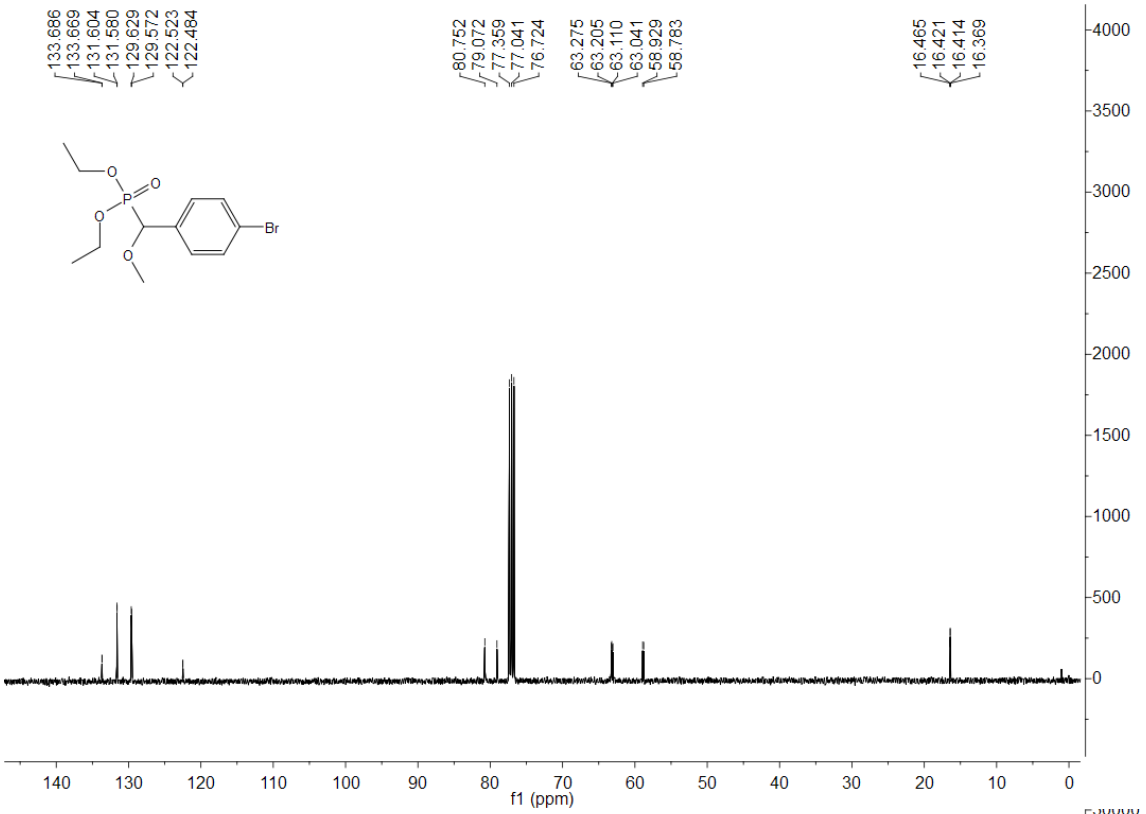

159.806
159.776
129.422
129.362
126.238
126.220
113.884
113.862
80.770
79.073
77.369
77.051
76.733
63.073
63.004
62.948
62.880
58.424
58.273
55.269
16.494
16.437
16.419
16.362
f1 (ppm)
7.369
7.348
7.338
7.316
7.260
4.474
4.435
4.108
4.089
4.081
4.065
4.049
4.031
4.015
3.999
3.980
3.961
3.941
3.921
3.356
1.256
1.238
1.220
1.201
0.031
4.00
0.99
4.00
3.00
6.00
f1 (ppm)

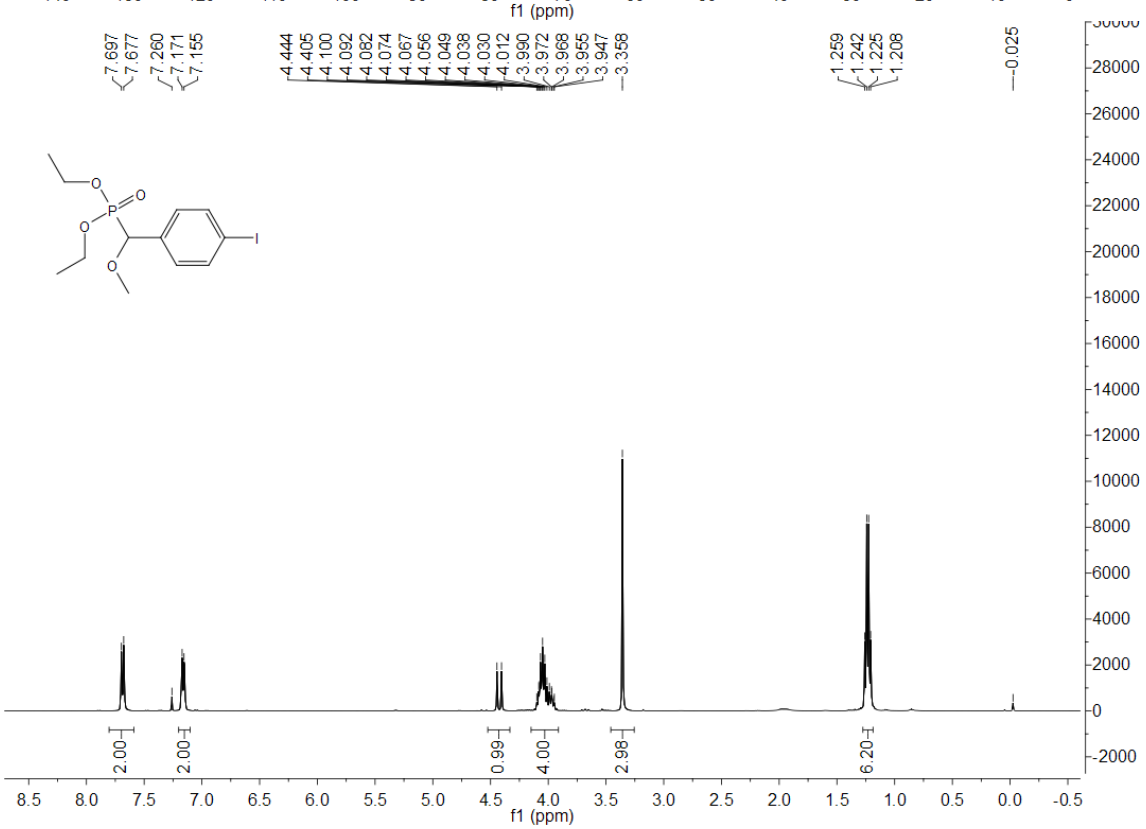

18.422
Cl
O
O
O
P
O
f1 (ppm)


134.308
134.271
133.131
133.111
129.308
129.251
128.637
128.612
80.669
78.988
77.398
77.080
76.762
63.242
63.173
63.075
63.008
58.876
58.731
16.449
16.401
16.350
Cl
O
O
O
P
O
f1 (ppm)


7.515
7.497
7.323
7.303
7.260
4.478
4.439
4.131
4.086
4.070
4.052
4.028
4.004
3.985
3.966
3.948
3.380
1.276
1.259
1.245
1.228
0.065
-0.004
Br
2.00
2.00
1.01
4.00
3.00
6.01
f1 (ppm)


18.258
Br
f1 (ppm)


133.686
133.669
131.604
131.580
129.629
129.572
122.523
122.484
80.752
79.072
77.359
77.041
76.724
63.275
63.205
63.110
63.041
58.929
58.783
16.465
16.421
16.414
16.369
Br
f1 (ppm)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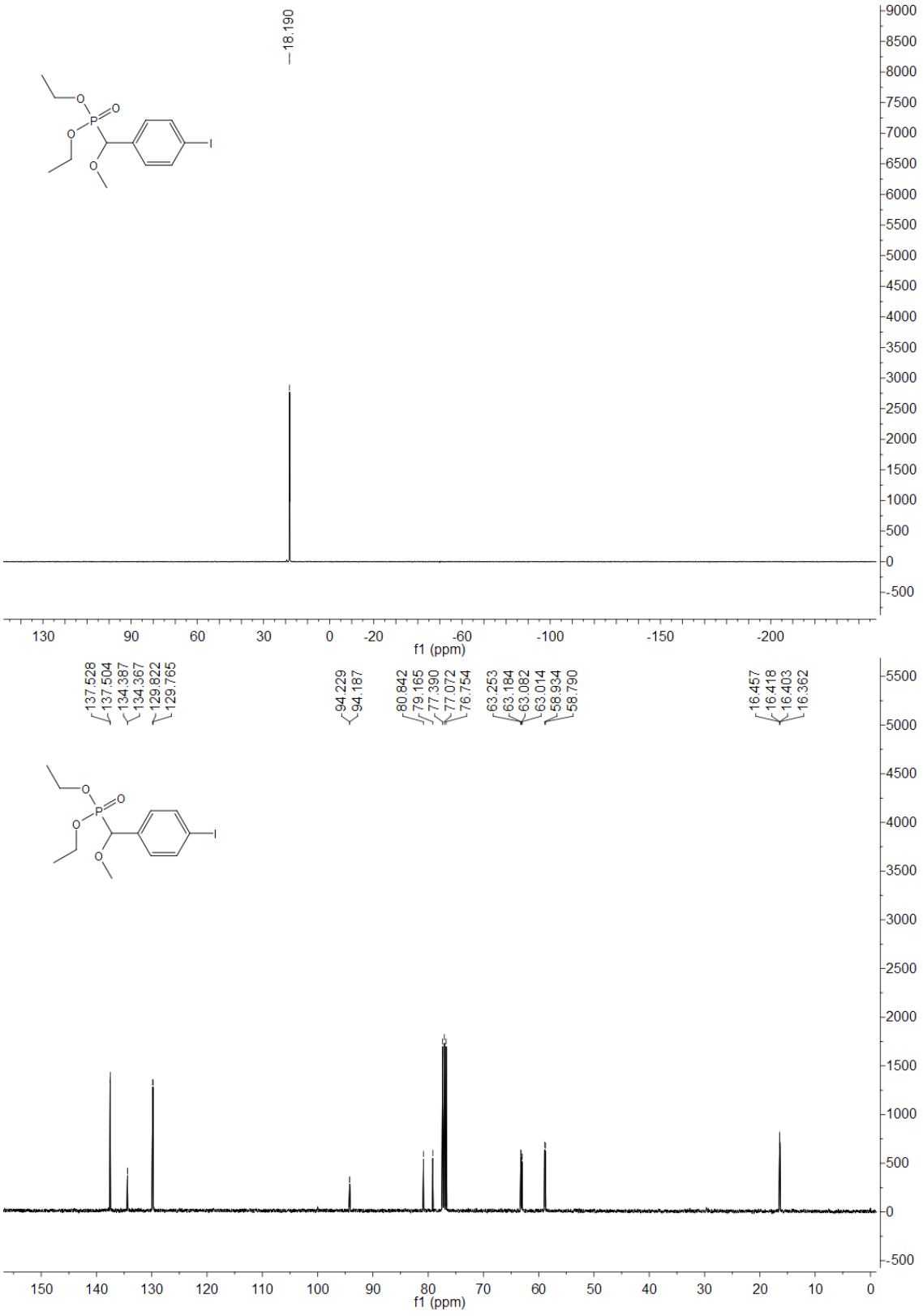
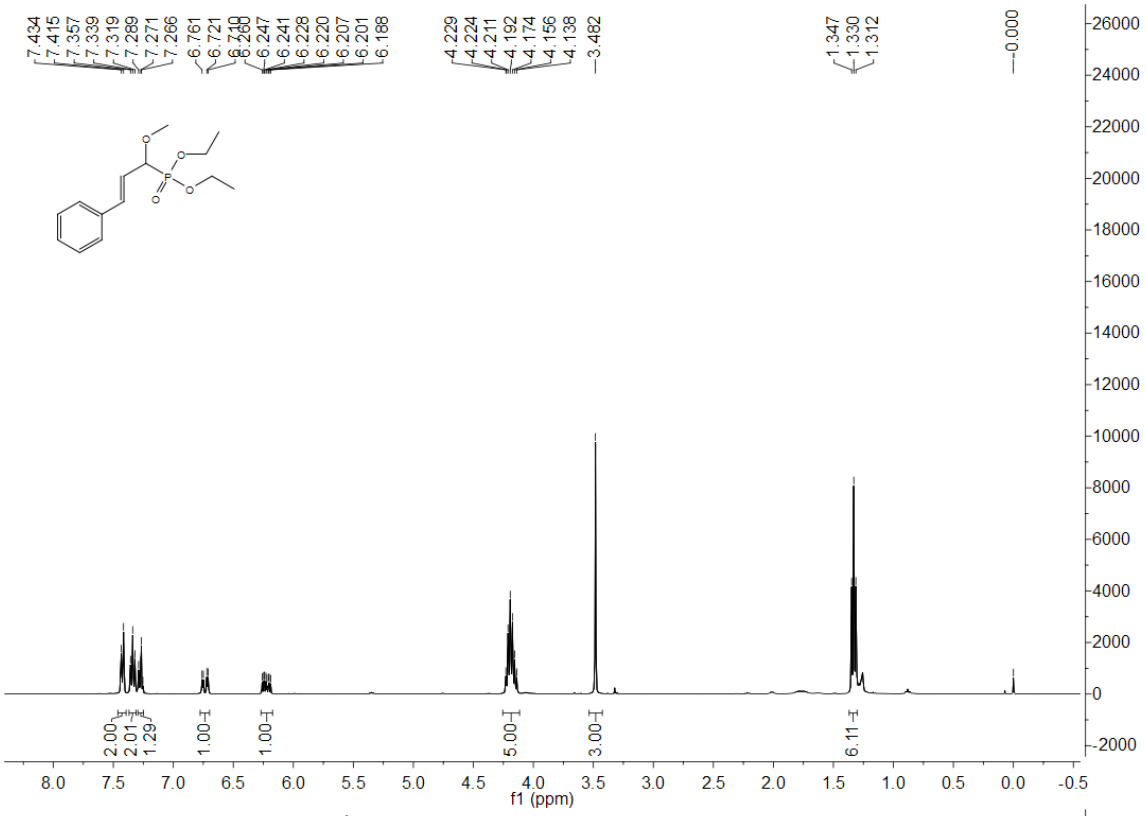
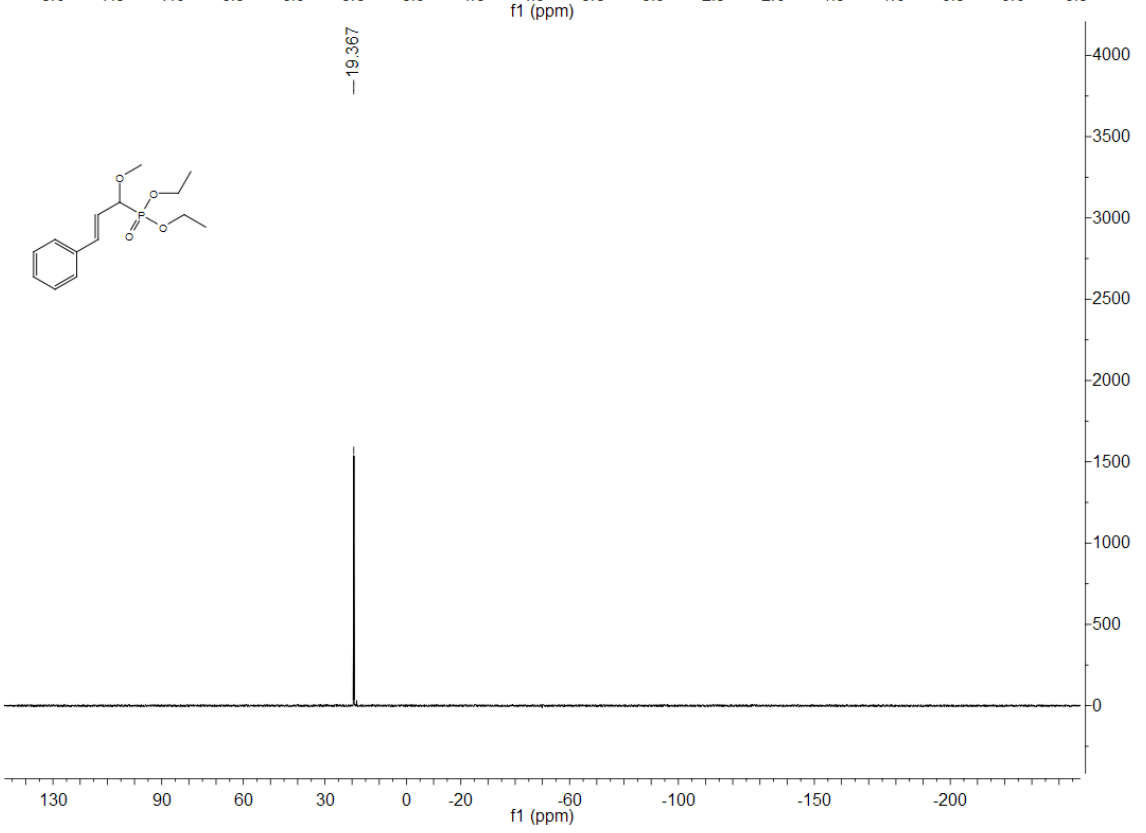
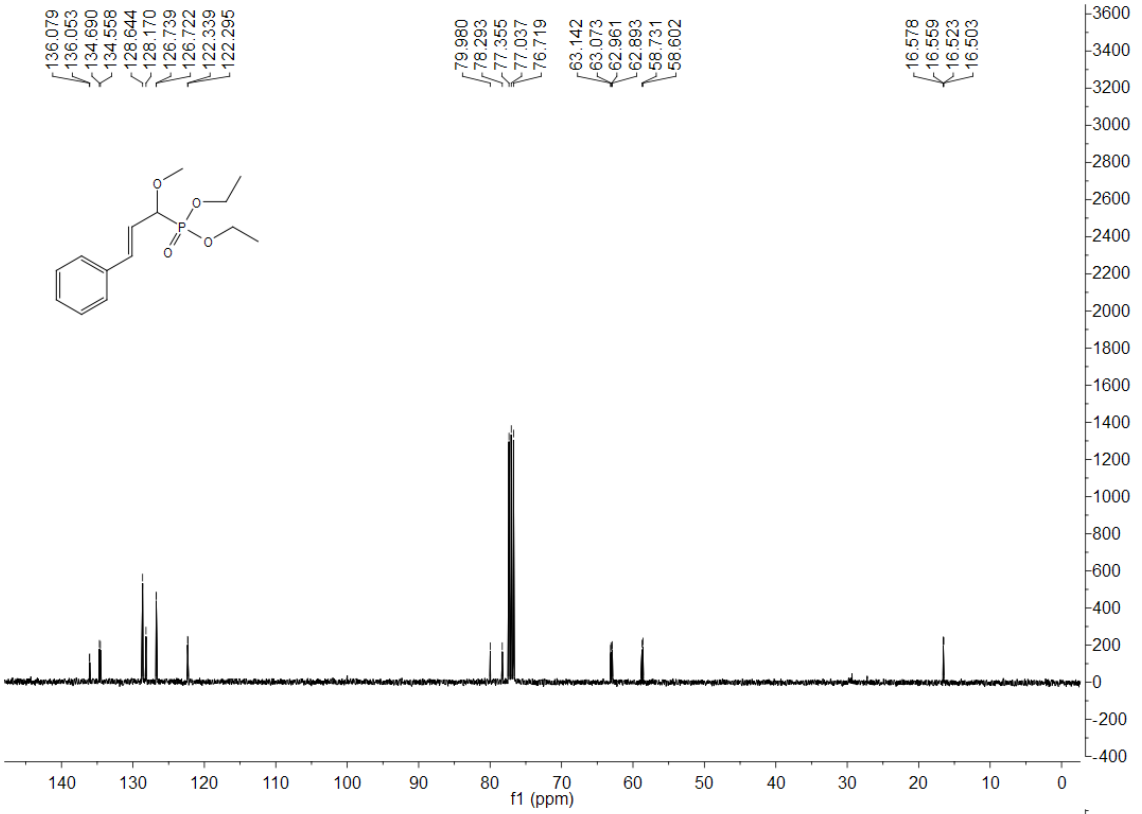
7.677
7.260
7.171
7.155
4.444
4.405
4.100
4.092
4.082
4.074
4.067
4.056
4.049
4.038
4.030
4.012
3.990
3.972
3.968
3.955
3.947
3.358
1.259
1.242
1.225
1.208
0.025
2.00
2.00
0.99
4.00
2.98
6.20
I
f1 (ppm)


18.190
f1 (ppm)
137.528
137.504
134.387
134.367
129.822
129.765
94.229
94.187
80.842
79.165
77.390
77.072
76.754
63.253
63.184
63.082
63.014
58.934
58.790
16.457
16.418
16.403
16.362
f1 (ppm)


7.434
7.415
7.357
7.339
7.319
7.289
7.271
7.266
6.761
6.721
6.247
6.241
6.228
6.220
6.207
6.201
6.188
4.229
4.224
4.211
4.192
4.174
4.156
4.138
3.482
1.347
1.330
1.312
0.000
2.00
2.01
1.29
1.00
1.00
5.00
3.00
6.11
f1 (ppm)


19.367
f1 (ppm)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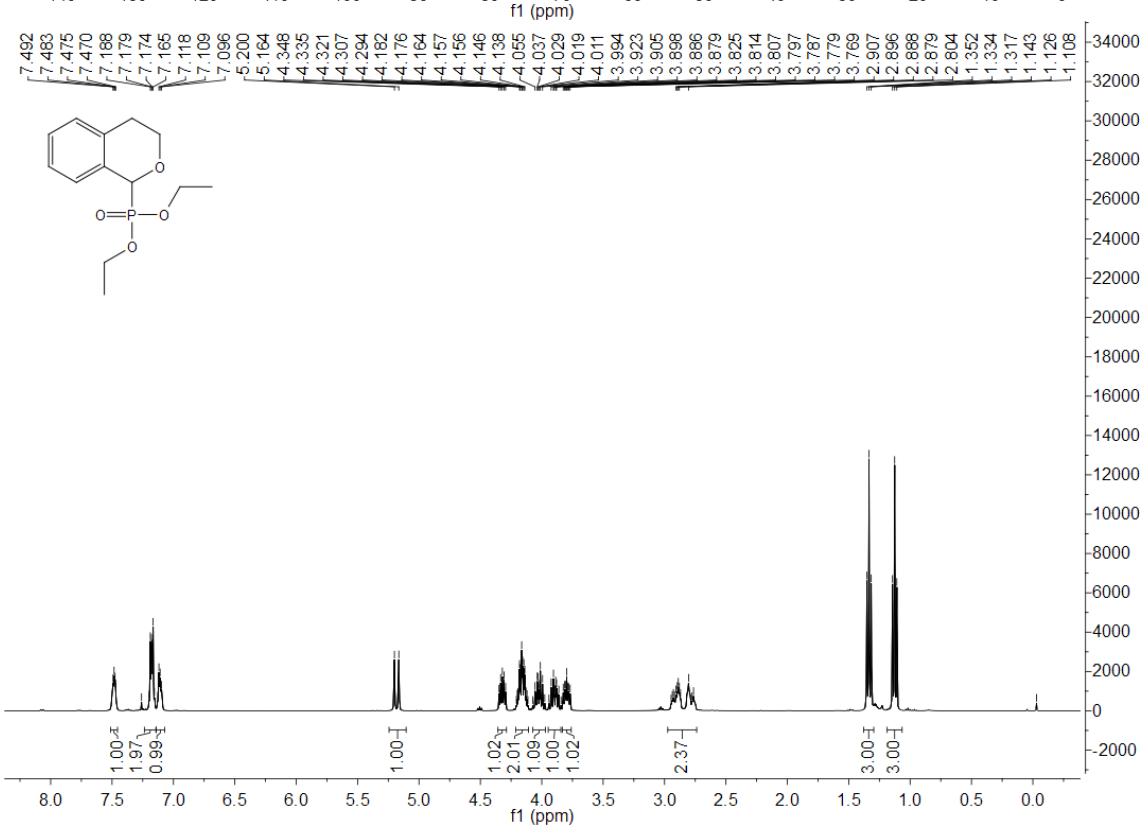
136.053
134.690
134.558
128.644
128.170
126.739
126.722
122.339
122.295
79.980
78.293
77.355
77.037
76.719
63.142
63.073
62.961
62.893
58.731
58.602
16.578
16.559
16.523
16.503
f1 (ppm)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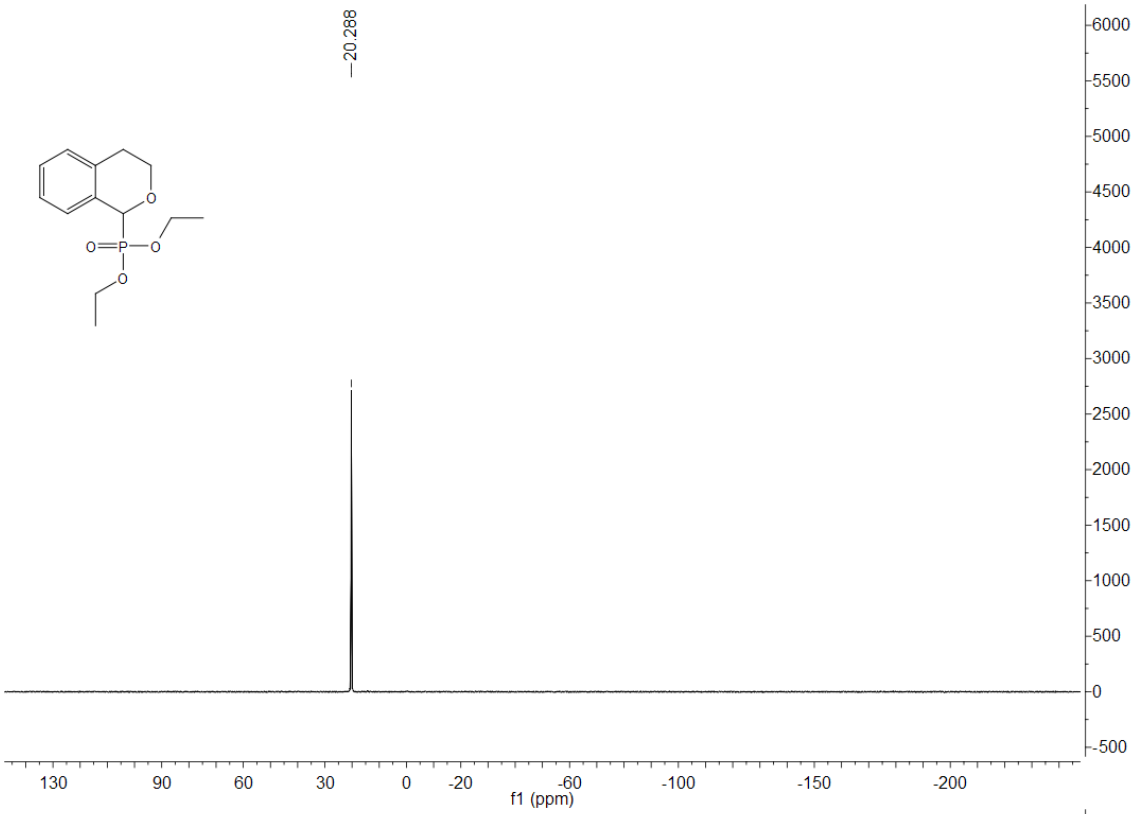
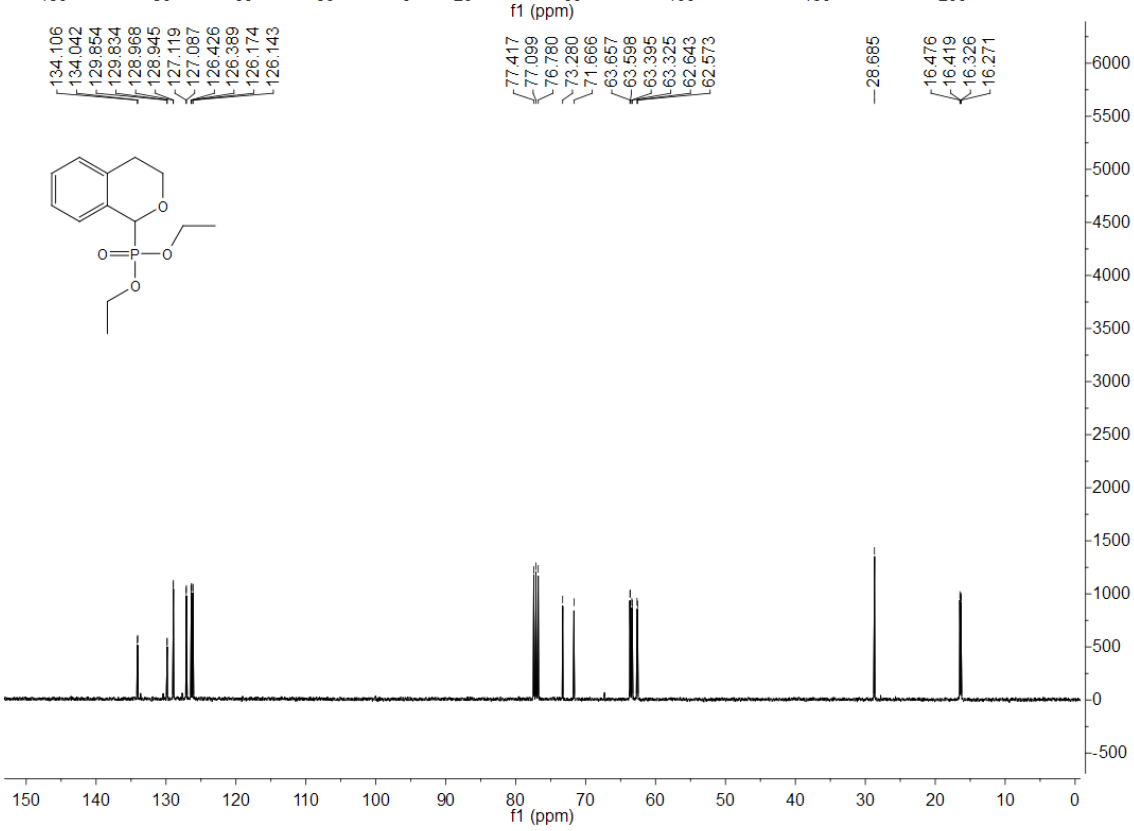
7.483
7.475
7.470
7.188
7.179
7.174
7.165
7.118
7.109
7.096
5.200
5.164
4.348
4.335
4.321
4.307
4.294
4.182
4.176
4.164
4.157
4.156
4.146
4.138
4.055
4.037
4.029
4.019
4.011
3.994
3.923
3.905
3.898
3.886
3.879
3.825
3.814
3.807
3.797
3.787
3.779
3.769
2.907
2.896
2.888
2.879
2.804
1.352
1.334
1.317
1.143
1.126
1.108
1.00
1.97
0.99
1.00
1.02
2.01
1.09
1.00
1.02
2.37
3.00
3.00
f1 (ppm)


20.288
f1 (ppm)


134.106
134.042
129.854
129.834
128.968
128.945
127.119
127.087
126.426
126.389
126.174
126.143
77.417
77.099
76.780
73.280
71.666
63.657
63.598
63.395
63.325
62.643
62.573
28.685
16.476
16.419
16.326
16.271
f1 (ppm)