Supporting Information

Phosphinoyl Radical-initiated Vicinal Cyanophosphinoylation of Alkenes

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1. Experimental Section

General Methods. ^{1}H NMR (400 or 300 MHz) and ^{13}C NMR (101 or 75 MHz) spectra were determined with $CDCl_3$ or DMSO-d_6 as solvent and tetramethylsilane (TMS) as internal standard or 85% H_3PO_4 as external standard for ^{31}P NMR (162 MHz). Chemical shifts were reported in ppm from internal TMS (δ), all coupling constants (J values) were reported in Hertz (Hz). High resolution mass spectra were recorded on a TOF machine (ESI). Column chromatography was performed with 300-400 mesh silica gel using flash column techniques. All of the reagents were used directly as obtained commercially unless otherwise noted. All alkenes were purified by flash column chromatography (Al_2O_3) before use.

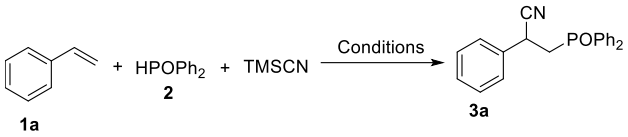
Abbreviations: Me: methyl; Bu: butyl; Ph: phenyl; HOTf: trifluormethanesulfonic acid; DCM: dichloromethane; DCE: 1,2-dichloroethane; EtOAc: ethyl acetate; THF: tetrahydrofurane; CH_3CN: acetonitrile; EtOH: ethanol; NMP: *N*-methyl pyrrolidone; HOAc: acetic acid; DMF: *N*,*N*-dimethylformamide; DMA: *N*,*N*-dimethylacetamide; DMSO: dimethylsulfoxide; TBHP: *tert*-butyl hydroperoxide; DTBP: di-*tert*-butyl peroxide.

2. Details for optimization of reaction conditions

We tested a series of reaction conditions, which could generate phosphoryl radicals (Table S1). For entries 1-3, to a Schlenk tube charged with a magnetic stirring bar, $Cu(OTf)_2$ (20 mol%) was added and exchanged with argon gas for three times. A solution of styrene (**1a**) (0.40 mmol), $HP(O)Ph_2$ (0.8 mmol) and TMSCN (0.80 mmol) in CH_3CN (3 mL) was injected and TBHP or DTBP (1.2 mmol) was injected followed by. The mixture was allowed to stir at 60 ^{o}C (monitored with TLC). For entries 4-8, to a Schlenk tube charged with a magnetic stirring bar, CuCN (20 mol%), other solid catalyst and oxidants were added and exchanged with argon gas for three times. A solution of **1a** (0.40 mmol), $HP(O)Ph_2$ (0.8 mmol) and TMSCN (0.80 mmol) in the solvent (3 mL) was then injected. The mixture was allowed to stir at different temperature (monitored with TLC). After the substrate was consumed, the solvent was removed under vacuum. The residue was purified by flash chromatography on silica gel (petroleum ether: ethyl acetate = 1:1) to give the product **3a**.

As shown in Table S1 below, only the reactions using Ag_2O or $Mn(OAc)_3$ as the oxidant could give the target product (Table S1, entries 7 and 10). Finally, we chose $Mn(OAc)_3$ as the oxidant because it is cost-effective and relatively environmental benign.

Table S1. Optimization of the reaction conditions[a]

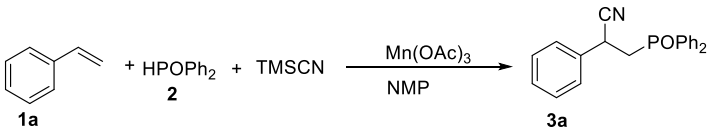


Entry	Oxidant	Solvent	Temp. (°C)	Yield[b]
1	TBHP 70% in H_2O (3 eq)/ $Cu(OTf)_2$ (0.2 eq)	CH_3CN	60	N. D.[c]
2	TBHP 5.5 M in decane(3 eq)/ $Cu(OTf)_2$ (0.2 eq)	CH_3CN	60	N. D.
3	DTBP(3 eq)/ $Cu(OTf)_2$ (0.2 eq)	CH_3CN	60	N. D.
4	$AgNO_3$ (0.05 eq)/ $K_2S_2O_8$ (2 eq) / CuCN (0.2 eq)	CH_3CN	100	Trace
5	$Mn(OAc)_2.4H_2O$ (0.1 eq)/ MnO_2 (3 eq)/ CuCN (0.2 eq)	CH_3CN	70	N. R.[d]
6[e]	Ag_2CO_3 (10 mol%)/$Mg(NO_3)_2$ (1.5 eq) / CuCN (0.2 eq)	CH_3CN	80	N. D.
7[f]	Ag_2O (3 eq) / CuCN (0.2 eq)	NMP	120	39%
8	$AgNO_3$ (0.1 eq)/ CuCN (0.2 eq)	CH_3CN	100	N. D.
9	AgOAc (3 eq) / CuCN (0.2 eq)	DCE	90	N. D.
10	**$Mn(OAc)_3.2H_2O$ (2.5 eq) / CuCN (0.2 eq)**	**NMP**	**55**	**27%**

[a] Reaction conditions: styrene (**1a**, 0.4 mmol), $HPOPh_2$ (1.5 eq), TMSCN (2 eq) under argon atmosphere for 3 h. [b] Isolated yields. [c] N. D. means none detected. [d] N. R. means no reaction. [e] 4Å MS (0.2 g) was added. [f] 1,10-Phen (10 mol%) was added.

The screening results of a range of copper salts are summarized in Table S2 below, which show that using CuCN as catalyst is a better choice.

Table S2. Copper catalyst screening results[a]

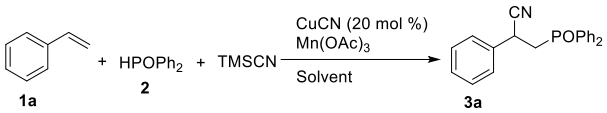


Entry	Cat. (20 mol%)	Solvent	Yield[b]
1	**CuCN**	**NMP**	**47%**
2	CuI	NMP	19%
3	CuBr	NMP	39%
4	Cu_2O	NMP	42%
5	CuCl	NMP	40%
6[c]	$CuCl_2.2H_2O$	NMP	20%
7	$Cu(OAc)_2.H_2O$	NMP	10%
8	$Cu(OTf)_2$	NMP	35%
9	$CuBr_2$	NMP	Trace

[a] Reaction conditions: styrene (**1a**, 0.4 mmol), $HPOPh_2$ (1.5 eq), TMSCN (2 eq), Mn $(OAc)_3.2H_2O$ (2.5 eq), NMP (3 mL) under argon atmosphere at 20 °C for 3 h. [b] Isolated yields.

The screening results of solvent are summarized in Table S3 below, which show that formamide such as DMF, $HCON(Et)_2$ is better than other solvents, finally, DMF was chosen as solvent for the reaction.

Table S3. Solvent screening results[a]



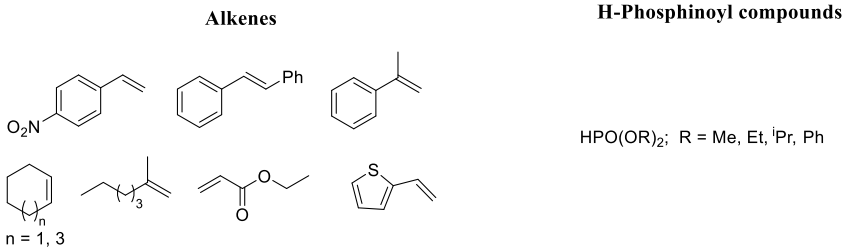
Entry	Solvent	Yield[b]	Entry	Solvent	Yield[b]
1	CH_3CN	Trace	10	HOAc	Trace
2	CH_3NO_2	Trace	11	THF	Trace
3	CH_2Cl_2	Trace	12	EA	N. D.[c]
4	DCE	35%	13	DMSO	Trace
5	Toluene	Trace	**14**	**DMF**	**70%**
6[c]	Chlorobenzene	Trace	15	DMA	Trace
7	Dioxane	21%	16	$HCON(n\text{-}Bu)_2$	Trace
8	EtOH	41%	17	$HCONEt_2$	51%
9	Acetone	Trace			

[a] Reaction conditions: styrene (**1a**, 0.4 mmol), $HPOPh_2$ (1.5 eq), TMSCN (2 eq), CuCN (20 mol%), Mn $(OAc)_3.2H_2O$ (2.5 eq), solvent (3 mL) under argon atmosphere at 20 ºC for 3 h. [b] Isolated yields. [c] N. D. means none detected.

3. The reactions of other alkenes with dialkylphosphites

The reactions of alkenes below with dialkylphosphites were performed, no desired product was detected under standard reaction conditions (Scheme S1).

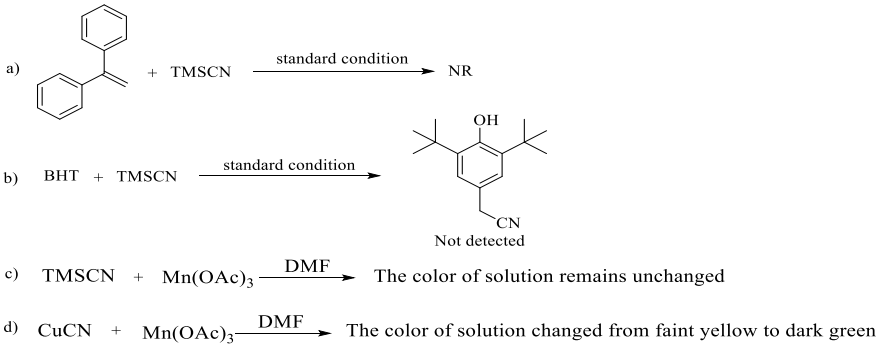
Scheme S1



4. Mechanistic exploration

The control experiments for observing cyano radical formation were done (Scheme S2)

Scheme S2. Control experiments for observing cyano radical formation



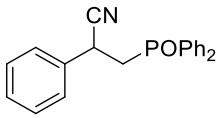
5. Typical procedure for the preparation of products 3a and 3c

Preparation of 3a (at 0.40 mmol scale)**.** To a Schlenk tube charged with a magnetic stirring bar, CuCN (0.08 mmol) and $Mn(OAc)_3 \cdot 2H_2O$ (1.2 mmol) were added and exchanged with argon gas for three times. A solution of styrene (**1a**, 0.40 mmol), $HP(O)Ph_2$ (0.8 mmol) and TMSCN (0.80 mmol) in DMF (3 mL) was then injected. The mixture was allowed to stir at 20 °C for 3 h (monitored with TLC). After the substrate was consumed, water (50 mL) was added to the reaction mixture, then extracted with dichloromethane (30 mL×3). The combined organic layer was washed with saturated sodium bicarbonate solution (50 mL×3) and dried with anhydrous Na_2SO_4, filtered, and concentrated in vacuo. The residue was purified by flash chromatography on silica gel with 1:1 petroleum ether:ethyl acetate to afford the desired product **3a**.

Preparation of 3c (at 1.0 mmol scale). To a Schlenk tube charged with a magnetic stirring bar, CuCN (0.20 mmol) and $Mn(OAc)_3 \cdot 2H_2O$ (3.0 mmol) were added and exchanged with argon gas for three times. A solution of 4-methoxystyrene (**1c**, 1.0 mmol), $HP(O)Ph_2$ (2.0 mmol) and TMSCN (2.0 mmol) in DMF (5 mL) was then injected. The mixture was allowed to stir at 20 °C for 3 h (monitored with TLC). After the substrate was consumed, water (80 mL) was added to the reaction mixture, then extracted with dichloromethane (35 mL×3). The combined organic layer was washed with saturated sodium bicarbonate solution (50 mL×3) and dried with anhydrous Na_2SO_4, filtered, and concentrated in vacuo. The residue was purified by flash chromatography on silica gel with 1:1 petroleum ether:ethyl acetate to afford the desired product **3c** (242 mg, 67% yield).

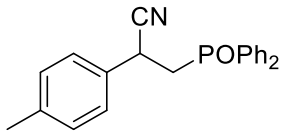
6. Structure characterization of compounds 3 and 5-8 (reactions at 0.40 mmol scale)

3-(Diphenylphosphoryl)-2-phenylpropanenitrile **(3a)**



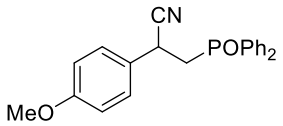
White solid; 114 mg, 86% yield. Mp: 103−105 °C. ^{1}H NMR (400 MHz, DMSO-d_6): δ 7.92 – 7.81 (m, 2H), 7.77 – 7.69 (m, 2H), 7.58 – 7.48 (m, 4H), 7.46 – 7.37 (m, 4H), 7.33 – 7.23 (m, 3H), 4.39 (td, J = 9.4, 5.8 Hz, 1H), 3.12 – 2.94 (m, 1H), 2.95 – 2.56 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 134.90 (d, J = 6.9 Hz), 132.00 (d, J = 2.5 Hz), 131.51 (d, J = 2.5 Hz), 130.47 (d, J = 9.6 Hz), 130.04 (d, J = 9.5 Hz), 128.69, 128.49, 128.37, 128.25, 128.13, 128.00, 127.04, 119.35 (d, J = 8.7 Hz), 35.80 (d, J = 68.0 Hz), 30.05 (d, J = 2.2 Hz). ^{31}P NMR (162 MHz, DMSO-d_6): δ 26.17. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{21}H_{19}NOP$ 332.1204, found 332.1196.

3-(Diphenylphosphoryl)-2-(p-tolyl)propanenitrile **(3b)**



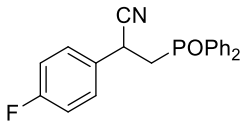
White solid; 124 mg, 90% yield. Mp: 142−144 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 – 7.73 (m, 2H), 7.62 – 7.48 (m, 5H), 7.45 (t, J = 7.3 Hz, 1H), 7.35 (t, J = 6.6 Hz, 2H), 7.18 (d, J = 7.8 Hz, 2H), 7.02 (d, J = 7.6 Hz, 2H), 4.50 – 4.35 (d, J = 6.6 Hz, 1H), 3.06 – 2.95 (m, 1H), 2.83 – 2.70 (t, J = 14.9 Hz, 1H), 2.26 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 137.84, 131.82, 131.62 (d, J = 64.2 Hz), 130.45 (d, J = 9.2 Hz), 130.07 (d, J = 9.1 Hz), 129.27, 128.46, 128.34, 128.18, 128.06, 126.92, 119.53 (d, J = 8.5 Hz), 35.88 (d, J = 68.1 Hz,), 29.70, 20.52. ^{31}P NMR (162 MHz, $CDCl_3$): δ 27.53. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{22}H_{21}NOP$ 346.1361, found 346.1355.

3-(Diphenylphosphoryl)-2-(4-methoxyphenyl)propanenitrile **(3c)**



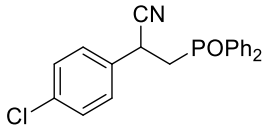
White solid; 101 mg, 69% yield. Mp: 130−132 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 – 7.72 (m, 2H), 7.63 – 7.49 (m, 5H), 7.46 – 7.41 (m, 1H), 7.39 – 7.30 (m, 2H), 7.25 – 7.09 (m, 2H), 6.79 – 6.65 (m, 2H), 4.65 – 4.22 (m, 1H), 3.73 (s, 3H), 3.14 – 2.95 (m, 1H), 2.86 – 2.73 (m, 1H). ^{13}C NMR (101 MHz, DMSO): δ 158.91, 133.36, 132.66, 131.62 (d, J = 31.0 Hz), 130.54 (d, J = 9.3 Hz), 130.30 (d, J = 9.3 Hz), 128.95, 128.62, 128.50, 128.39, 127.67 (d, J = 8.1 Hz), 120.58 (d, J = 8.1 Hz), 114.10, 55.13, 33.33 (d, J = 67.8 Hz), 29.08. ^{31}P NMR (162 MHz, $CDCl_3$): δ 27.27. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{22}H_{21}NO_2P$ 362.1310, found 362.1301.

3-(Diphenylphosphoryl)-2-(4-fluorophenyl)propanenitrile **(3d)**



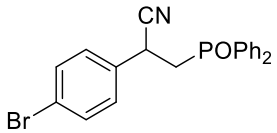
White solid; 121 mg, 87% yield. Mp: 170−172 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (dd, J = 11.3, 7.7 Hz, 2H), 7.68 – 7.46 (m, 6H), 7.45 – 7.26 (m, 4H), 6.93 (t, J = 8.5 Hz, 2H), 4.76 – 4.28 (m, 1H), 3.29 – 2.97 (m, 1H), 2.89 – 2.46 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 161.99 (d, J = 248.3 Hz), 132.03, 131.50, 130.38, 130.29, 130.07, 129.98, 129.11, 129.02, 128.47 (d, J = 11.5 Hz), 128.18 (d, J = 11.5 Hz), 119.37 (d, J = 10.2 Hz), 115.55 (d, J = 22.0 Hz), 35.72 (d, J = 67.9 Hz), 29.51. ^{31}P NMR (162 MHz, $CDCl_3$): δ 27.33. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{21}H_{18}FNOP$ 350.1110, found 350.1106.

***2-(4-Chlorophenyl)-3-(diphenylphosphoryl)propanenitrile* (3e)**



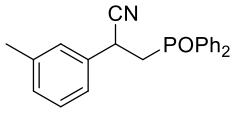
White solid; 130 mg, 89% yield. Mp: 173−174 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88 – 7.75 (m, 2H), 7.63 – 7.49 (m, 6H), 7.45 – 7.37 (m, 2H), 7.33 – 7.25 (m, 2H), 7.23 – 7.15 (m, 2H), 4.69 – 4.34 (m, 1H), 3.23 – 3.03 (m, 1H), 2.96 – 2.76 (m, 1H). ^{13}C NMR (101 MHz, DMSO): δ 134.64 (d, J = 8.0 Hz), 133.55 (d, J = 12.7 Hz), 132.83, 132.51, 131.83, 131.46, 130.50 (d, J = 9.6 Hz), 130.30 (d, J = 9.5 Hz), 129.79, 128.63 (d, J = 2.9 Hz), 128.52 (d, J = 1.9 Hz), 128.39, 120.13 (d, J = 9.1 Hz), 32.80 (d, J = 68.4 Hz), 29.38. ^{31}P NMR (162 MHz, DMSO): δ 26.05. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{21}H_{18}ClNOP$ 366.0815, found 366.0817.

***2-(4-Bromophenyl)-3-(diphenylphosphoryl)propanenitrile* (3f)**



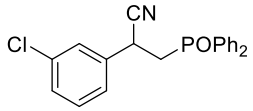
White solid; 136 mg, 83% yield. Mp: 167−169 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 – 7.71 (m, 2H), 7.57 – 7.44 (m, 6H), 7.38 – 7.27 (m, 4H), 7.21 – 7.11 (d, J = 7.0 Hz, 2H), 4.53 – 4.33 (m, 1H), 3.10 – 2.96 (m, 1H), 2.84 – 2.72 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 133.49, 132.08, 131.71, 131.47, 130.31 (d, J = 29.8 Hz), 129.01, 128.41 (d, J = 26.9 Hz), 122.23, 119.07, 34.90 (d, J = 109.2 Hz), 29.80. ^{31}P NMR (162 MHz, $CDCl_3$): δ 27.20. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{21}H_{18}BrNOP$ 410.0309, found 410.0280.

***3-(Diphenylphosphoryl)-2-(m-tolyl)propanenitrile* (3g)**



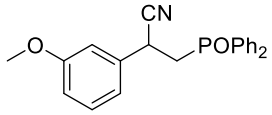
White solid; 123 mg, 84% yield. Mp: 112−114 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 – 7.73 (m, 2H), 7.63 – 7.48 (m, 5H), 7.47 – 7.40 (m, 1H), 7.40 – 7.30 (m, 2H), 7.18 – 7.06 (m, 3H), 7.05 – 6.94 (m, 1H), 4.47 – 4.36 (m, 1H), 3.07 – 2.95 (m, 1H), 2.82 – 2.71 (m, 1H), 2.23 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 138.5, 134.70 (d, J = 6.9 Hz), 131.97 (d, J = 2.8 Hz), 131.70 (d, J = 19.7 Hz), 131.46 (d, J = 2.8 Hz), 130.70 (d, J = 19.2 Hz), 130.42 (d, J = 9.7 Hz), 130.00 (d, J = 9.6 Hz), 128.65 (d, J = 11.4 Hz), 128.41 (d, J = 12.0 Hz), 128.15, 128.03, 127.78, 124.00, 119.49 (d, J = 9.1 Hz), 35.74 (d, J = 68.2 Hz), 29.96 (d, J = 2.4 Hz), 20.74. ^{31}P NMR (162 MHz, $CDCl_3$): δ 27.18. HRMS (ESI-TOF) *m/z*: $(M+Na)^+$ Calcd for $C_{22}H_{20}NOPNa$ 368.1180, found 368.1176.

2-(3-Chlorophenyl)-3-(diphenylphosphoryl)propanenitrile **(3h)**



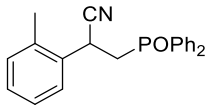
White solid; 127 mg, 87% yield. Mp: 147−148 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 – 7.73 (m, 2H), 7.60 – 7.48 (m, 5H), 7.47 – 7.42 (m, 1H), 7.40 – 7.26 (m, 3H), 7.23 – 7.11 (m, 3H), 4.60 – 4.32 (m, 1H), 3.10 – 2.95 (m, 1H), 2.83 – 2.73 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 136.38, 134.47, 132.10, 131.65, 130.27 (d, J = 34.1 Hz), 129.95, 128.42 (d, J = 22.0 Hz), 128.23, 127.49, 125.50, 118.92, 35.69 (d, J = 69.4 Hz), 29.88. ^{31}P NMR (162 MHz, $CDCl_3$): δ 27.07. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{21}H_{18}ClNOP$ 366.0815, found 366.0802.

3-(Diphenylphosphoryl)-2-(3-methoxyphenyl)propanenitrile **(3i)**



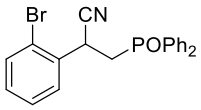
Yellow solid; 127 mg, 88% yield. Mp: 108−109 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 – 7.73 (m, 2H), 7.65 – 7.42 (m, 6H), 7.36 (td, J = 7.5, 2.9 Hz, 2H), 7.15 (t, J = 8.0 Hz, 1H), 6.90 (d, J = 7.7 Hz, 1H), 6.81 (d, J = 1.9 Hz, 1H), 6.72 (dd, J = 8.3, 2.3 Hz, 1H), 4.41 (dt, J = 9.8, 7.3 Hz, 1H), 3.73 (s, 3H), 3.09 – 2.92 (m, 1H), 2.85 – 2.72 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 159.5, 136.2 (d, J = 7.0 Hz), 132.0 (d, J = 2.6 Hz), 131.5 (d, J = 2.6 Hz), 130.4 (d, J = 9.6 Hz), 130.0 (d, J = 9.5 Hz), 129.8, 128.5, 128.4, 128.2, 128.1, 119.3 (d, J = 9.0 Hz), 119.2, 113.7, 112.62, 54.8, 35.7 (d, J = 68.0 Hz), 30.1 (d, J = 2.3 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 27.41. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{22}H_{21}NO_2P$ 362.1310, found 362.1301.

3-(Diphenylphosphoryl)-2-(o-tolyl)propanenitrile **(3j)**



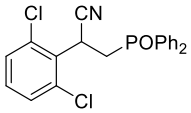
White solid; 104 mg, 75% yield. Mp: 137−140 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88 – 7.76 (m, 2H), 7.65 – 7.42 (m, 6H), 7.37 (d, J = 6.8 Hz, 3H), 7.19 – 7.09 (m, 2H), 7.06 (d, J = 3.3 Hz, 1H), 4.69 – 4.54 (dd, J = 14.5, 8.5 Hz, 1H), 3.24 – 2.92 (m, 1H), 2.85 – 2.56 (m, 1H), 2.34 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 134.87, 133.33 (d, J = 6.2 Hz), 132.03, 131.55, 130.70, 130.56, 130.47, 130.05, 129.96, 128.43 (d, J = 11.6 Hz), 128.24, 128.10, 127.18, 126.40, 119.46 (d, J = 7.0 Hz), 34.77 (d, J = 69.4 Hz), 26.64, 18.80. ^{31}P NMR (162 MHz, $CDCl_3$): δ 27.30. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{22}H_{21}NOP$ 346.1361, found 346.1352.

2-(2-Bromophenyl)-3-(diphenylphosphoryl)propanenitrile **(3k)**



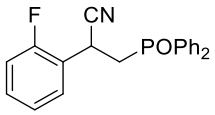
Light yellow solid; 106 mg, 65% yield. Mp: 134−136 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.80 (dd, J = 11.4, 7.5 Hz, 2H), 7.70 (dd, J = 11.5, 7.5 Hz, 2H), 7.57 – 7.40 (m, 8H), 7.30 – 7.23 (m, 1H), 7.11 (t, J = 7.6 Hz, 1H), 5.00 – 4.47 (m, 1H), 3.09 – 2.73 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 134.02 (d, J = 8.6 Hz), 133.16, 131.96 (d, J = 2.3 Hz), 131.69 (d, J = 2.3 Hz), 130.49 (d, J = 9.5 Hz), 130.27 (d, J = 9.5 Hz), 129.73, 129.49, 128.44, 128.32, 128.19, 127.84, 122.20, 118.19 (d, J = 6.1 Hz), 33.56 (d, J = 67.2 Hz), 30.72. ^{31}P NMR (162 MHz, $CDCl_3$): δ 26.86. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{21}H_{18}BrNOP$ 410.0309, found 410.0306.

2-(2,6-Dichlorophenyl)-3-(diphenylphosphoryl)propanenitrile **(3l)**



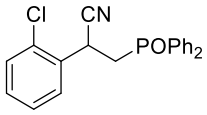
White solid; 133 mg, 79% yield. Mp: 115−1116 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.88 – 7.77 (m, 2H), 7.68 – 7.49 (m, 5H), 7.47 – 7.39 (m, 1H), 7.39 – 7.30 (m, 2H), 7.23 – 7.16 (m, 2H), 7.14 – 7.02 (m, 1H), 5.38 (dt, J = 10.0, 7.1 Hz, 1H), 3.32 – 3.17 (m, 1H), 3.13 – 2.98 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 134.82, 132.02 (d, J = 2.5 Hz), 131.58 (d, J = 2.5 Hz), 130.35 (d, J = 9.5 Hz), 130.10 (d, J = 9.5 Hz), 129.92, 128.48 (d, J = 12.0 Hz), 128.09 (d, J = 12.0 Hz), 116.95 (d, J = 10.3 Hz), 30.94 (d, J = 68.2 Hz), 25.91. ^{31}P NMR (162 MHz, $CDCl_3$): δ 27.17. HRMS (ESI-TOF) *m/z*: $(M+Na)^+$ Calcd for $C_{21}H_{16}Cl_2NOPNa$ 422.0244, found 422.0225.

3-(Diphenylphosphoryl)-2-(2-fluorophenyl)propanenitrile **(3m)**



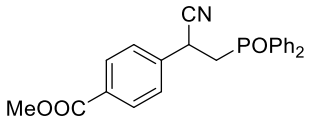
White solid; 120 mg, 86% yield. Mp: 122−124 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 (dd, J = 11.6, 7.4 Hz, 2H), 7.63 – 7.46 (m, 5H), 7.42 (t, J = 7.1 Hz, 1H), 7.34 (dd, J = 9.3, 5.8 Hz, 3H), 7.18 (dd, J = 13.3, 6.4 Hz, 1H), 7.01 (t, J = 7.5 Hz, 1H), 6.95 – 6.80 (m, 1H), 4.57 (dd, J = 16.4, 7.5 Hz, 1H), 3.07 – 2.85 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 160.02 (d, J = 248.8 Hz), 132.41 (d, J = 2.8 Hz), 132.08 (d, J = 39.3 Hz), 131.96 (d, J = 2.8 Hz), 131.07 (d, J = 39.2 Hz), 130.78 (d, J = 9.6 Hz), 130.73 (d, J = 8.4 Hz), 130.53 (d, J = 9.6 Hz), 129.98 (d, J = 3.1 Hz), 128.88 (d, J = 12.0 Hz), 128.61 (d, J = 12.0 Hz), 124.72 (d, J = 3.6 Hz), 122.10 (dd, J = 13.4, 6.7 Hz), 118.72 (d, J = 9.8 Hz), 115.98 (d, J = 20.9 Hz), 33.73 (d, J = 68.2 Hz), 25.84 (t, J = 2.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 26.71. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{21}H_{18}FNOP$ 350.1110, found 350.1113.

2-(2-Chlorophenyl)-3-(diphenylphosphoryl)propanenitrile **(3n)**



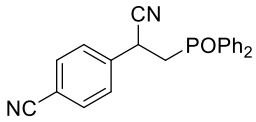
White solid; 130 mg, 89% yield. Mp: 128−130 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 – 7.75 (m, 2H), 7.70 – 7.63 (m, 2H), 7.59 – 7.44 (m, 5H), 7.42 – 7.35 (m, 2H), 7.25 (dd, J = 7.1, 2.2 Hz, 1H), 7.23 – 7.12 (m, 2H), 4.74 (ddd, J = 9.7, 8.0, 6.5 Hz, 1H), 3.00 – 2.87 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 132.24, 132.18 (d, J = 8.1 Hz), 131.94 (d, J = 2.8 Hz), 131.61 (d, J = 2.7 Hz), 130.58 (d, J = 3.0 Hz), 130.42 (d, J = 9.6 Hz), 130.17 (d, J = 9.6 Hz), 129.81, 129.55 (d, J = 3.2 Hz), 128.44, 128.32, 128.25, 128.13, 127.15, 118.16 (d, J = 7.3 Hz), 33.17 (d, J = 67.7 Hz), 28.47 (d, J = 2.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 26.90. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{21}H_{18}ClNOP$ 366.0815, found 366.0816.

Methyl 4-(1-cyano-2-(diphenylphosphoryl)ethyl)benzoate **(3o)**



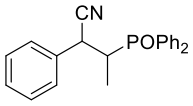
White solid; 124 mg, 80% yield. Mp: 147−149 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87 (d, J = 8.1 Hz, 2H), 7.78 (dd, J = 11.4, 7.6 Hz, 2H), 7.54 (dd, J = 18.9, 9.8 Hz, 5H), 7.45 – 7.31 (m, 5H), 4.51 (dd, J = 16.6, 7.2 Hz, 1H), 3.89 (s, 3H), 3.12 – 2.95 (m, 1H), 2.84 – 2.71 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 165.72, 139.49 (d, J = 6.6 Hz), 132.12 (d, J = 2.7 Hz), 131.62 (d, J = 2.7 Hz), 131.37 (d, J = 19.0 Hz), 130.41 (d, J = 9.7 Hz), 130.02 (d, J = 9.6 Hz), 129.89, 129.85, 128.49 (d, J = 12.1 Hz), 128.24 (d, J = 12.1 Hz), 128.03 (d, J = 12.1 Hz), 127.25, 118.80 (d, J = 9.3 Hz), 51.80, 35.45 (d, J = 67.8 Hz), 30.06 (d, J = 2.1 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 36.10. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{23}H_{21}NO_3P$ 390.1259, found 390.1257.

4-(1-Cyano-2-(diphenylphosphoryl)ethyl)benzonitrile **(3p)**



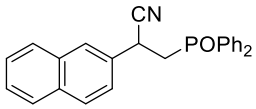
White solid; 127 mg, 84% yield. Mp: 194−196 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.86 – 7.67 (m, 2H), 7.62 – 7.43 (m, 10H), 7.36 (t, J = 6.8 Hz, 2H), 4.63 – 4.50 (m, 1H), 3.14 – 3.00 (m, 1H), 2.88 – 2.73 (m, 1H). ^{13}C NMR (101 MHz, DMSO-d_6): δ 140.90 (d, J = 7.8 Hz), 133.49 (d, J = 12.9 Hz), 132.58, 131.86 (d, J = 1.7 Hz), 131.55 (d, J = 2.6 Hz), 130.64 (d, J = 9.8 Hz), 130.48 (d, J = 9.7 Hz), 130.31 (d, J = 9.5 Hz), 129.11, 128.61 (d, J = 11.8 Hz), 128.48 (d, J = 11.9 Hz), 119.72 (d, J = 9.8 Hz), 118.35, 110.98, 32.31 (d, J = 68.0 Hz), 30.02 (d, J = 2.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 26.91. HRMS (ESI-TOF) *m/z*: $(M+Na)^+$ Calcd for $C_{22}H_{17}N_2NaOP$ 379.0976, found 379.0970.

3-(Diphenylphosphoryl)-2-phenylbutanenitrile **(3q)**



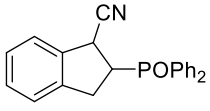
White solid; 110 mg, 80% yield. Mp: 180−182 °C. dr = 14:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 – 7.93 (m, 0.28H), 7.92 – 7.77 (m, 4H), 7.73 – 7.64 (m, 0.28H), 7.64 – 7.54 (m, 4H), 7.52 – 7.47 (m, 2H), 7.46 – 7.42 (m, 0.14H), 7.35 – 7.28 (m, 5H), 7.28 – 7.26 (m, 0.35H), 4.57 (dd, J = 9.0, 4.0 Hz, 1H), 4.40 (dd, J = 9.5, 6.8 Hz, 0.07H), 3.16 – 3.08 (m, 0.07H), 2.89 – 2.76 (m, 1H), 1.35 (dd, J = 16.1, 7.3 Hz, 3H), 1.09 (dd, J = 15.7, 7.4 Hz, 0.21H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 134.31, 134.22, 131.95 (d, J = 2.4 Hz), 131.84 (d, J = 2.4 Hz), 131.63 (d, J = 2.4 Hz), 131.46 (d, J = 2.4 Hz), 131.31, 131.13 (d, J = 8.8 Hz), 130.58 (d, J = 7.5 Hz), 130.39 (d, J = 8.9 Hz), 128.59, 128.56, 128.47, 128.34 (d, J = 2.6 Hz), 128.29, 128.21, 128.17, 128.08, 127.87, 127.34, 117.49 (d, J = 4.7 Hz), 38.33 (d, J = 68.8 Hz), 36.63 (d, J = 69.7 Hz), 36.47, 35.88, 10.99, 10.29. ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.96, 32.49. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{22}H_{21}NOP$ 346.1361, found 346.1359.

3-(Diphenylphosphoryl)-2-(naphthalen-2-yl)propanenitrile **(3r)**



White solid; 133 mg, 87% yield. Mp: 160−161 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 – 7.69 (m, 6H), 7.58 – 7.44 (m, 7H), 7.39 (dd, J = 8.5, 1.6 Hz, 1H), 7.26 – 7.11 (m, 3H), 4.64 (dt, J = 9.8, 7.2 Hz, 1H), 3.14 – 3.05 (m, 1H), 2.91 – 2.82 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 132.46 (d, J = 16.0 Hz), 131.99 (d, J = 2.8 Hz), 131.78 (d, J = 6.5 Hz), 131.60 (d, J = 14.7 Hz), 131.16 (d, J = 2.8 Hz), 130.60 (d, J = 15.3 Hz), 130.41 (d, J = 9.7 Hz), 129.90 (d, J = 9.6 Hz), 128.83, 128.49, 128.37, 127.97, 127.85, 127.43, 127.09, 126.74, 126.18, 124.02, 119.43 (d, J = 9.7 Hz)., 35.75 (d, J = 68.2 Hz), 30.28 (d, J = 2.2 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 27.14. ^{31}P NMR (162 MHz, $CDCl_3$): δ 26.91. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{25}H_{21}NOP$ 382.1361, found 382.1355.

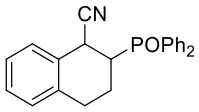
2-(Diphenylphosphoryl)-2,3-dihydro-1H-indene-1-carbonitrile **(3s)**



White solid; 121 mg, 88% yield. Mp: 174−176 °C. dr > 20:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01 – 7.87 (m, 4H), 7.66 – 7.53 (m, 6H), 7.37 (dd, J = 8.1, 4.7 Hz, 1H), 7.31 – 7.25 (m, 2H), 7.20 (dd, J = 8.1, 4.6 Hz, 1H), 4.66 (dd, J = 13.8, 9.5 Hz, 1H), 3.73 – 3.58 (m, 1H), 3.54 – 3.42 (m, 1H), 3.19 – 3.07 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 140.17 (d, J = 8.7 Hz), 135.98 (d, J = 7.6 Hz), 132.28 (d, J = 2.7 Hz), 131.91 (d, J = 2.7 Hz), 130.99 (d, J = 79.3 Hz), 130.72 (d, J = 9.4 Hz), 130.39, 130.30, 129.61, 128.64

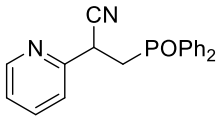
(d, J = 1.4 Hz), 128.52 (d, J = 1.4 Hz), 128.47, 127.33, 124.04 (d, J = 50.0 Hz), 119.18 (d, J = 2.6 Hz), 42.55 (d, J = 73.2 Hz), 34.35, 32.07. ^{31}P NMR (162 MHz, $CDCl_3$): δ 30.32. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{22}H_{19}NOP$ 344.1204, found 344.1212.

2-(Diphenylphosphoryl)-1,2,3,4-tetrahydronaphthalene-1-carbonitrile **(3t)**



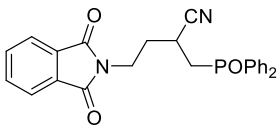
White solid; 90 mg, 63% yield. Mp: 187−189 °C. dr > 20:1. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97 – 7.90 (m, 2H), 7.89 – 7.82 (m, 2H), 7.60 – 7.47 (m, 6H), 7.33 (dd, J = 10.5, 5.4 Hz, 1H), 7.19 (dd, J = 6.0, 2.9 Hz, 2H), 7.10 – 7.04 (m, 1H), 4.45 (dd, J = 12.4, 8.3 Hz, 1H), 3.08 (ddd, J = 13.0, 9.4, 4.6 Hz, 1H), 2.85 (ddd, J = 15.2, 12.6, 4.2 Hz, 2H), 2.12 – 1.87 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 135.73 (d, J = 0.6 Hz), 132.14 (d, J = 2.8 Hz), 131.76 (d, J = 2.7 Hz), 130.88 (d, J = 9.1 Hz), 130.48 (d, J = 8.8 Hz), 129.83 (d, J = 16.3 Hz), 128.65, 128.58 (d, J = 2.5 Hz), 128.46 (d, J = 2.7 Hz), 128.32, 128.21, 128.09, 127.63, 126.61, 119.97 (d, J = 5.8 Hz), 36.77 (d, J = 70.5 Hz), 28.85, 27.52 (d, J = 9.7 Hz), 21.41 (d, J = 1.9 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.26. HRMS (ESI-TOF) *m/z*: $(M-CN+H)^+$ Calcd for $C_{22}H_{21}OP$ 332.1330, found 332.1301.

3-(Diphenylphosphoryl)-2-(pyridin-2-yl)propanenitrile **(3u)**



Yellow oil; 100 mg, 75% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.48 (d, J = 3.9 Hz, 1H), 7.85 – 7.74 (m, 2H), 7.67 – 7.48 (m, 6H), 7.46 – 7.41 (m, 1H), 7.39 – 7.33 (m, 3H), 7.15 (dd, J = 6.9, 4.9 Hz, 1H), 4.64 (dt, J = 10.3, 7.0 Hz, 1H), 3.40 – 3.24 (m, 1H), 3.15 – 3.03 (m, 1H). ^{13}C NMR (101 MHz, DMSO-d_6): δ 154.1 (d, J = 8.1 Hz), 149.4, 137.4, 133.6 (d, J = 6.4 Hz), 132.6 (d, J = 6.5 Hz), 131.9 (d, J = 2.6 Hz), 131.6 (d, J = 2.6 Hz), 130.6 (d, J = 9.6 Hz), 130.3 (d, J = 9.6 Hz), 128.6 (d, J = 11.8 Hz), 128.5 (d, J = 11.8 Hz), 123.4, 123.0, 119.6 (d, J = 9.2 Hz), 32.3 (d, J = 2.3 Hz), 31.7 (d, J = 68.9 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 27.75. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{20}H_{18}N_2OP$ 333.1157, found 333.1167.

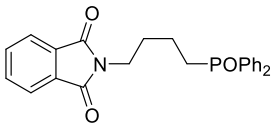
4-(1,3-Dioxoisoindolin-2-yl)-2-((diphenylphosphoryl)methyl)butanenitrile **(5a)**



White solid; 117 mg, 65% yield. Mp: 127−129 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.85 – 7.81 (m, 2H), 7.79 – 7.70 (m, 6H), 7.56 – 7.44 (m, 6H), 3.83 (t, J = 6.3 Hz, 2H), 3.16 – 3.06 (m, 1H), 2.81 – 2.71 (m,

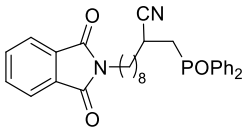
1H), 2.69 – 2.57 (m, 1H), 2.34 – 2.25 (m, 1H), 2.17 – 2.05 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 167.73, 133.64, 132.43, 132.01 (d, J = 2.7 Hz), 131.90 (d, J = 2.8 Hz), 131.44, 130.56 (d, J = 9.5 Hz), 130.05 (d, J = 9.6 Hz), 129.54, 128.54 (d, J = 2.4 Hz), 128.42 (d, J = 2.4 Hz), 122.95, 119.67 (d, J = 11.3 Hz), 34.50, 31.39 (d, J = 58.4 Hz), 31.02 (d, J = 5.0 Hz), 22.74 (d, J = 2.2 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 28.23. HRMS (ESI-TOF) *m/z*: $(M+Na)^+$ Calcd for $C_{25}H_{21}N_2NaO_3P$ 451.1187, found 451.1175.

2-(4-(Diphenylphosphoryl)butyl)isoindoline-1,3-dione **(5a')[1]**



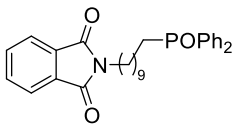
White solid; 28 mg, 14% yield. Mp: 102−104 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 – 7.67 (m, 8H), 7.53 – 7.41 (m, 6H), 3.66 (t, J = 7.0 Hz, 2H), 2.41 – 2.28 (m, 2H), 1.90 – 1.76 (m, 2H), 1.71 – 1.58 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 168.32, 133.94, 132.77 (d, J = 98.3 Hz), 132.03, 131.73 (d, J = 2.6 Hz), 130.78 (d, J = 9.3 Hz), 128.66 (d, J = 11.6 Hz), 123.21, 36.98, 29.51 (d, J = 4.5 Hz), 29.08 (d, J = 61.7 Hz), 18.66 (d, J = 3.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.47. MS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{24}H_{23}NO_3P$ 404.1, found 404.1.

10-(1,3-Dioxoisoindolin-2-yl)-2-((diphenylphosphoryl)methyl)decanenitrile **(5b)**



White solid; 109 mg, 53% yield. Mp: 107−108 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.08 – 7.67 (m, 6H), 7.67 – 7.50 (m, 8H), 3.45 (t, J = 6.8 Hz, 2H), 3.18 – 3.08 (m, 1H), 2.81 – 2.73 (m, 1H), 2.58 – 2.49 (m, 1H), 1.92 – 1.84 (m, 3H), 1.48 – 1.40 (m, 3H), 1.30 (d, J = 4.4 Hz, 8H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 132.57, 132.05 (d, J = 2.7 Hz), 131.87 (d, J = 2.7 Hz), 131.57, 130.90 (s), 130.54 (d, J = 9.5 Hz), 130.03 (d, J = 9.5 Hz), 129.03, 128.55 (d, J = 2.4 Hz), 128.43 (d, J = 2.3 Hz), 120.51 (d, J = 10.9 Hz), 119.59, 33.50, 32.95 (d, J = 5.7 Hz), 32.24, 32.08 (d, J = 69.6 Hz), 28.51, 28.18, 28.05, 27.55, 26.25, 24.83 (d, J = 2.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 26.38. HRMS (ESI-TOF) *m/z*: $(M\text{-}PhCOCON)^+$ Calcd for $C_{23}H_{29}NOP$ 366.1987, found 366.1972.

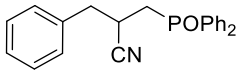
2-(10-(Diphenylphosphoryl)decyl)isoindoline-1,3-dione (5b')



White solid; 47 mg, 25% yield. Mp: 90−93 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.81 (dd, J = 5.3, 3.0 Hz,

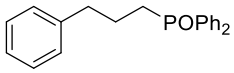
2H), 7.76 – 7.64 (m, 6H), 7.52 – 7.41 (m, 6H), 3.64 (t, J = 7.3 Hz, 2H), 2.30 – 2.17 (m, 2H), 1.68 – 1.52 (m, 4H), 1.41 – 1.32 (m, 2H), 1.30 – 1.17 (m, 10H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 168.44, 133.83, 132.14, 131.62, 130.82, 130.73, 128.61 (d, J = 10.5 Hz), 123.13, 38.03, 30.98, 30.86, 29.32, 29.23, 29.07, 28.98, 28.55, 26.80, 21.41. ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.69. HRMS (ESI-TOF) *m/z*: (M-PhCOCO+Na+H_2O)$^+$ Calcd for $C_{22}H_{32}NO_2PNa$ 396.2068, found 396.2087.

2-Benzyl-3-(diphenylphosphoryl)propanenitrile **(5c)**



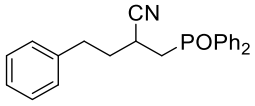
White solid; 84 mg, 57% yield. Mp: 145−146 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.83 – 7.67 (m, 4H), 7.61 – 7.46 (m, 6H), 7.36 – 7.26 (m, 3H), 7.25 – 7.19 (m, 2H), 3.43 – 3.23 (m, 1H), 3.15 (d, J = 11.9 Hz, 1H), 3.00 (d, J = 7.0 Hz, 1H), 2.68 – 2.56 (m, 1H), 2.40 – 1.89 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 140.32, 133.47, 131.29, 130.39, 130.30, 129.59 (d, J = 6.3 Hz), 128.90, 128.27, 128.16, 128.07, 127.96, 125.65, 115.11, 36.19 (d, J = 14.7 Hz), 28.87 (d, J = 6.3 Hz), 22.53. ^{31}P NMR (162 MHz, $CDCl_3$): δ 28.91. HRMS (ESI-TOF) *m/z*: (M+Na)$^+$ Calcd for $C_{22}H_{20}NNaOP$ 368.1180, found 368.1171.

Diphenyl(3-phenylpropyl)phosphine oxide **(5c')**[2]



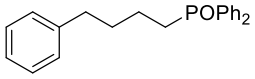
White solid; 29 mg, 18% yield. Mp: 114−116 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 – 7.64 (m, 4H), 7.53 – 7.44 (m, 6H), 7.28 – 7.24 (m, 2H), 7.21 – 7.16 (m, 1H), 7.14 – 7.08 (m, 2H), 2.72 (t, J = 6.8 Hz, 2H), 2.33 – 2.22 (m, 2H), 2.02 – 1.93 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 140.83, 132.95 (d, J = 98.1 Hz), 131.70 (d, J = 2.6 Hz), 130.75 (d, J = 9.2 Hz), 128.70, 128.56 (d, J = 6.5 Hz), 128.43, 126.12, 36.65 (d, J = 14.8 Hz), 28.94 (d, J = 72.1 Hz), 23.03 (d, J = 3.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.50. MS (ESI-TOF) *m/z*: (M+H)$^+$ Calcd for $C_{21}H_{22}OP$ 321.1, found 321.1.

2-((Diphenylphosphoryl)methyl)-4-phenylbutanenitrile **(5d)**



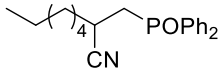
White solid; 76 mg, 50% yield. Mp: 79−80 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.72 (dd, J = 10.8, 7.6 Hz, 4H), 7.58 – 7.41 (m,6H), 7.23 (d, J = 7.5 Hz, 2H), 7.19 – 7.05 (m, 3H), 2.59 (t, J = 7.1 Hz, 2H), 2.47 – 2.36 (m, 1H), 2.36 – 2.24 (m, 2H), 1.79 – 1.66 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 141.39, 134.85, 133.31 (d, J = 7.1 Hz), 132.03, 131.56 (d, J = 2.1 Hz), 131.25, 130.70, 130.50 (d, J = 9.6 Hz), 130.00 (d, J = 9.5 Hz), 128.43 (d, J = 11.9 Hz), 128.18 (d, J = 12.0 Hz), 127.98 (d, J = 25.3 Hz), 126.79 (d, J = 78.0 Hz), 125.32, 119.46 (d, J = 7.4 Hz), 34.97 (d, J = 13.0 Hz), 26.63, 20.72, 18.78. ^{31}P NMR (162 MHz, $CDCl_3$): δ 33.21. HRMS (ESI-TOF) *m/z*: (M+Na)$^+$ Calcd for $C_{23}H_{22}NNaOP$ 382.1337, found 382.1331.

Diphenyl(4-phenylbutyl)phosphine oxide **(5d')**[2]



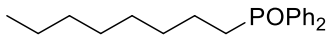
White solid; 40 mg, 24% yield. Mp: 148−150 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.76 – 7.66 (m, 4H), 7.50 – 7.38 (m, 6H), 7.25 – 7.17 (m, 2H), 7.16 – 7.05 (m, 3H), 2.56 (t, J = 5.6 Hz, 2H), 2.33 – 2.19 (m, 2H), 1.78 – 1.60 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 141.36, 132.61 (d, J = 97.9 Hz), 131.21 (d, J = 2.5 Hz), 130.27 (d, J = 9.2 Hz), 128.23, 128.12, 127.85, 125.32, 34.88, 32.15 (d, J = 14.4 Hz), 29.11 (d, J = 71.9 Hz), 20.71 (d, J = 3.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.38. MS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{22}H_{24}OP$ 335.2, found 335.2.

2-((Diphenylphosphoryl)methyl)pentanenitrile **(5e)**



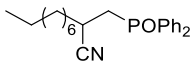
Colourless oil; 71 mg, 52% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 – 7.70 (m, 4H), 7.60 – 7.46 (m, 6H), 3.17 – 2.98 (m, 1H), 2.80 – 2.63 (m, 1H), 2.55 – 2.41 (m, 1H), 1.81 – 1.71 (m, 1H), 1.68 – 1.57 (m, 1H), 1.53 – 1.43 (m, 1H), 1.42 – 1.33 (m, 1H), 1.29 – 1.17 (m, 6H), 0.85 (t, J = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 132.72, 131.98 (d, J = 2.8 Hz), 131.80 (d, J = 2.8 Hz), 131.40 (d, J = 64.5 Hz), 130.54 (d, J = 9.5 Hz), 130.03 (d, J = 9.5 Hz), 128.51 (d, J = 2.0 Hz), 128.39 (d, J = 2.0 Hz), 120.56 (d, J = 10.8 Hz), 32.99 (d, J = 5.7 Hz), 32.12 (d, J = 69.7 Hz), 30.91, 27.98, 26.27, 24.83 (d, J = 2.4 Hz), 21.97, 13.50. ^{31}P NMR (162 MHz, $CDCl_3$): δ 28.24. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{21}H_{27}NOP$ 340.1830, found 340.1843.

Octyldiphenylphosphine oxide **(5e')**[2]



White solid; 61 mg, 39% yield. Mp: 56−59 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.77 – 7.69 (m, 4H), 7.53 – 7.44 (m, 6H), 2.33 – 2.18 (m, 2H), 1.66 – 1.56 (m, 2H), 1.43 – 1.35 (m, 2H), 1.29 – 1.21 (m, 10H), 0.85 (t, J = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 132.74 (d, J = 97.4 Hz), 132.05 (d, J = 9.1 Hz), 131.14, 131.11, 130.35, 130.26, 128.18, 128.06, 31.27, 30.50 (d, J = 14.7 Hz), 29.26 (d, J = 72.2 Hz), 29.22, 28.53, 22.11, 20.92 (d, J = 3.9 Hz), 13.58. ^{31}P NMR (162 MHz, $CDCl_3$): δ 32.58. MS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{20}H_{28}OP$ 315.2, found 315.2.

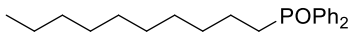
2-((Diphenylphosphoryl)methyl)pentanenitrile **(5f)**



Colourless oil; 104 mg, 47% yield. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.89 – 7.70 (m, 4H), 7.61 – 7.45 (m, 6H), 3.30 – 2.93 (m, 1H), 2.77 – 2.66 (m, 1H), 2.54 – 2.43 (m, 1H), 1.81 – 1.58 (m, 2H), 1.55 – 1.34 (m, 2H), 1.31 – 1.19 (m, 10H), 0.87 (t, J = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$); δ 132.03 (d, J = 2.4

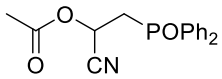
Hz), 131.83 (d, *J* = 2.4 Hz), 130.56 (d, *J* = 9.5 Hz), 130.05 (d, *J* = 9.4 Hz), 128.94, 128.53 (d, *J* = 1.9 Hz), 128.41 (d, *J* = 1.9 Hz), 120.54 (d, *J* = 10.6 Hz), 115.03, 33.03 (d, *J* = 5.7 Hz), 32.13 (d, *J* = 69.7 Hz), 31.37, 31.28, 28.66 (d, *J* = 8.3 Hz), 28.33, 26.32, 24.83 (d, *J* = 2.0 Hz), 22.13, 13.60. ^{31}P NMR (162 MHz, $CDCl_3$): *δ* 28.63. HRMS (ESI-TOF) *m/z*: $(M+Na)^+$ Calcd for $C_{23}H_{30}NNaOP$ 390.1963, found 390.1983.

Decyldiphenylphosphine oxide **(5f')**[3]



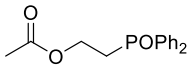
Colourless oil; 68 mg, 40% yield. ^{1}H NMR (400 MHz, $CDCl_3$): *δ* 7.73 (dd, *J* = 10.6, 7.6 Hz, 4H), 7.54 – 7.42 (m, 6H), 2.34 – 2.19 (m, 2H), 1.69 – 1.52 (m, 2H), 1.44 – 1.33 (m, 2H), 1.28 – 1.17 (m, 12H), 0.86 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): *δ* 132.05 (d, *J* = 2.7 Hz), 131.87 (d, *J* = 2.7 Hz), 130.59, 130.49, 130.08, 129.98, 128.55 (d, *J* = 2.4 Hz), 128.43 (d, *J* = 2.3 Hz), 33.50, 32.95 (d, *J* = 5.7 Hz), 32.24, 32.08 (d, *J* = 69.6 Hz), 29.22, 28.51, 28.11 (d, *J* = 13.1 Hz), 27.55, 26.25, 24.83 (d, *J* = 2.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): *δ* 33.11. MS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{22}H_{32}OP$ 343.2, found 343.2.

1-Cyano-2-(diphenylphosphoryl)ethyl acetate **(5g)**



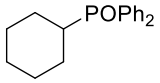
Light yellow solid; 65 mg, 52% yield. Mp: 97−99 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.79 – 7.68 (m, 4H), 7.58 – 7.46 (m, 6H), 5.75 (td, *J* = 9.2, 4.2 Hz, 1H), 3.12 – 3.01 (m, 1H), 2.92 – 2.81 (m, 1H), 1.69 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 167.87, 132.16, 131.89, 130.38 (d, *J* = 8.4 Hz), 130.16 (d, *J* = 8.5 Hz), 128.61, 128.50, 128.41, 128.30, 115.71 (d, *J* = 12.0 Hz), 55.52, 32.80 (d, *J* = 67.3 Hz), 19.21. ^{31}P NMR (162 MHz, $CDCl_3$): *δ* 25.40. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{17}H_{17}NO_3P$ 314.0946, found 314.0929.

2-(Diphenylphosphoryl)ethyl acetate **(5g')**



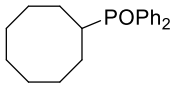
White solid; 46 mg, 32% yield. Mp: 101−103 °C. ^{1}H NMR (400 MHz, $CDCl_3$): *δ* 7.73 (dd, *J* = 11.1, 7.6 Hz, 4H), 7.53 – 7.43 (m, 6H), 4.41 (dt, *J* = 10.8, 7.3 Hz, 2H), 2.68 (dt, *J* = 11.6, 7.2 Hz, 2H), 1.77 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$): *δ* 170.27, 132.66, 131.66, 131.50, 131.47, 130.22, 130.13, 128.32, 128.20, 57.88, 29.19 (d, *J* = 70.3 Hz), 20.06. ^{31}P NMR (162 MHz, $CDCl_3$): *δ* 28.54. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{16}H_{18}O_3P$ 289.0994, found 289.0991.

***Cyclohexyldiphenylphosphine oxide* (5h')[3]**



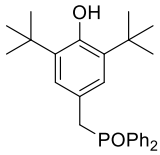
White solid, 77 mg, 68% yield, mp 165−166 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 – 7.72 (m, 4H), 7.54 – 7.44 (m, 6H), 2.34 – 2.22 (m, 2H), 1.85 – 1.66 (m, 5H), 1.61 – 1.48 (m, 2H), 1.28 – 1.17 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 131.95 (d, J = 2.4 Hz), 131.01, 130.64 (d, J = 8.5 Hz), 128.10 (d, J = 11.0 Hz), 36.74 (d, J = 72.4 Hz), 28.83, 25.97, 25.84, 25.28, 24.32. ^{31}P NMR (162 MHz, $CDCl_3$): δ 35.54. MS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{18}H_{22}OP$ 285.1, found 285.1.

***Cyclooctyldiphenylphosphine oxide* (5i')[3]**



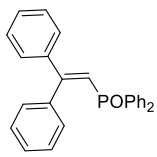
White solid, 81 mg, 64% yield, mp 143−145 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.93 – 7.70 (m, 4H), 7.54 – 7.42 (m, 6H), 2.58 – 2.39 (m, 1H), 2.28 – 2.06 (m, 2H), 1.97 – 1.77 (m, 2H), 1.76 – 1.68 (m, 3H), 1.64 – 1.52 (m, 5H), 1.47 – 1.38 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 132.20 (d, J = 93.9 Hz), 130.93, 130.56 (d, J = 8.3 Hz), 128.07 (d, J = 11.0 Hz), 35.14 (d, J = 70.5 Hz), 29.21, 26.47, 26.35, 25.91, 25.78, 25.47, 25.32. ^{31}P NMR (162 MHz, $CDCl_3$): δ 39.75. MS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{20}H_{26}OP$ 313.2, found 313.2.

***(3,5-Di-tert-butyl-4-hydroxybenzyl)diphenylphosphine oxide* (BHT-POPh$_2$) (6)**



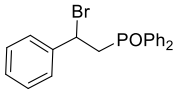
White solid, 30 mg, 35% yield, mp 177−179 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 – 7.62 (m, 4H), 7.53 – 7.48 (m, 2H), 7.47 – 7.40 (m, 4H), 6.73 (d, J = 2.1 Hz, 2H), 5.06 (s, 1H), 3.57 (d, J = 13.8 Hz, 2H), 1.28 (s, 18H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 152.30 (d, J = 3.4 Hz), 135.30 (d, J = 2.6 Hz), 132.02, 131.27 (d, J = 2.6 Hz), 131.00, 130.91, 127.97, 127.86, 126.50 (d, J = 5.0 Hz), 120.56 (d, J = 8.0 Hz), 37.48 (d, J = 67.2 Hz), 33.65, 29.64. ^{31}P NMR (162 MHz, $CDCl_3$): δ 30.91. HRMS (ESI-TOF) *m/z*: $(M+Na)^+$ Calcd for $C_{27}H_{33}NaO_2P$ 443.2116, found 443.2124.

***(2,2-Diphenylvinyl)diphenylphosphine oxide* (7)**



White solid, 51 mg, 74% yield, mp 217−218 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.73 – 7.63 (m, 4H), 7.39 – 7.28 (m, 11H), 7.25 – 7.20 (m, 2H), 7.15 – 7.05 (m, 3H), 6.84 – 6.74 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 161.67 (d, J = 2.2 Hz), 141.36 (d, J = 16.2 Hz), 137.50 (d, J = 6.7 Hz), 133.71 (d, J = 106.2 Hz), 130.64 (d, J = 2.6 Hz), 130.40 (d, J = 9.5 Hz), 129.82, 129.08, 128.17, 127.89 (d, J = 3.2 Hz), 127.82, 127.75, 127.12, 119.86 (d, J = 103.9 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 19.21. HRMS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{26}H_{22}OP$ 381.1408, found 381.1395.

***(2-Bromo-2-phenylethyl)diphenylphosphine oxide* (8)[3]**



White solid, 25 mg, 15% yield, mp 147−148 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.79 – 7.71 (m, 2H), 7.56 – 7.41 (m, 5H), 7.35 (td, J = 7.4, 1.4 Hz, 1H), 7.31 – 7.27 (m, 2H), 7.23 (dd, J = 7.3, 3.0 Hz, 2H), 7.16 – 7.05 (m, 3H), 5.62 – 5.55 (m, 1H), 3.48 – 3.32 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$): δ 140.92 (d, J = 4.3 Hz), 133.42, 132.42, 132.02 (d, J = 2.8 Hz), 131.43 (d, J = 2.8 Hz), 130.68 (d, J = 7.4 Hz), 130.58 (d, J = 7.5 Hz), 128.79, 128.63 (d, J = 7.0 Hz), 128.46, 128.31 (d, J = 12.0 Hz), 127.38, 46.40, 41.58 (d, J = 64.7 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 26.77. MS (ESI-TOF) *m/z*: $(M+H)^+$ Calcd for $C_{20}H_{19}BrOP$ 385.0, found 385.0.

References:

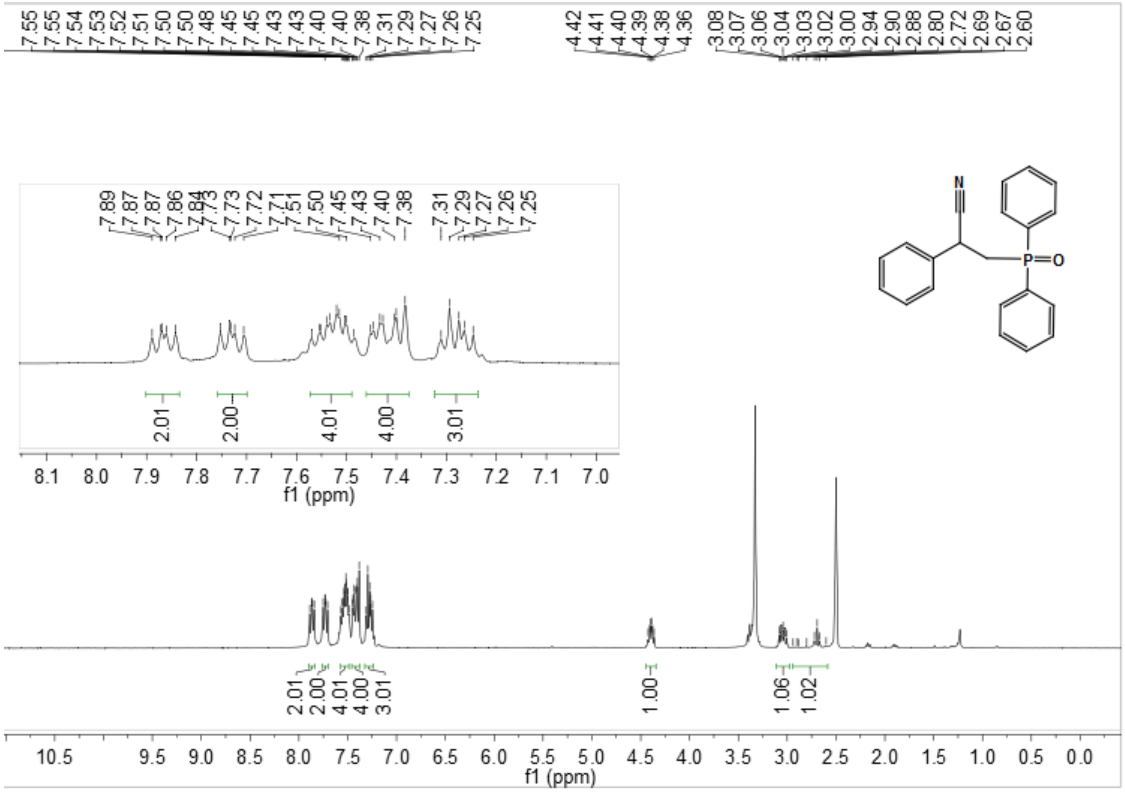
1. Novák, T.; Bakó, P.; Keglevich, G.; Dobó, A.; Vékey, K.; Tóke, L. *J. Incl. Phenom. Macro*. **2001**, *40*, 207.

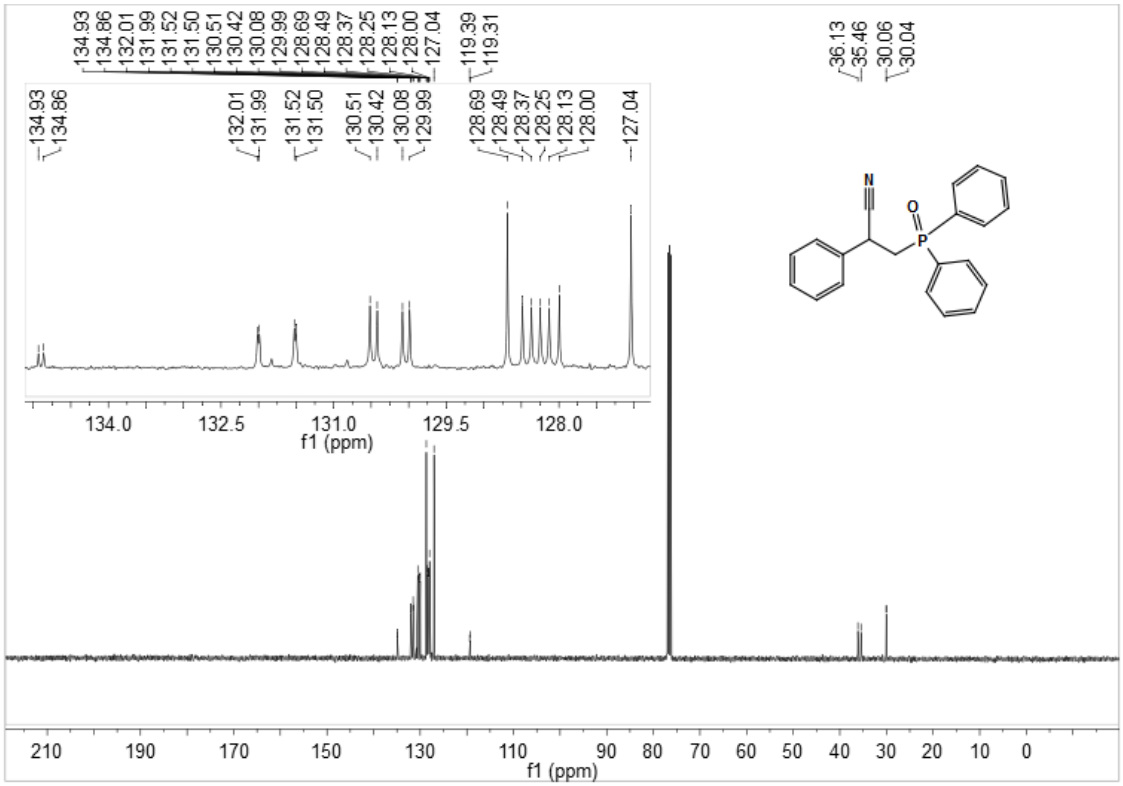
2. Zhou, S. F.; Li, D. P.; Liu, K.; Zou, J. P.; Asekun, O. T. *J. Org. Chem*. **2015**, *80*, 1214.

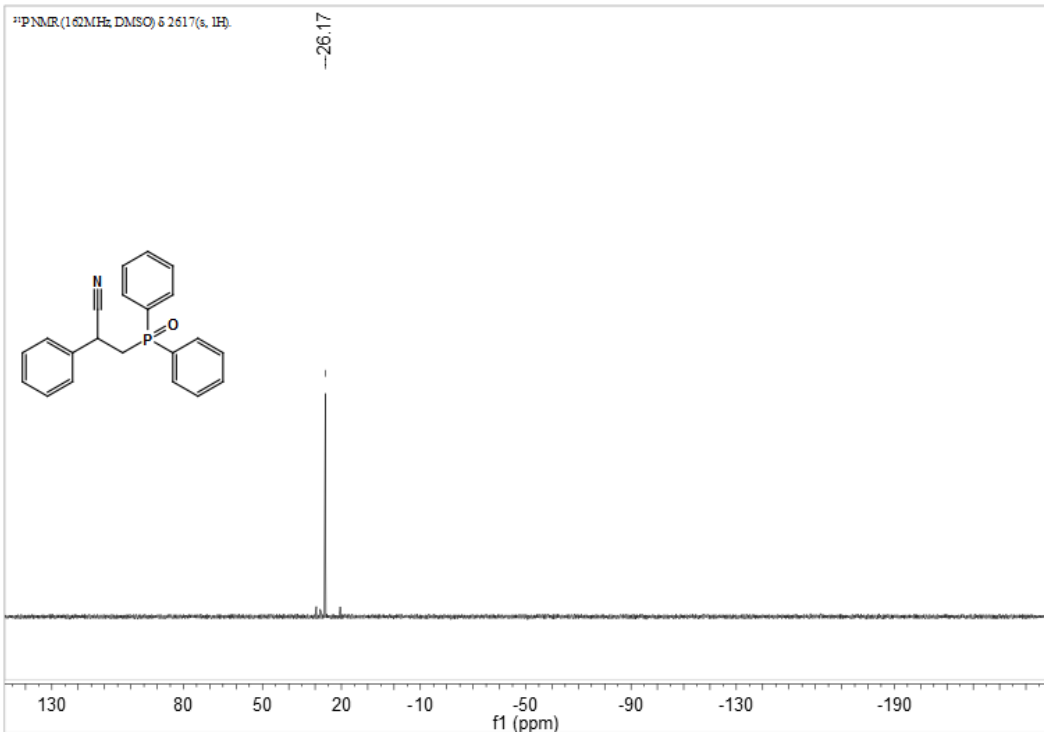
3. Yoo, W. J.; Kobayashi, S. *Green Chem*. **2013**, *15*, 1844.

7. ^{1}H, ^{13}C and ^{31}P NMR spectra of compounds 3 and 5-8

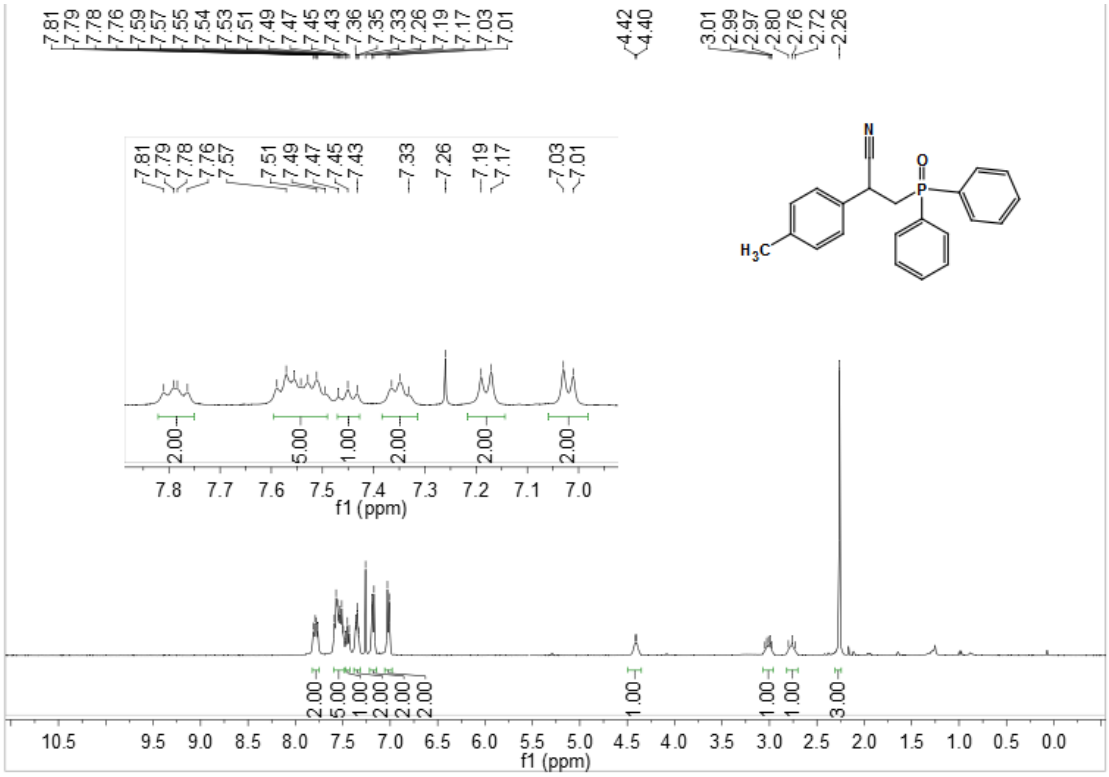
Compound 3a

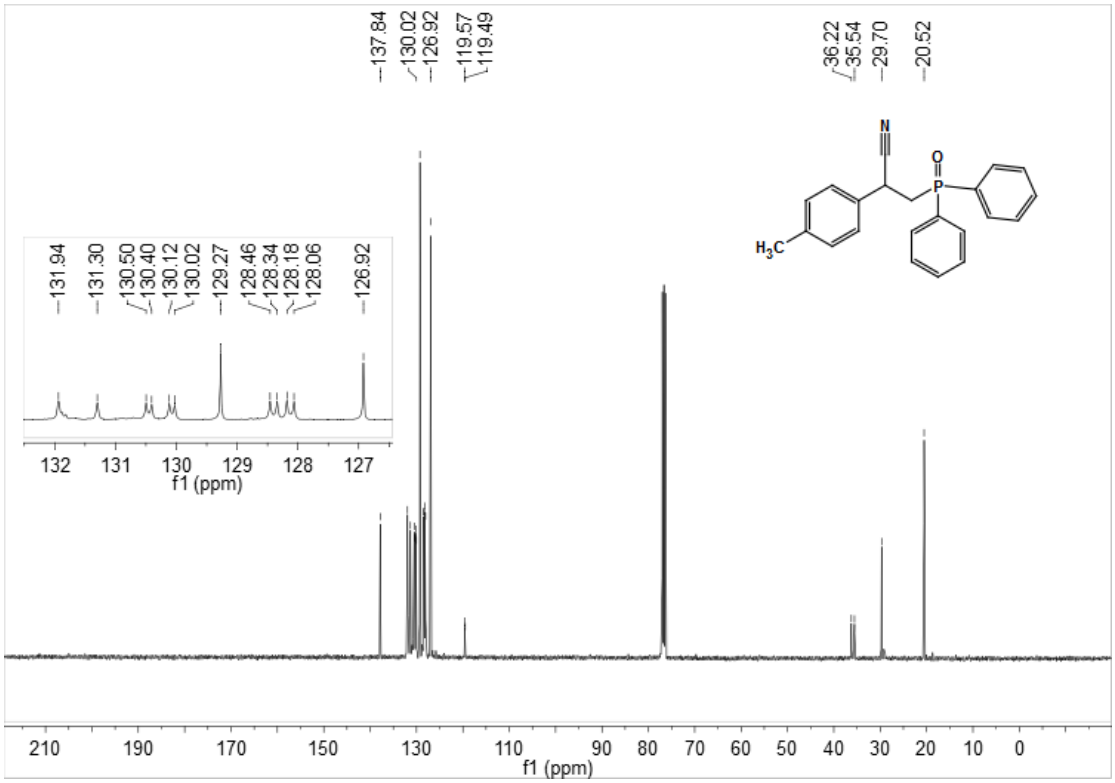


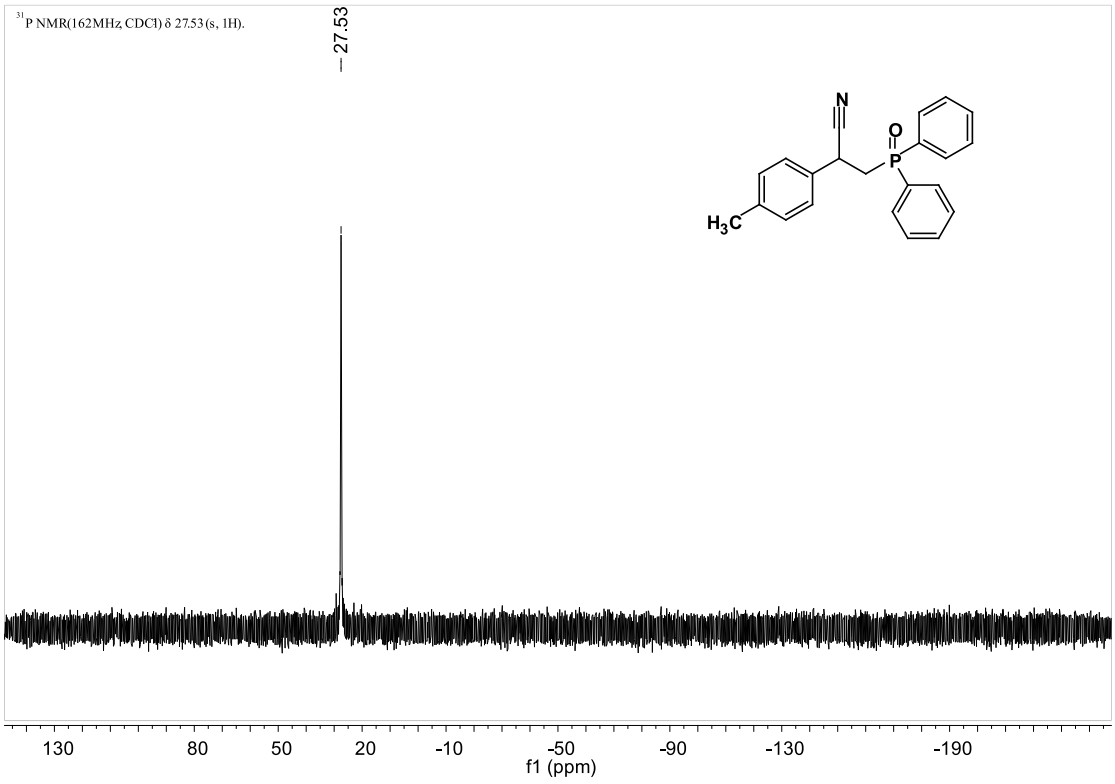



31P NMR (162 MHz, DMSO) δ 26.17 (s, 1H).
26.17
N
O
P
130
80
50
20
-10
-50
-90
-130
-190
f1 (ppm)

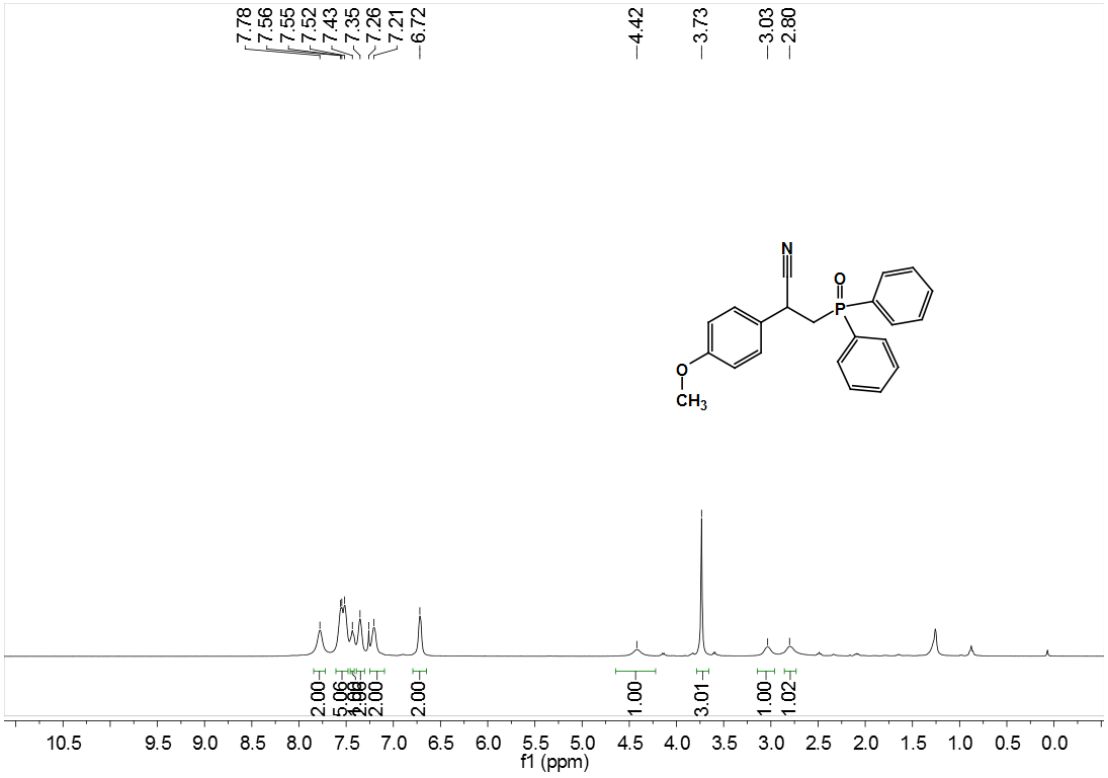
Compound 3b

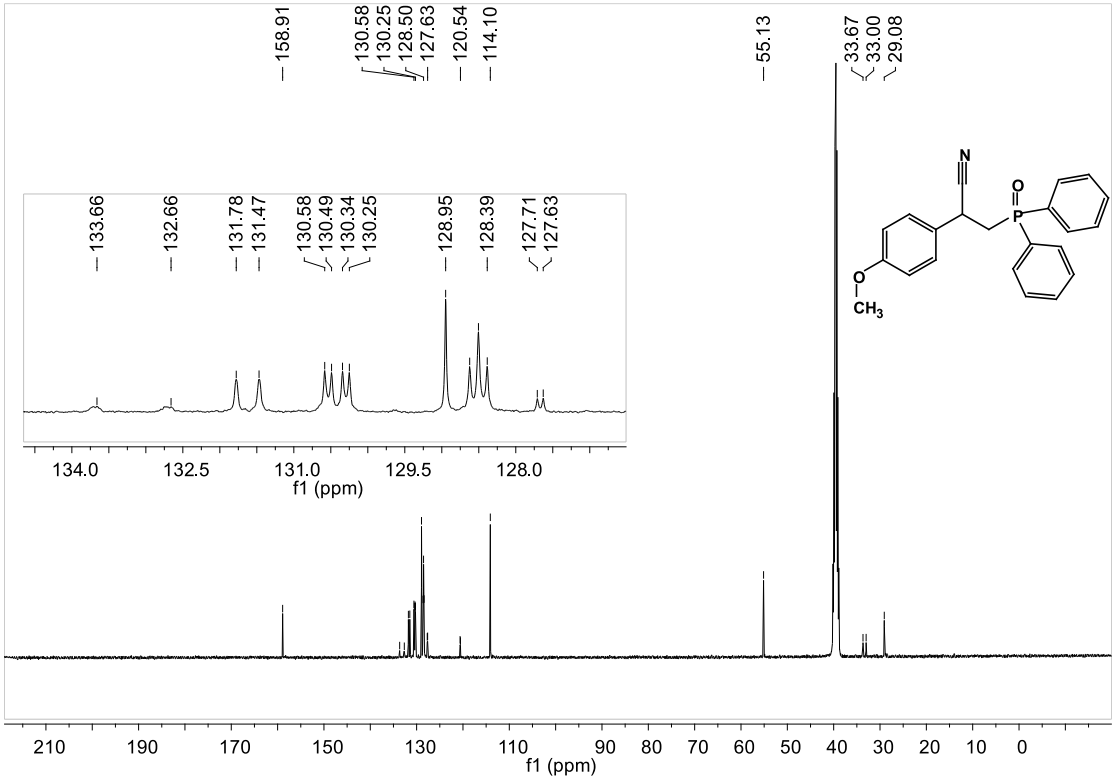

7.81
7.79
7.78
7.76
7.59
7.57
7.55
7.54
7.53
7.51
7.49
7.47
7.45
7.43
7.36
7.35
7.33
7.26
7.19
7.17
7.03
7.01
4.42
4.40
3.01
2.99
2.97
2.80
2.76
2.72
2.26
N
O
P
H3C
2.00
5.00
1.00
2.00
2.00
2.00
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
f1 (ppm)
2.00
5.00
1.00
2.00
2.00
1.00
1.00
1.00
3.00
10.5
9.5
9.0
8.5
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)

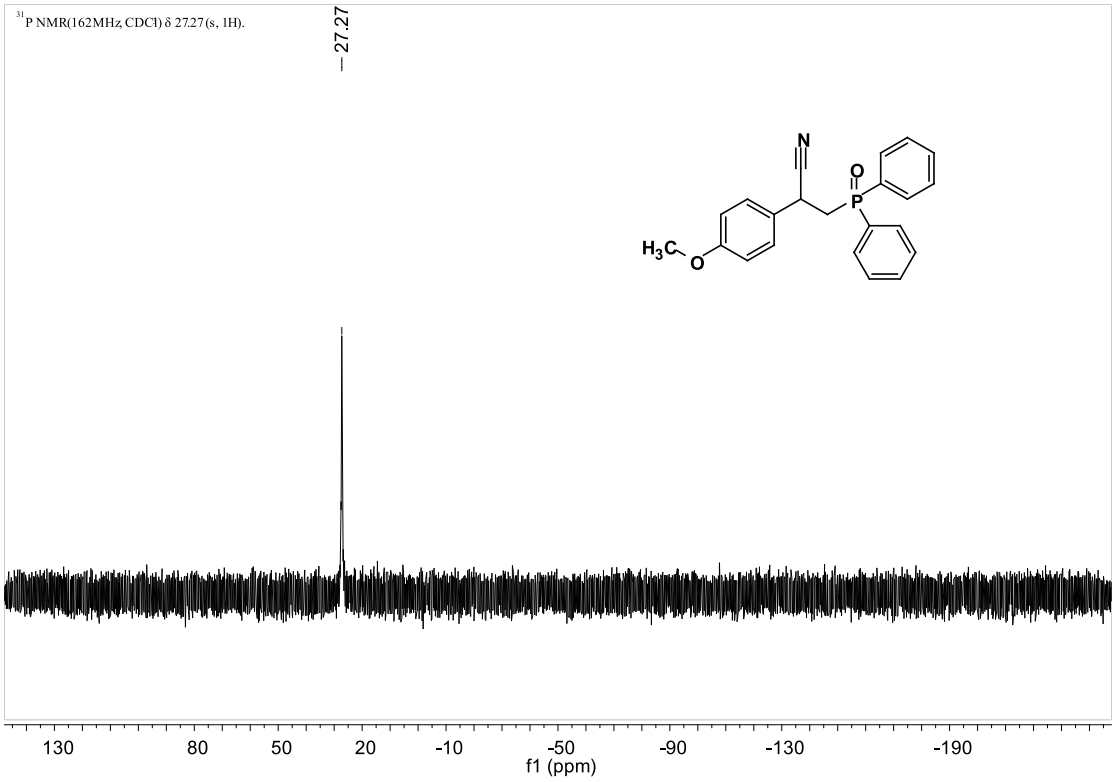

137.84
130.02
126.92
119.57
119.49
36.22
35.54
29.70
20.52
131.94
131.30
130.50
130.40
130.12
130.02
129.27
128.46
128.34
128.18
128.06
126.92
132 131 130 129 128 127
f1 (ppm)
N
O
P
H3C
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


31P NMR(162MHz, CDCl) δ 27.53 (s, 1H).
27.53
N
O
P
H3C
130 80 50 20 -10 -50 -90 -130 -190
f1 (ppm)

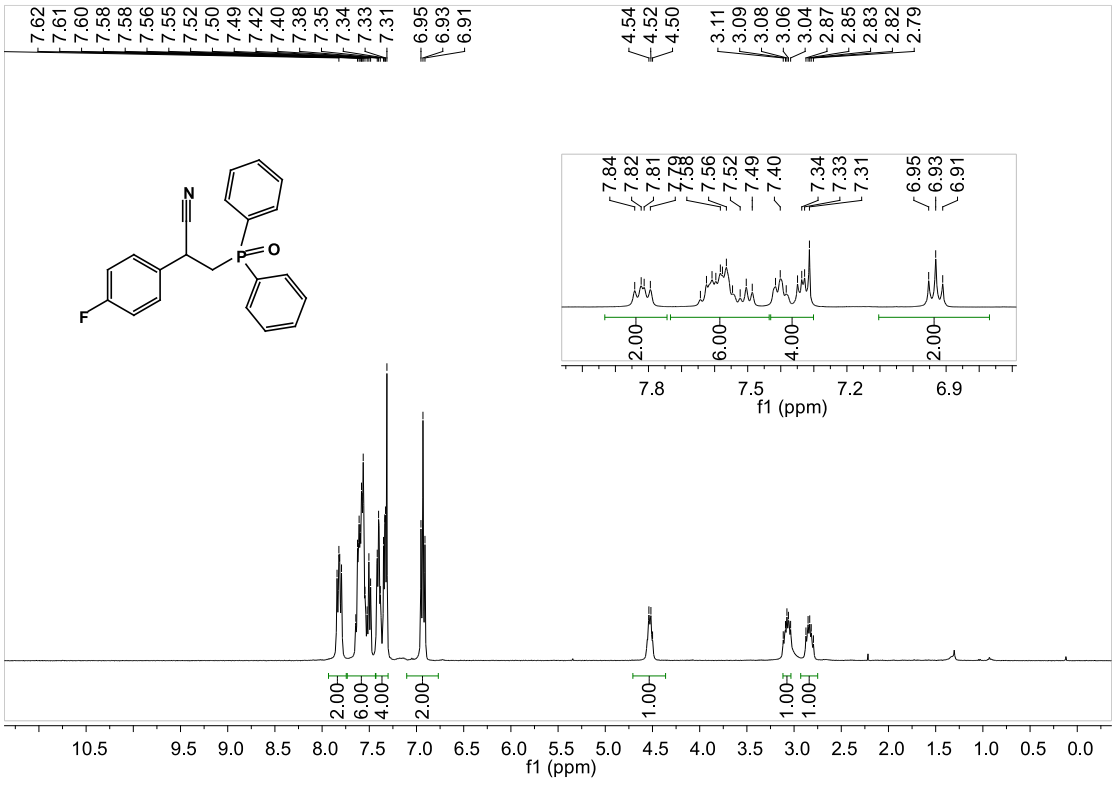
Compound 3c

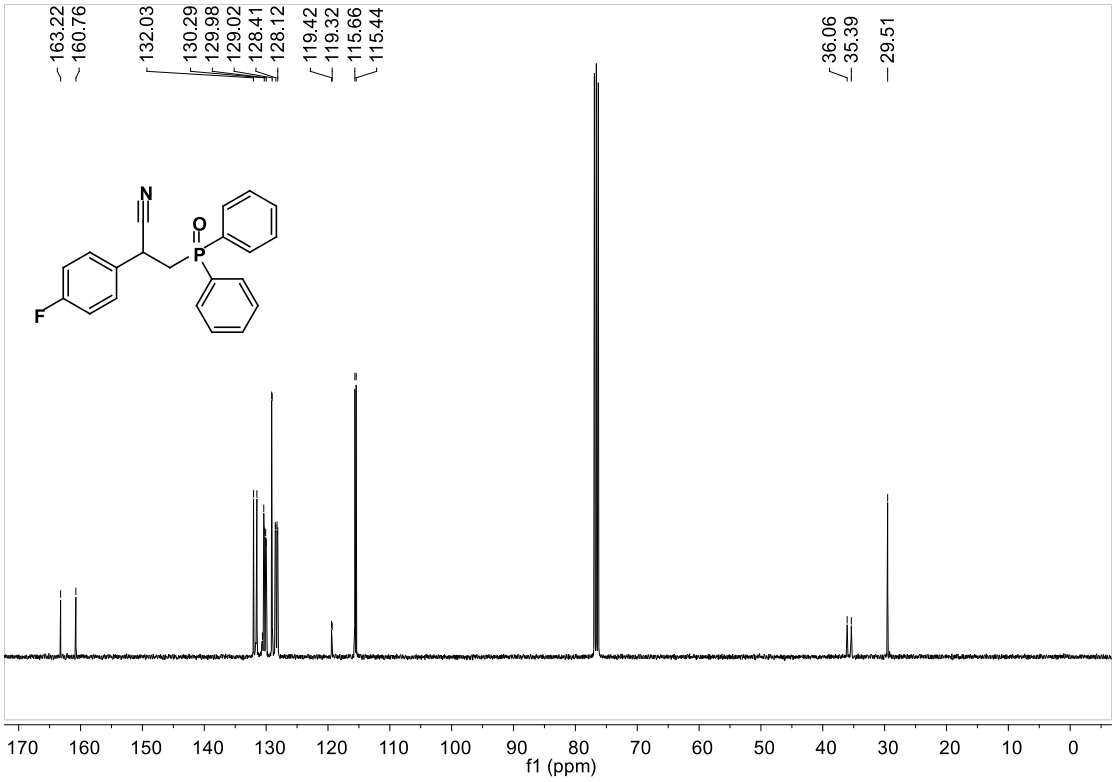

7.78
7.56
7.55
7.52
7.43
7.35
7.26
7.21
6.72
4.42
3.73
3.03
2.80
N
O
P
O
CH3
2.00
5.06
2.00
2.00
1.00
3.01
1.00
1.02
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

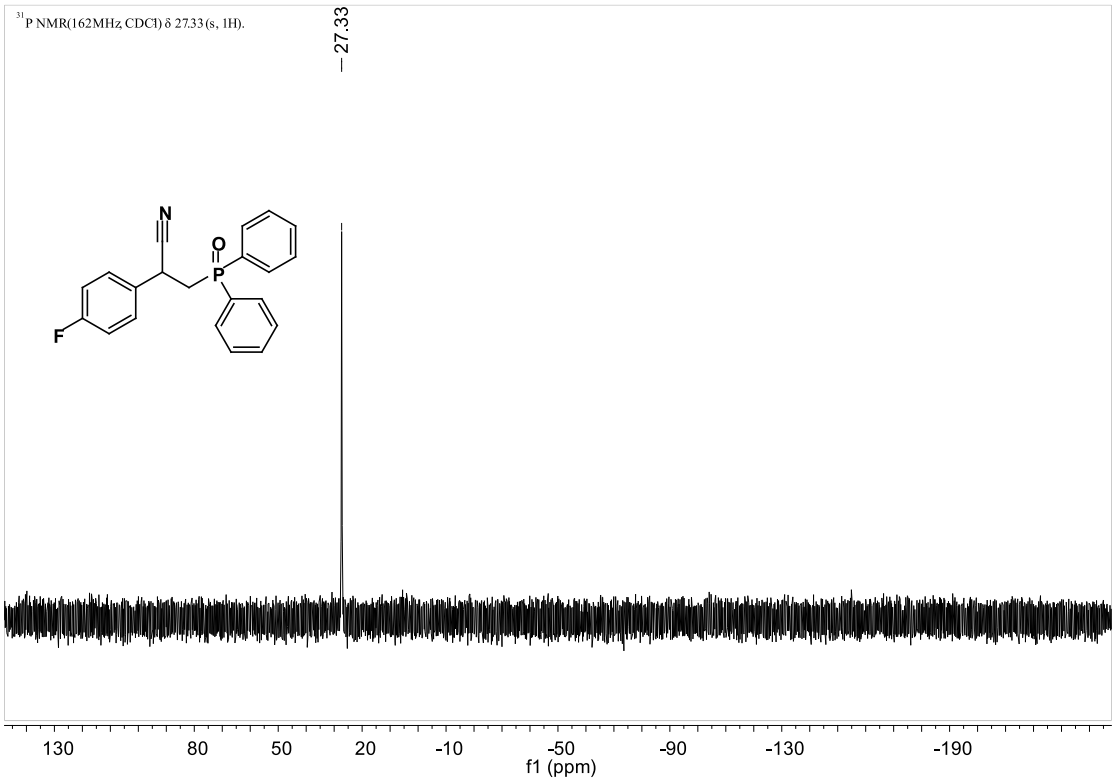

158.91
130.58
130.25
128.50
127.63
120.54
114.10
55.13
33.67
33.00
29.08
133.66
132.66
131.78
131.47
130.58
130.49
130.34
130.25
128.95
128.39
127.71
127.63
N
O
P
O
CH3
134.0
132.5
131.0
129.5
128.0
f1 (ppm)
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


31P NMR(162MHz, CDCl) δ 27.27 (s, 1H).
27.27
N
O
P
H3C
O
130
80
50
20
-10
-50
-90
-130
-190
f1 (ppm)

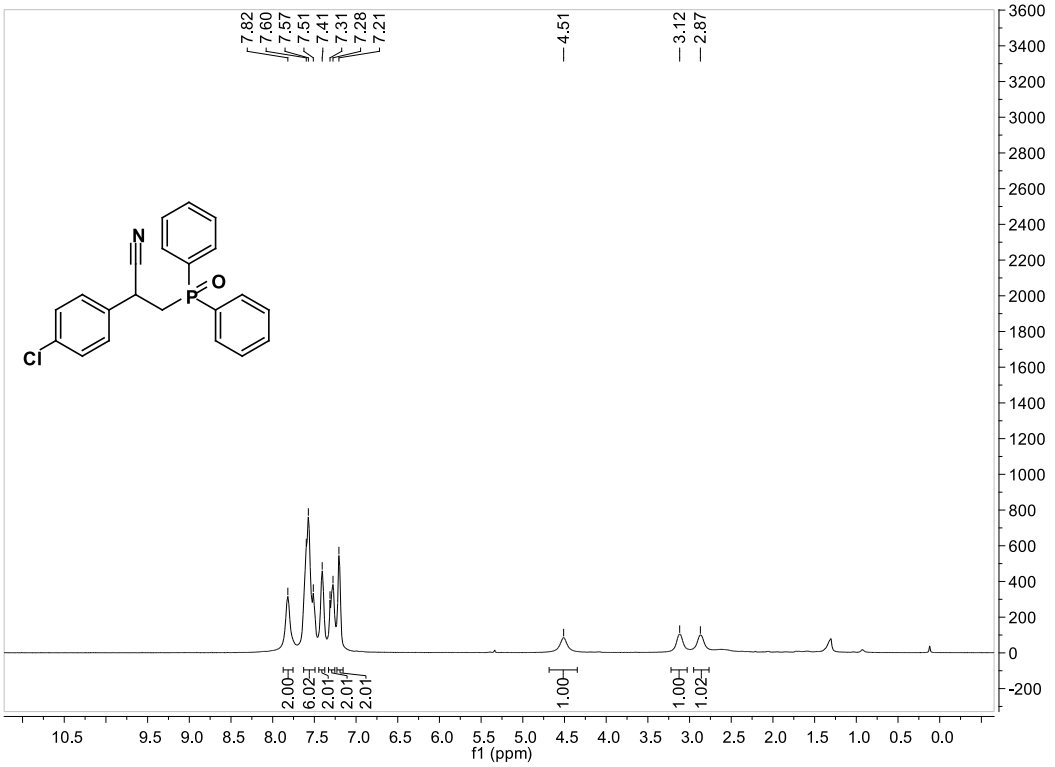
Compound 3d

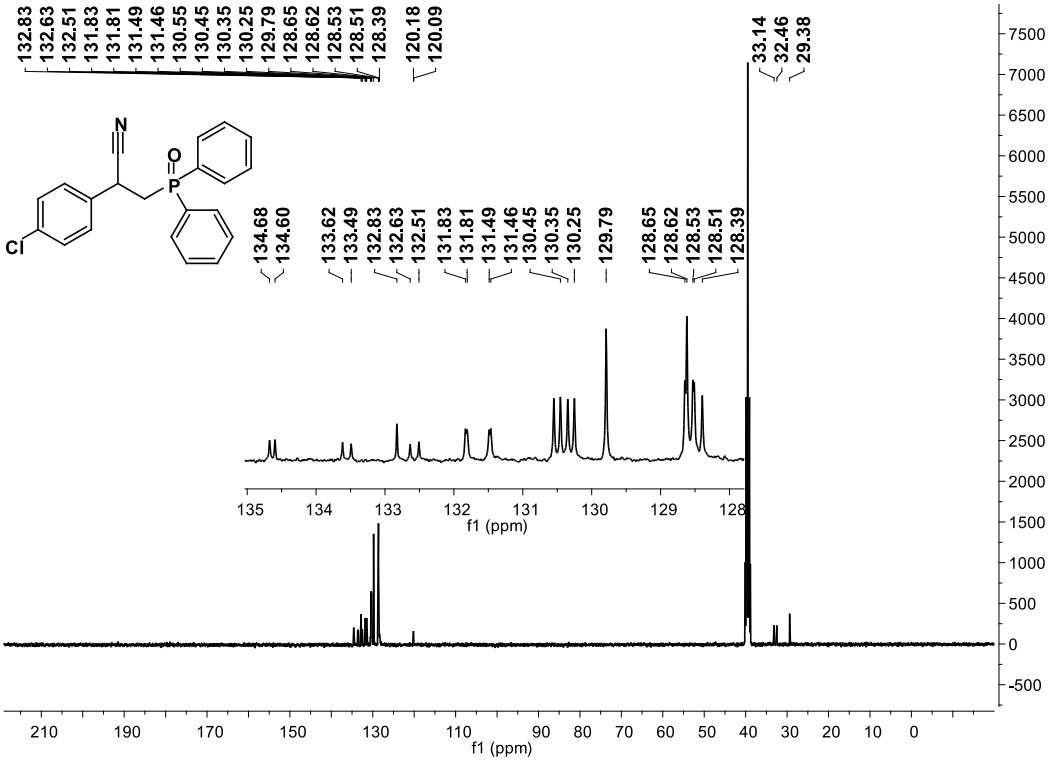

7.62
7.61
7.60
7.58
7.58
7.56
7.55
7.52
7.50
7.49
7.42
7.40
7.38
7.35
7.34
7.33
7.31
6.95
6.93
6.91
4.54
4.52
4.50
3.11
3.09
3.08
3.06
3.04
2.87
2.85
2.83
2.82
2.79
N
P
O
F
7.84
7.82
7.81
7.79
7.58
7.56
7.52
7.49
7.40
7.34
7.33
7.31
6.95
6.93
6.91
2.00
6.00
4.00
2.00
7.8
7.5
7.2
6.9
f1 (ppm)
2.00
6.00
4.00
2.00
1.00
1.00
1.00
10.5
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)

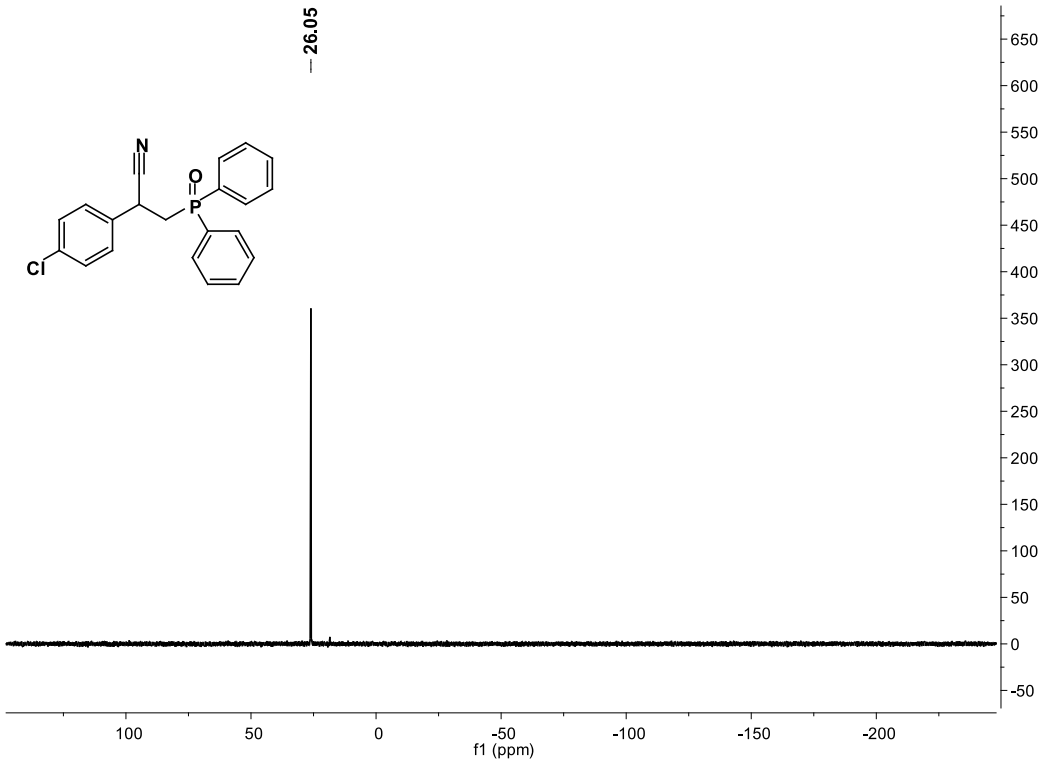

163.22
160.76
132.03
130.29
129.98
129.02
128.41
128.12
119.42
119.32
115.66
115.44
36.06
35.39
29.51
N
O
P
F
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


31P NMR(162MHz, CDCl) δ 27.33 (s, 1H).
27.33
N
O
P
F
130 80 50 20 -10 -50 -90 -130 -190
f1 (ppm)

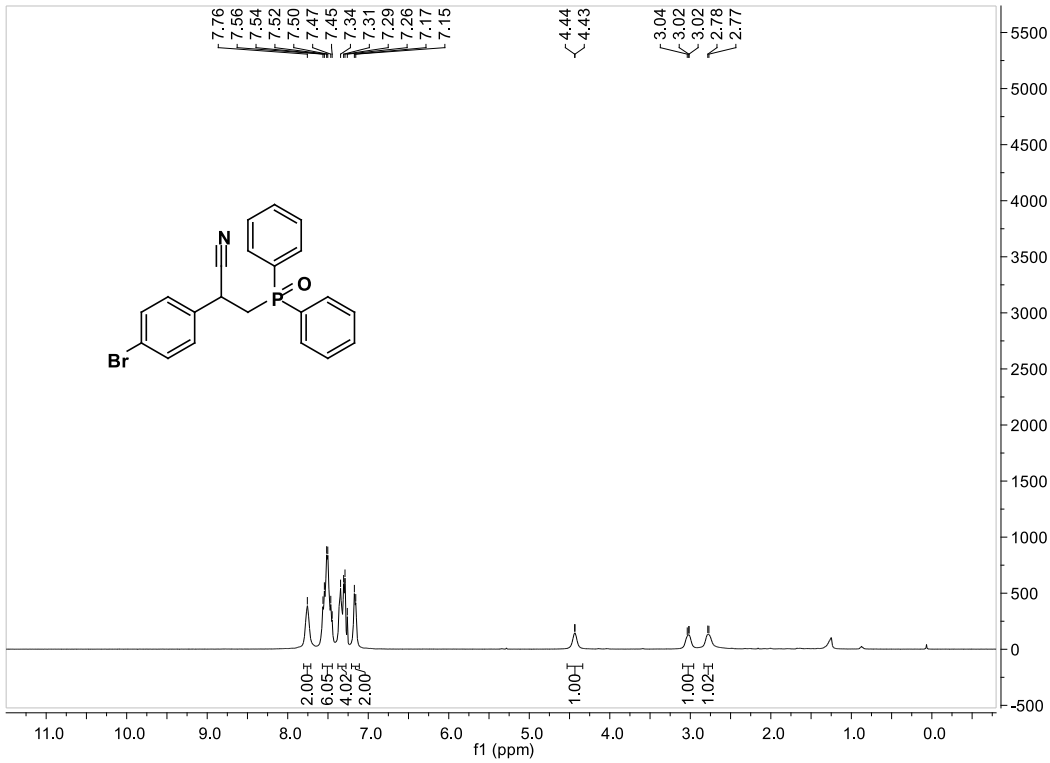
Compound 3e

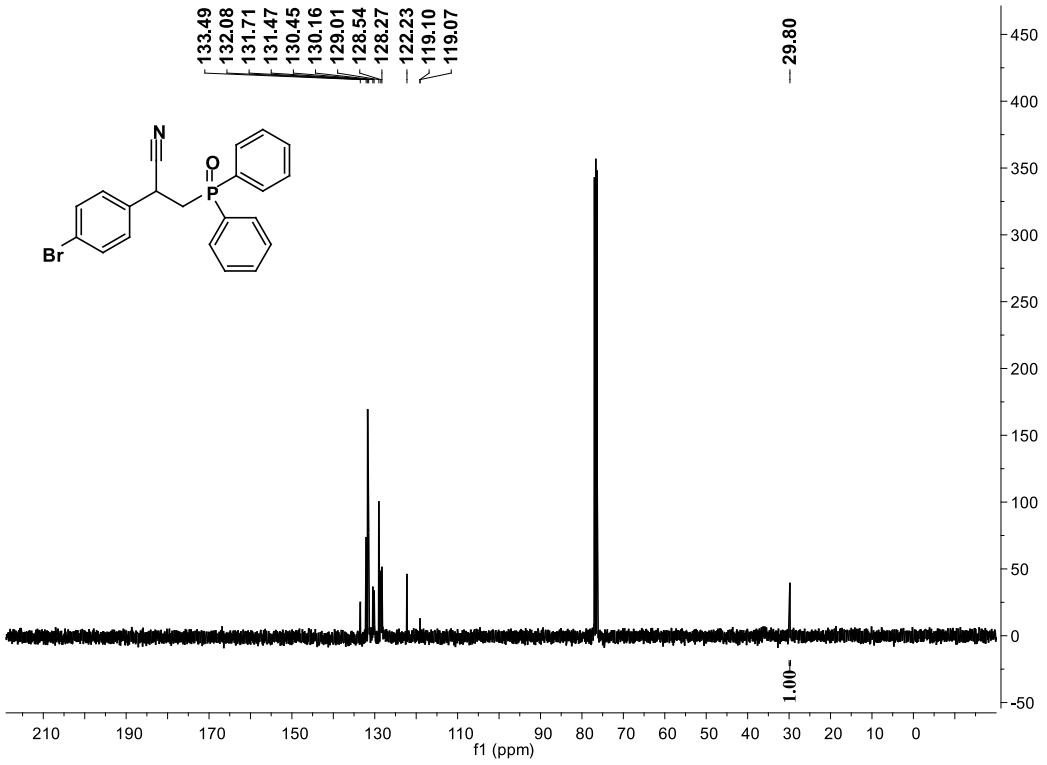

7.82
7.60
7.57
7.51
7.41
7.31
7.28
7.21
4.51
3.12
2.87
N
O
P
Cl
2.00
6.02
2.01
2.01
2.01
1.00
1.00
1.02
f1 (ppm)

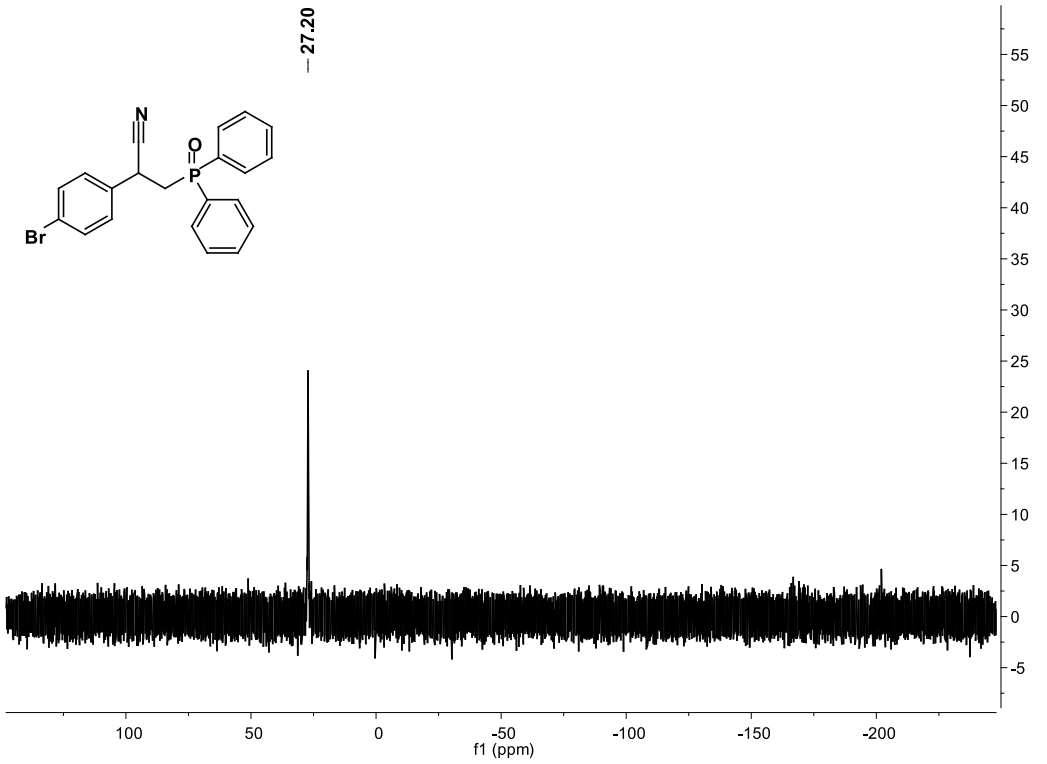

132.83
132.63
132.51
131.83
131.81
131.49
131.46
130.55
130.45
130.35
130.25
129.79
128.65
128.62
128.53
128.51
128.39
120.18
120.09
33.14
32.46
29.38
N
O
P
Cl
134.68
134.60
133.62
133.49
132.83
132.63
132.51
131.83
131.81
131.49
131.46
130.45
130.35
130.25
129.79
128.65
128.62
128.53
128.51
128.39
f1 (ppm)
f1 (ppm)


26.05
N
O
P
Cl
650
600
550
500
450
400
350
300
250
200
150
100
50
0
-50
100
50
0
-50
-100
-150
-200
f1 (ppm)

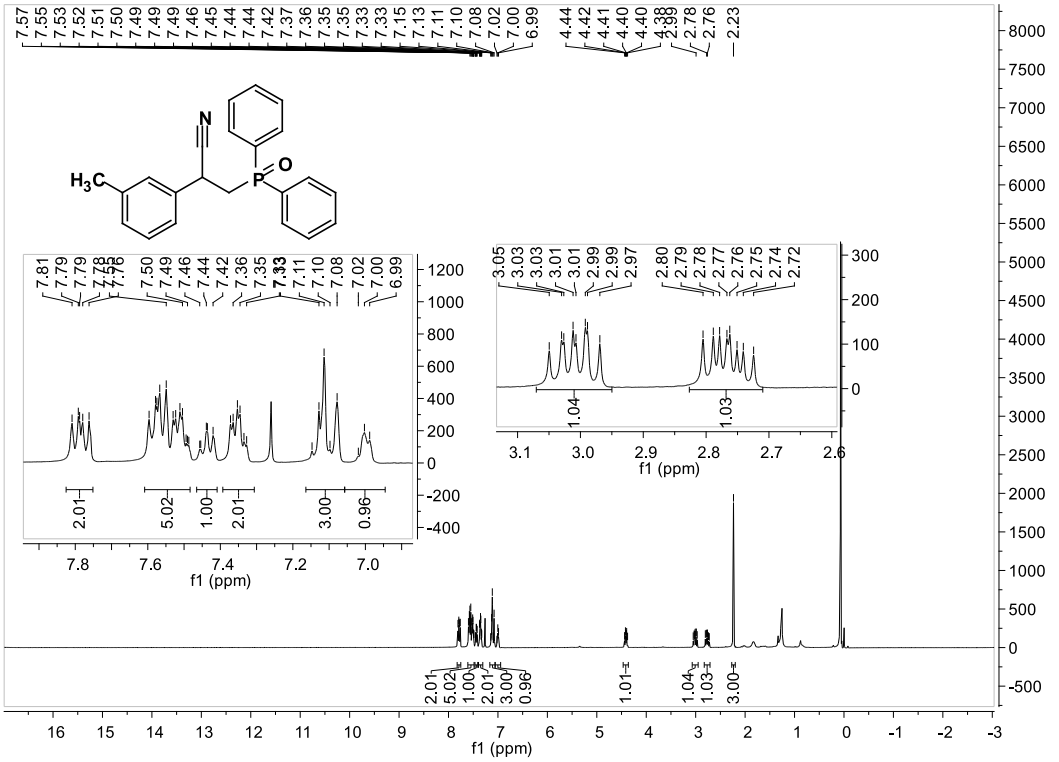
Compound 3f

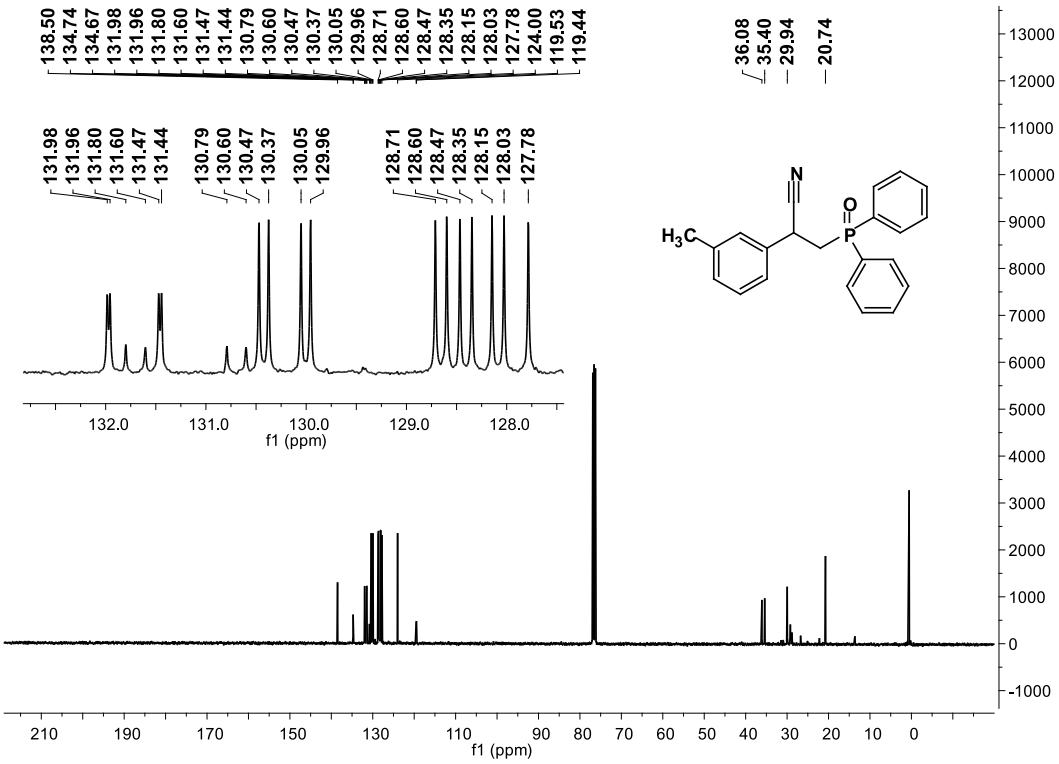

7.76
7.56
7.54
7.52
7.50
7.47
7.45
7.34
7.31
7.29
7.26
7.17
7.15
4.44
4.43
3.04
3.02
3.02
2.78
2.77
N
O
P
Br
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
2.00
6.05
4.02
2.00
1.00
1.00
1.02
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

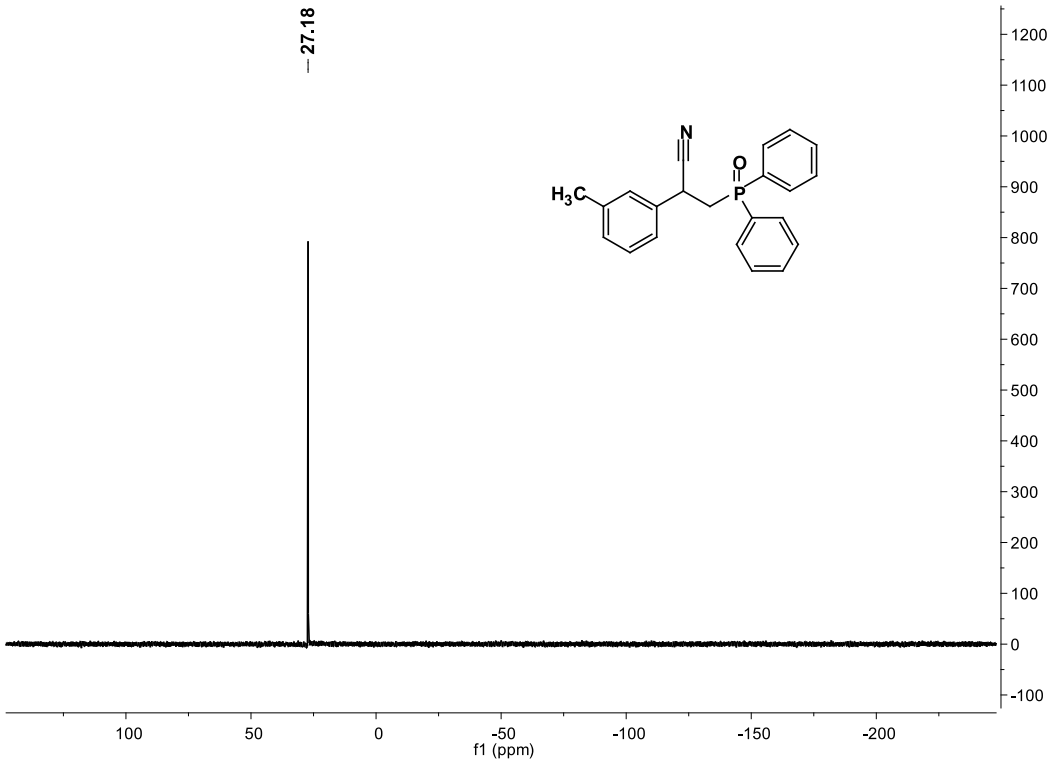

133.49
132.08
131.71
131.47
130.45
130.16
129.01
128.54
128.27
122.23
119.10
119.07
29.80
1.00
N
O
P
Br
f1 (ppm)


27.20
N
O
P
Br
f1 (ppm)

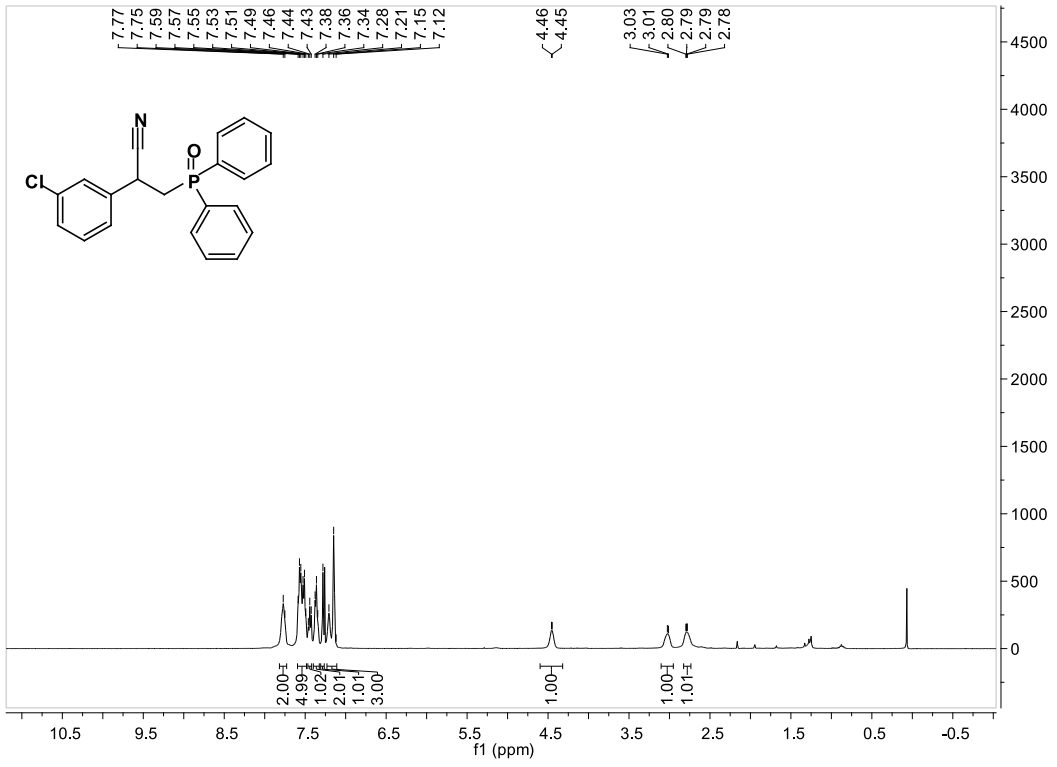
Compound 3g

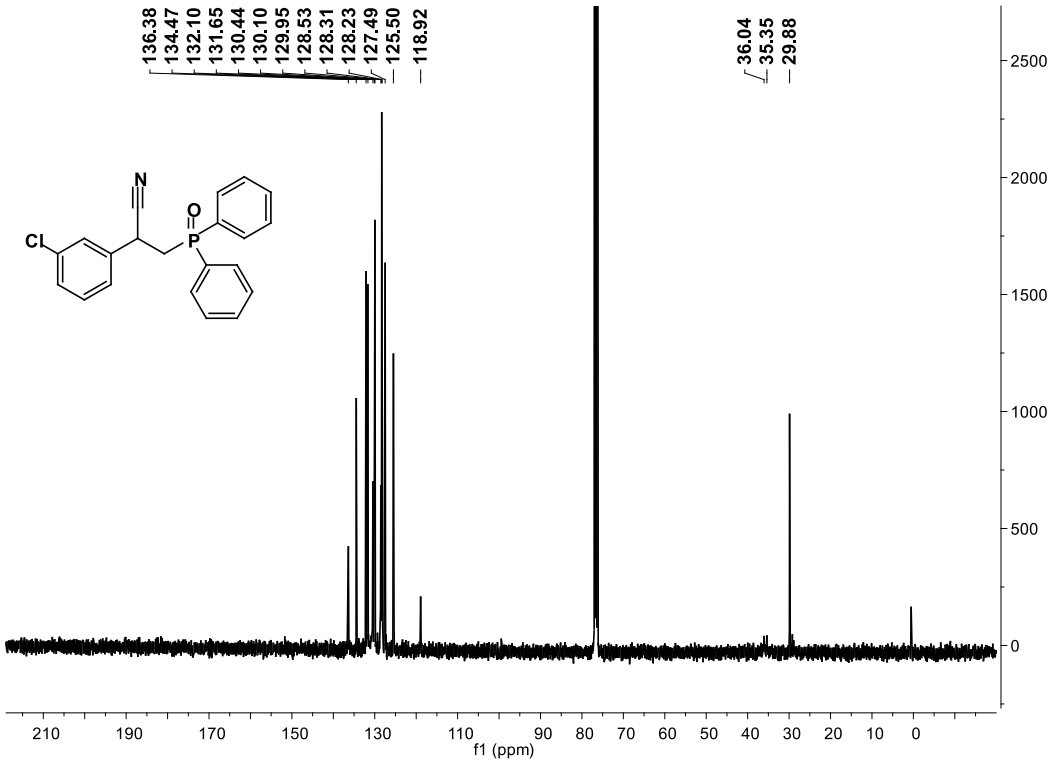

7.57 7.55 7.53 7.52 7.51 7.50 7.49 7.49 7.49 7.46 7.45 7.44 7.44 7.42 7.37 7.36 7.35 7.33 7.33 7.15 7.13 7.11 7.10 7.08 7.02 7.00 6.99
4.44 4.42 4.41 4.40 4.40 4.38 2.99 2.78 2.76 2.23
N
O
P
H3C
7.81 7.79 7.79 7.78 7.76 7.50 7.49 7.46 7.44 7.42 7.36 7.35 7.33 7.11 7.10 7.08 7.02 7.00 6.99
3.05 3.03 3.03 3.01 3.01 2.99 2.99 2.97 2.80 2.79 2.78 2.77 2.76 2.75 2.74 2.72
1.04 1.03
f1 (ppm)
2.01 5.02 1.00 2.01 3.00 0.96 1.01 1.04 1.03 3.00
f1 (ppm)

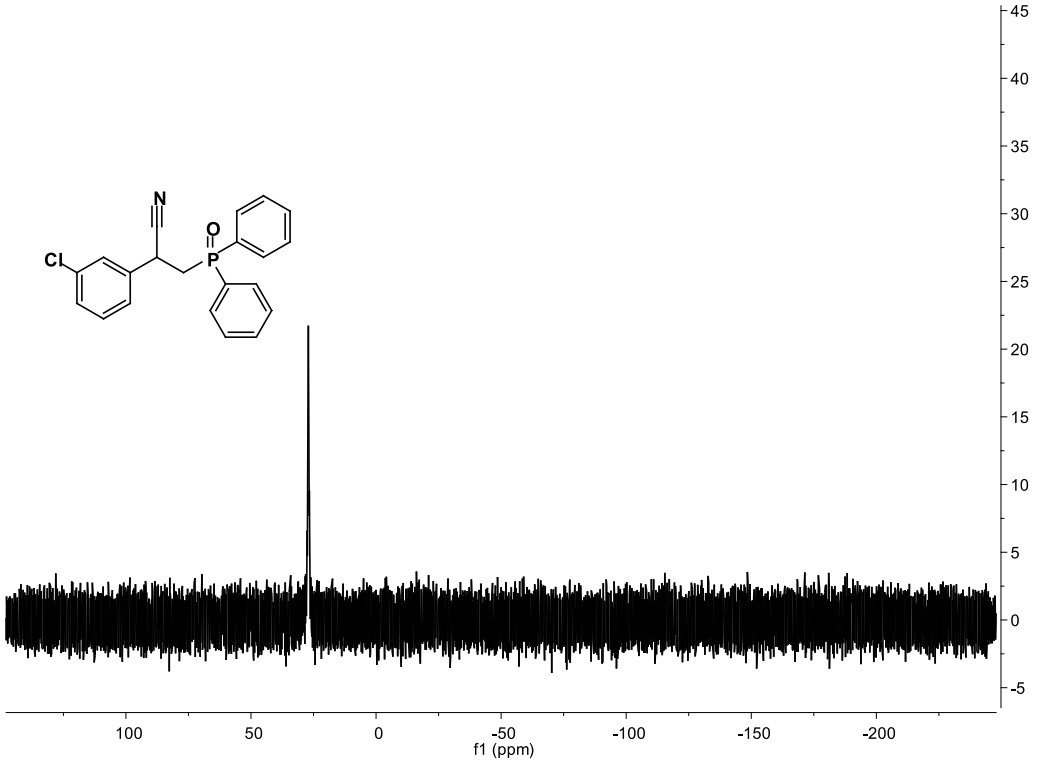

138.50 134.74 134.67 131.98 131.96 131.80 131.60 131.47 131.44 130.79 130.60 130.47 130.37 130.05 129.96 128.71 128.60 128.47 128.35 128.15 128.03 127.78 124.00 119.53 119.44
36.08 35.40 29.94 20.74
131.98 131.96 131.80 131.60 131.47 131.44 130.79 130.60 130.47 130.37 130.05 129.96 128.71 128.60 128.47 128.35 128.15 128.03 127.78
N
O
P
H3C
f1 (ppm)
f1 (ppm)


27.18
N
O
P
H3C
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
100
50
0
-50
-100
-150
-200
f1 (ppm)

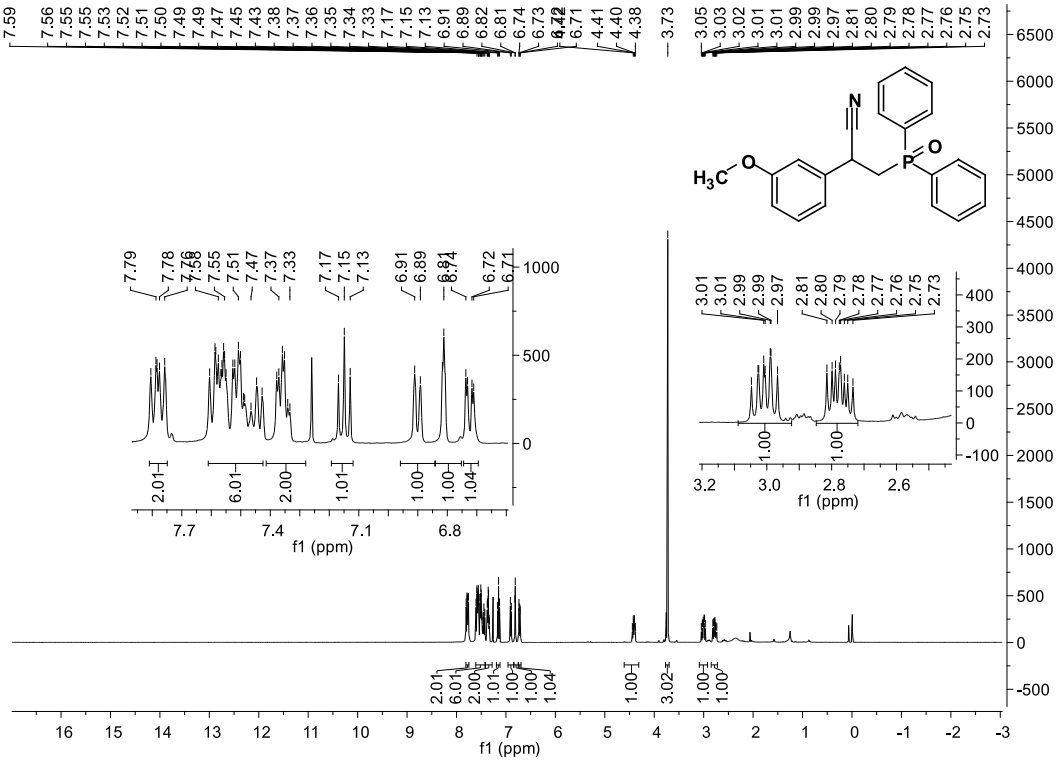
Compound 3h

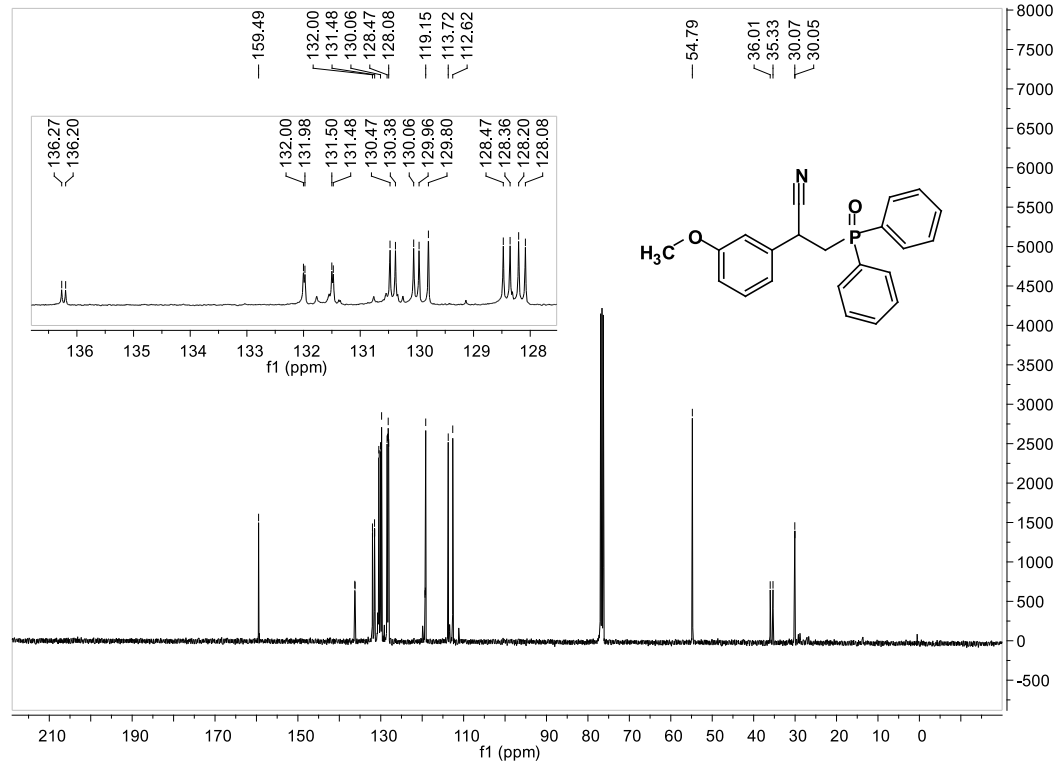

7.77
7.75
7.59
7.57
7.55
7.53
7.51
7.49
7.46
7.44
7.43
7.38
7.36
7.34
7.28
7.21
7.15
7.12
4.46
4.45
3.03
3.01
2.80
2.79
2.79
2.78
N
O
P
Cl
4500
4000
3500
3000
2500
2000
1500
1000
500
0
2.00
4.99
1.02
2.01
1.01
3.00
1.00
1.00
1.01
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

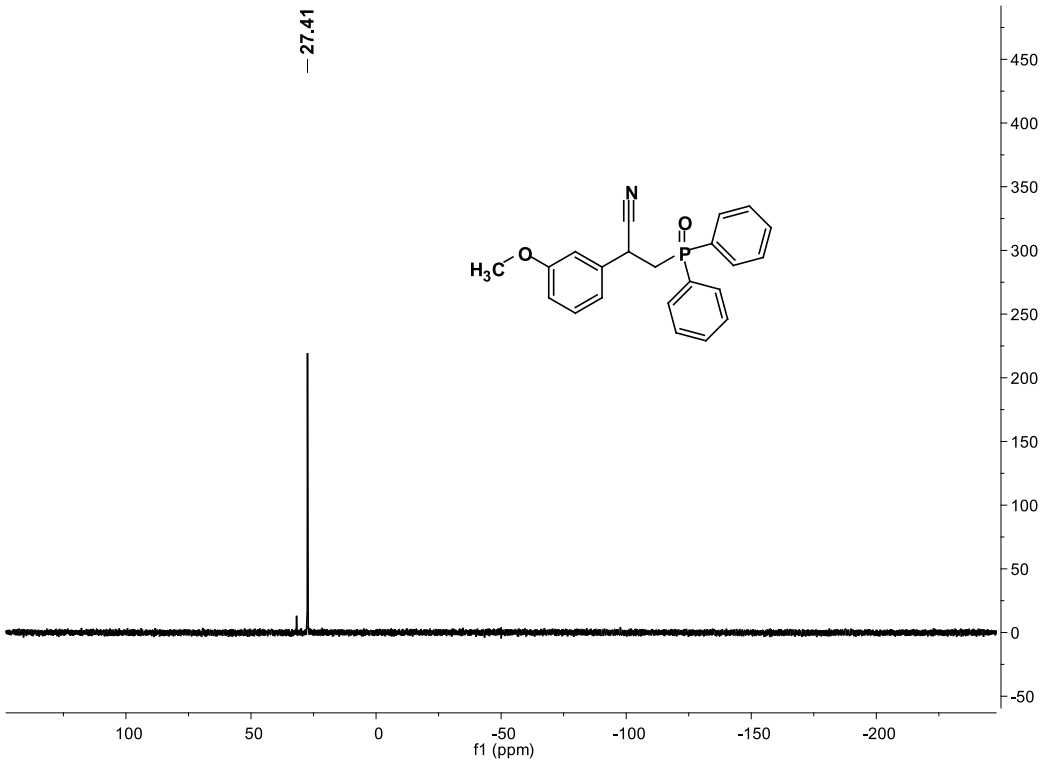

136.38
134.47
132.10
131.65
130.44
130.10
129.95
128.53
128.31
128.23
127.49
125.50
118.92
36.04
35.35
29.88
N
O
P
Cl
2500
2000
1500
1000
500
0
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


N
O
P
Cl
45
40
35
30
25
20
15
10
5
0
-5
100
50
0
-50
-100
-150
-200
f1 (ppm)

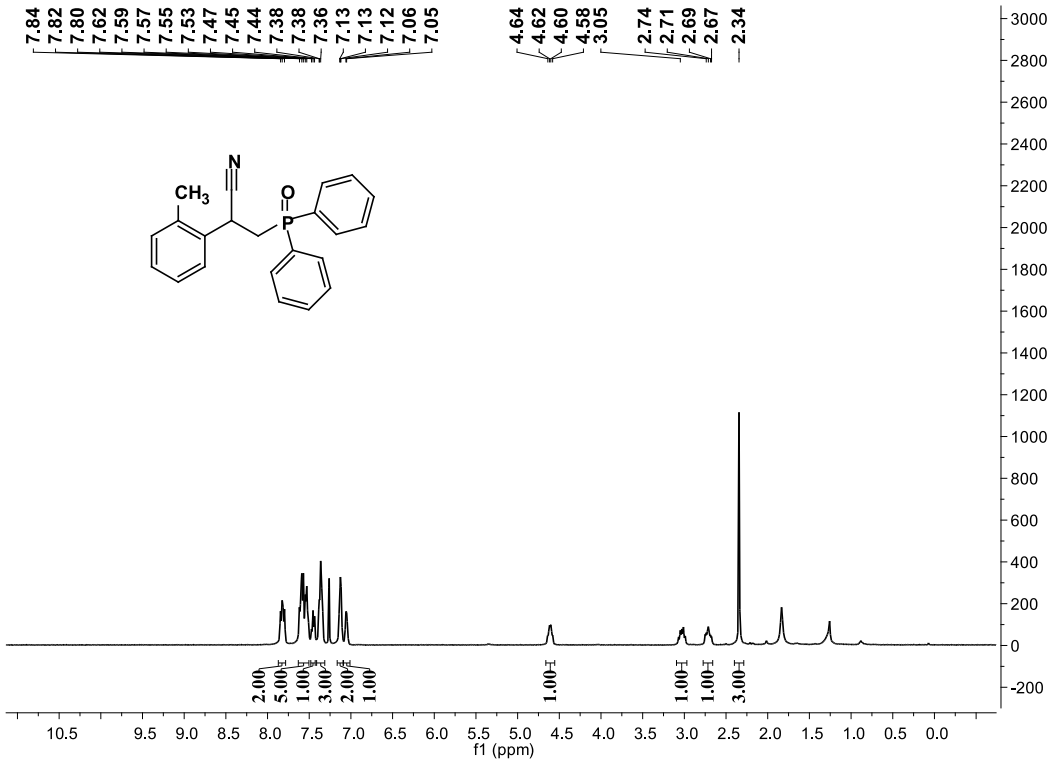
Compound 3i

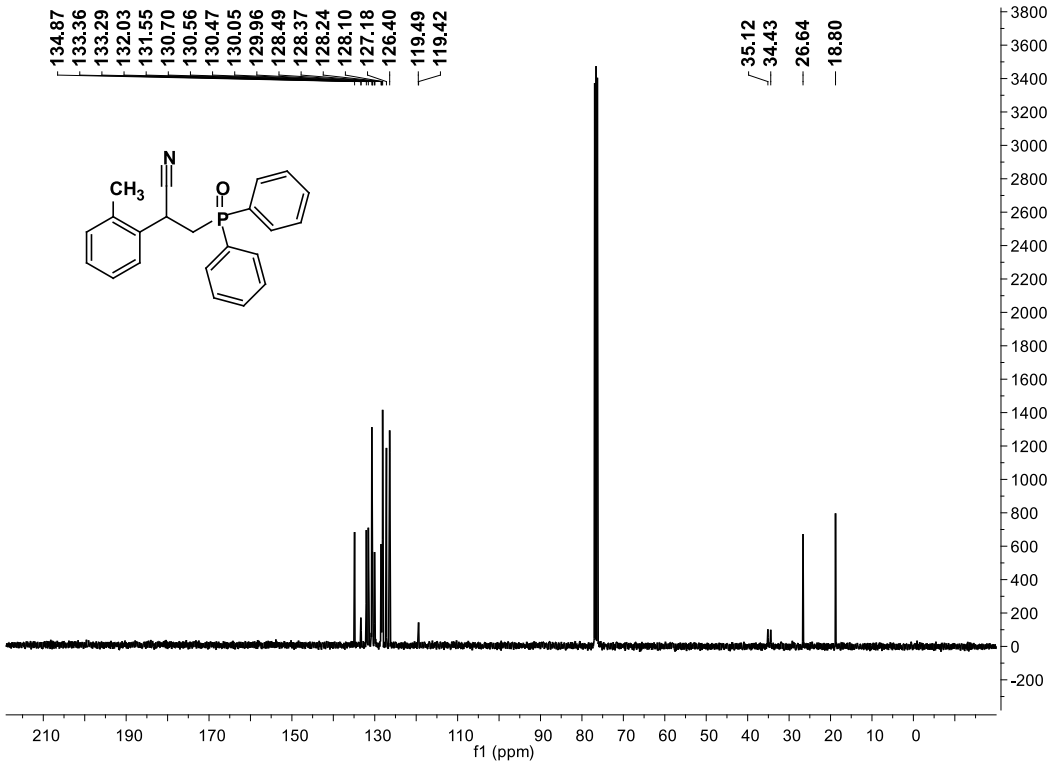


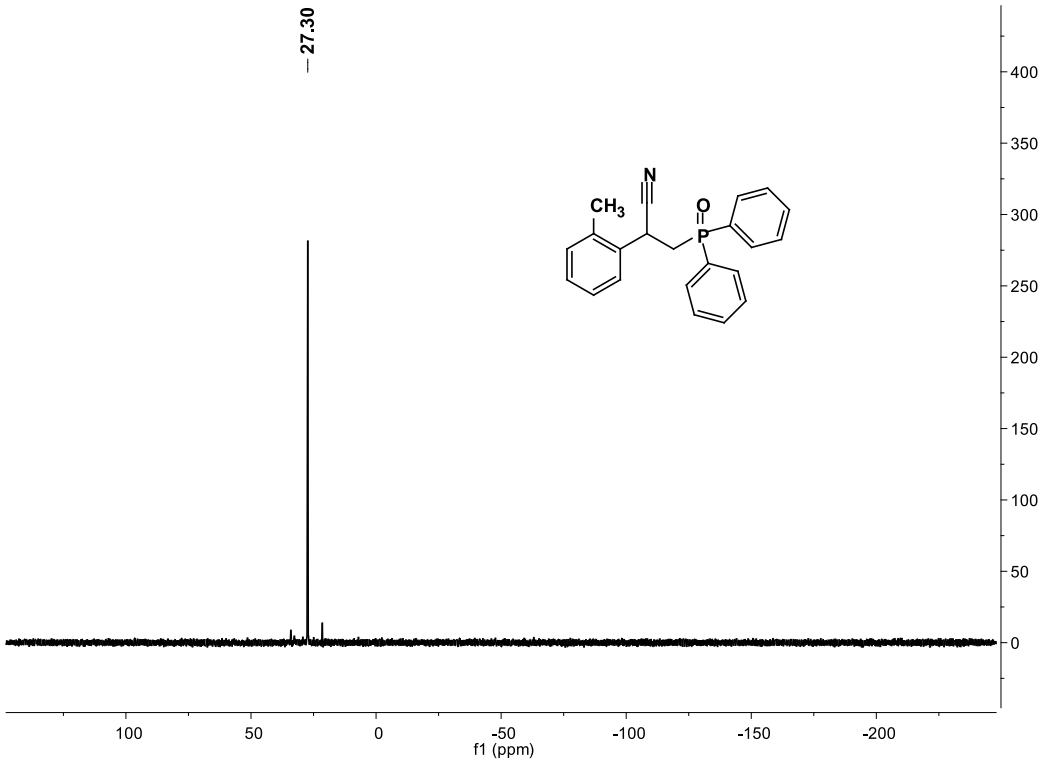



27.41
N
O
P
H3C
O
450
400
350
300
250
200
150
100
50
0
-50
100
50
0
-50
-100
-150
-200
f1 (ppm)

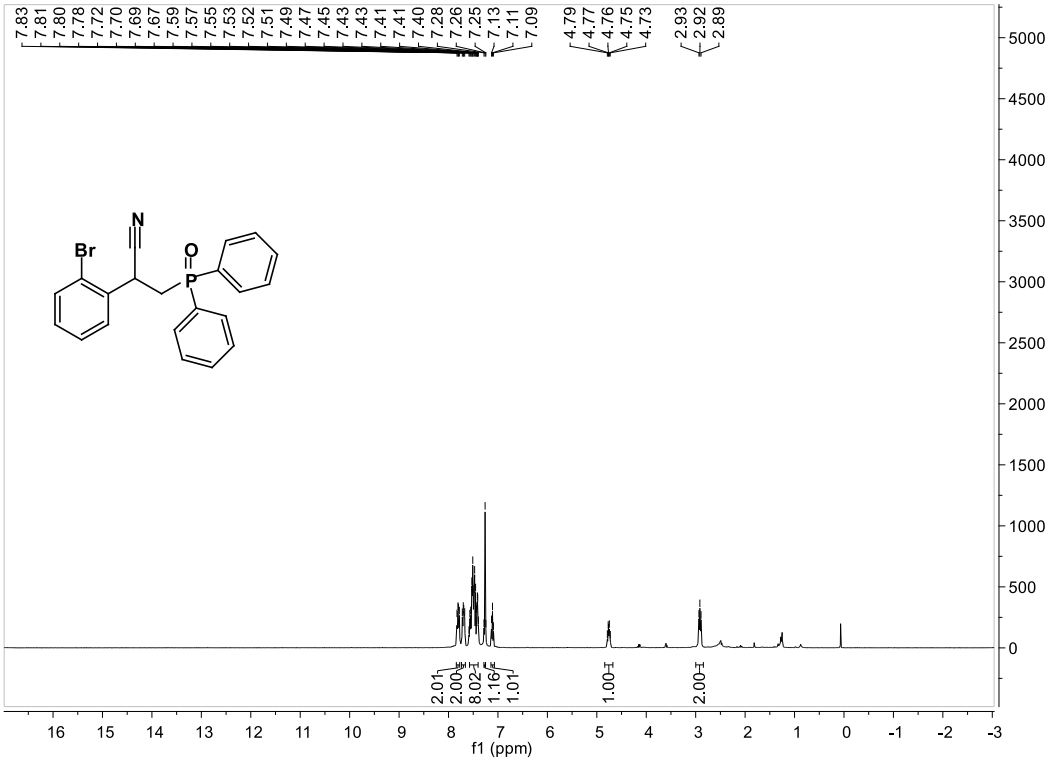
Compound 3j

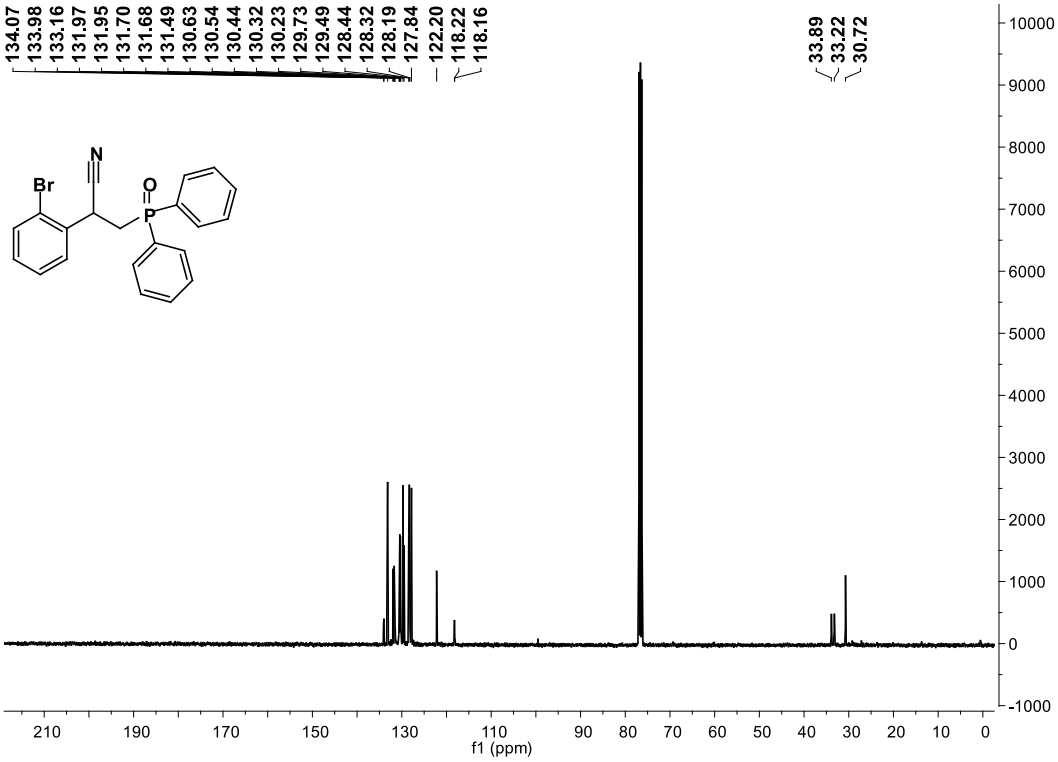

7.84
7.82
7.80
7.62
7.59
7.57
7.55
7.53
7.47
7.45
7.44
7.38
7.38
7.36
7.13
7.13
7.12
7.06
7.05
4.64
4.62
4.60
4.58
3.05
2.74
2.71
2.69
2.67
2.34
N
CH3
O
P
3000
2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
800
600
400
200
0
-200
2.00
5.00
1.00
3.00
2.00
1.00
1.00
1.00
1.00
3.00
10.5
9.5
9.0
8.5
8.0
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)

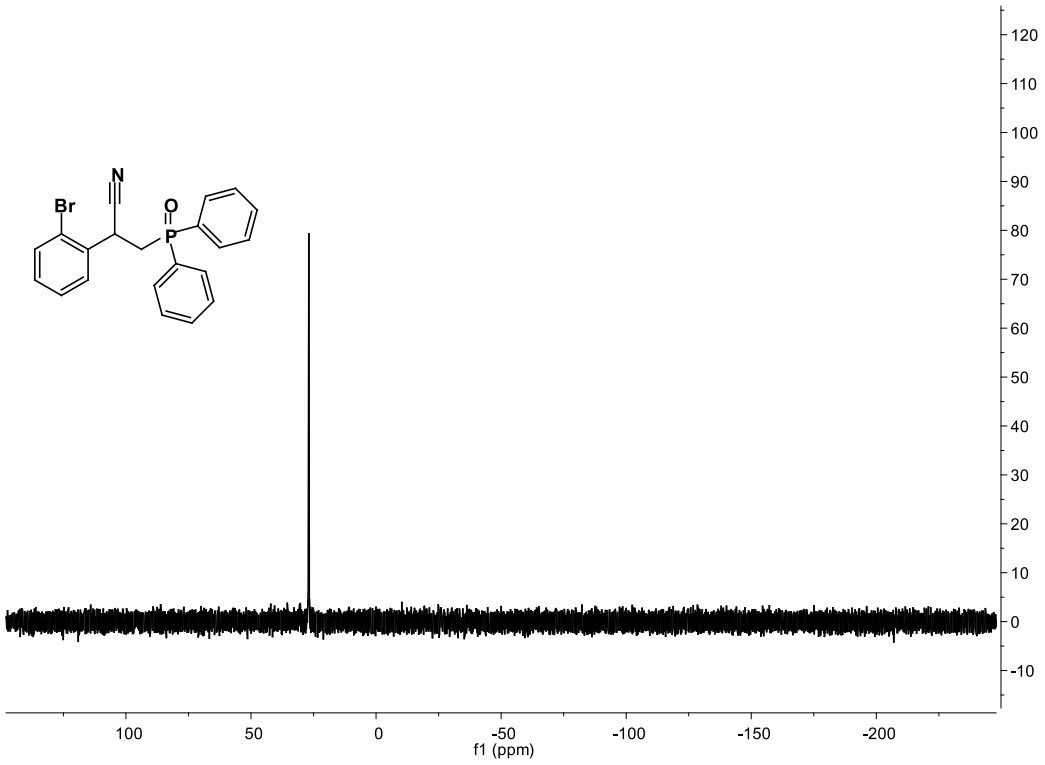

134.87
133.36
133.29
132.03
131.55
130.70
130.56
130.47
130.05
129.96
128.49
128.37
128.24
128.10
127.18
126.40
119.49
119.42
35.12
34.43
26.64
18.80
CH3
N
O
P
f1 (ppm)


27.30
CH3
N
O
P
f1 (ppm)

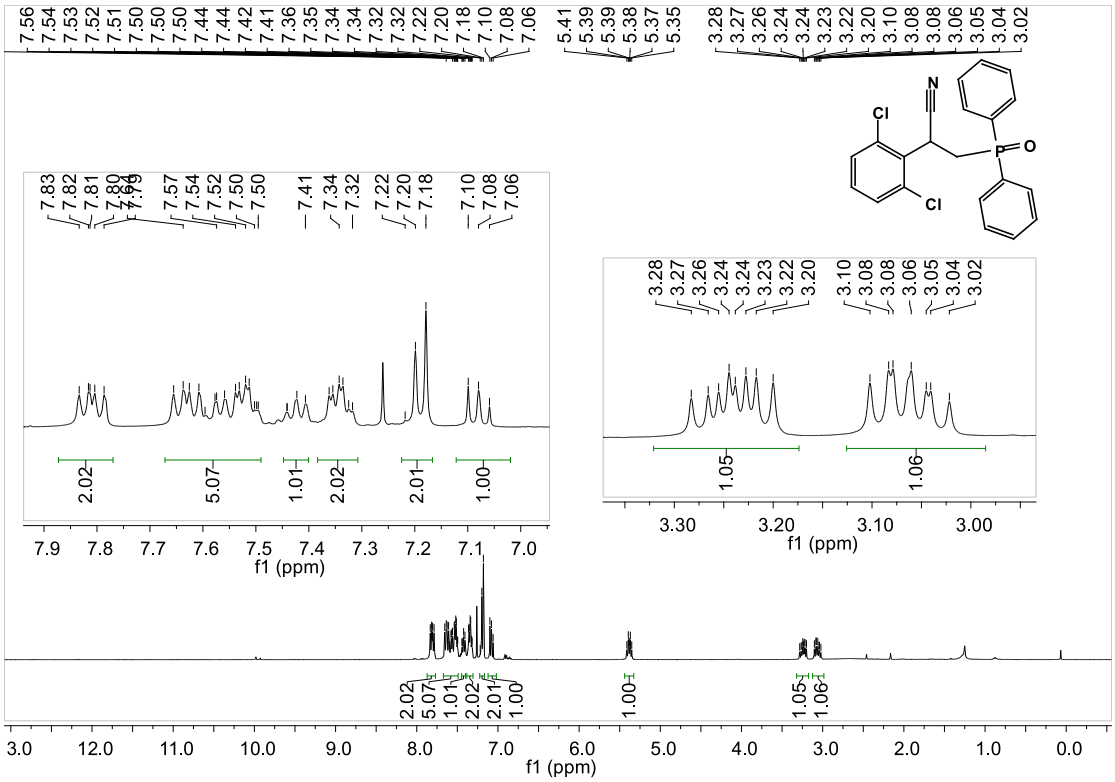
Compound 3k

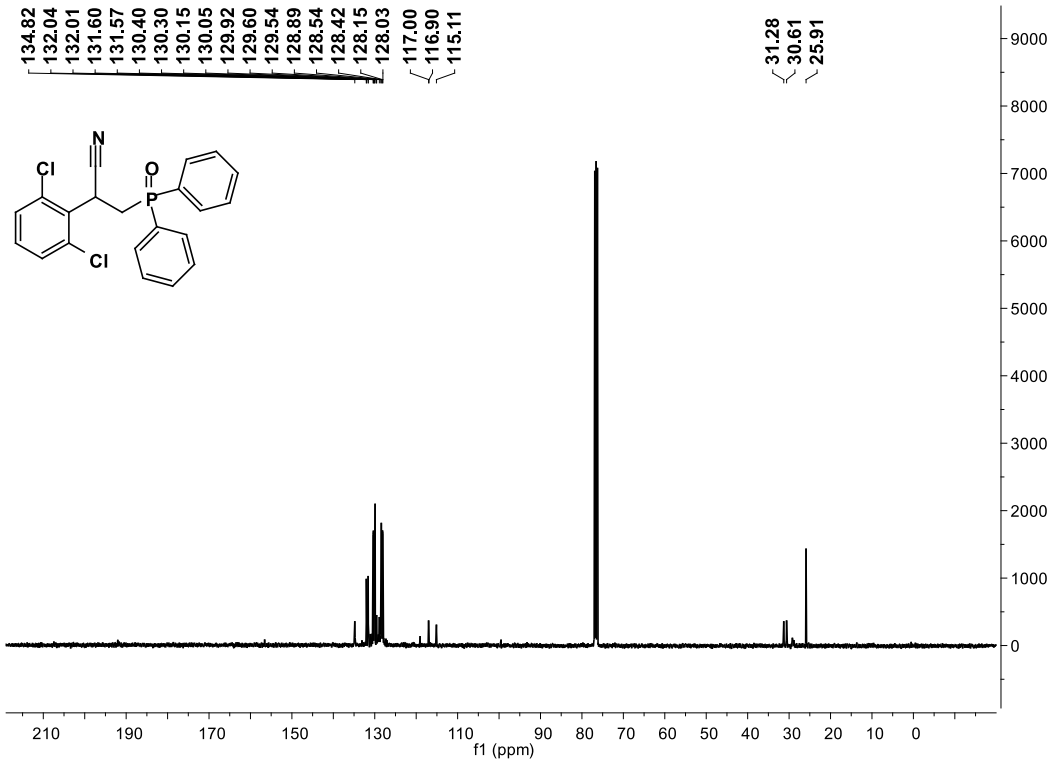

7.83
7.81
7.80
7.78
7.72
7.70
7.69
7.67
7.59
7.57
7.55
7.53
7.52
7.51
7.49
7.47
7.45
7.43
7.43
7.41
7.41
7.40
7.28
7.26
7.25
7.13
7.11
7.09
4.79
4.77
4.76
4.75
4.73
2.93
2.92
2.89
N
Br
O
P
2.01
2.00
8.02
1.16
1.01
1.00
2.00
f1 (ppm)

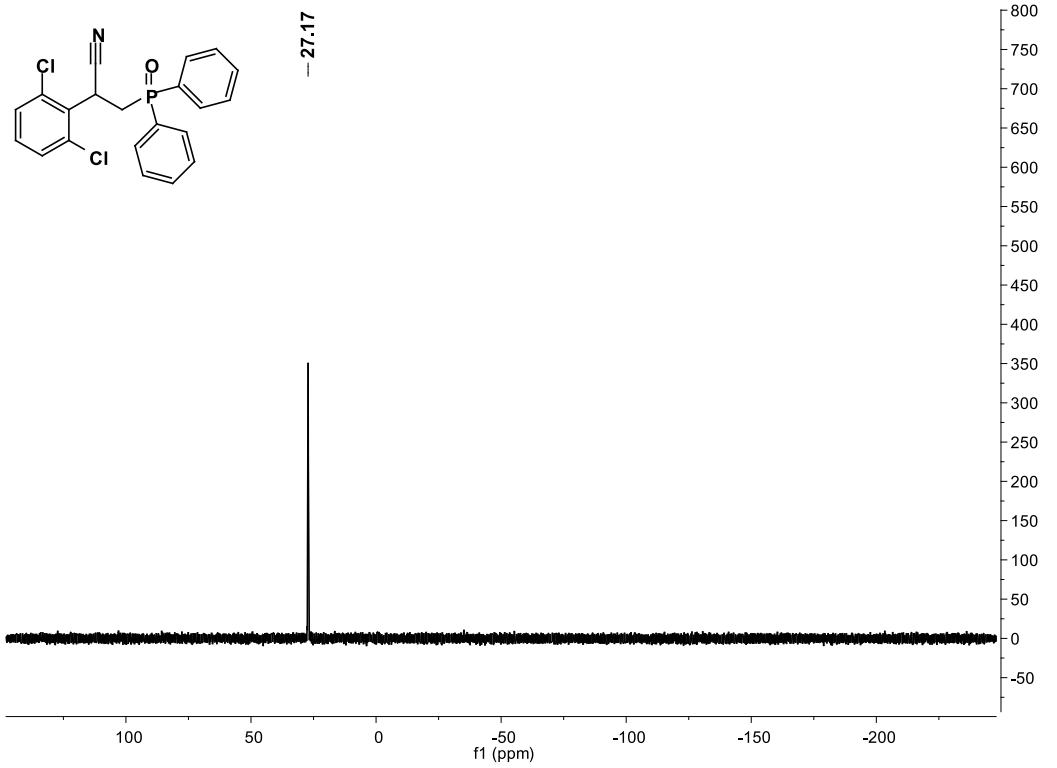

134.07
133.98
133.16
131.97
131.95
131.70
131.68
131.49
130.63
130.54
130.44
130.32
130.23
129.73
129.49
128.44
128.32
128.19
127.84
122.20
118.22
118.16
33.89
33.22
30.72
N
Br
O
P
f1 (ppm)


Br
N
O
P
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
100
50
0
-50
-100
-150
-200
f1 (ppm)

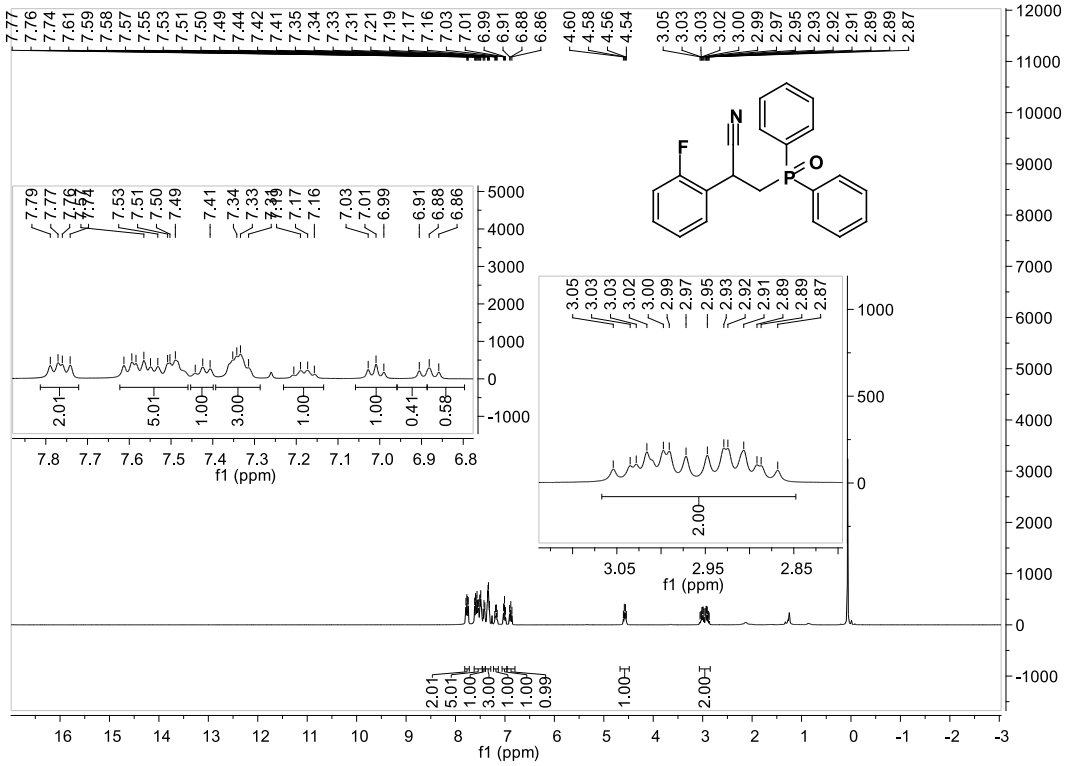
Compound 3l

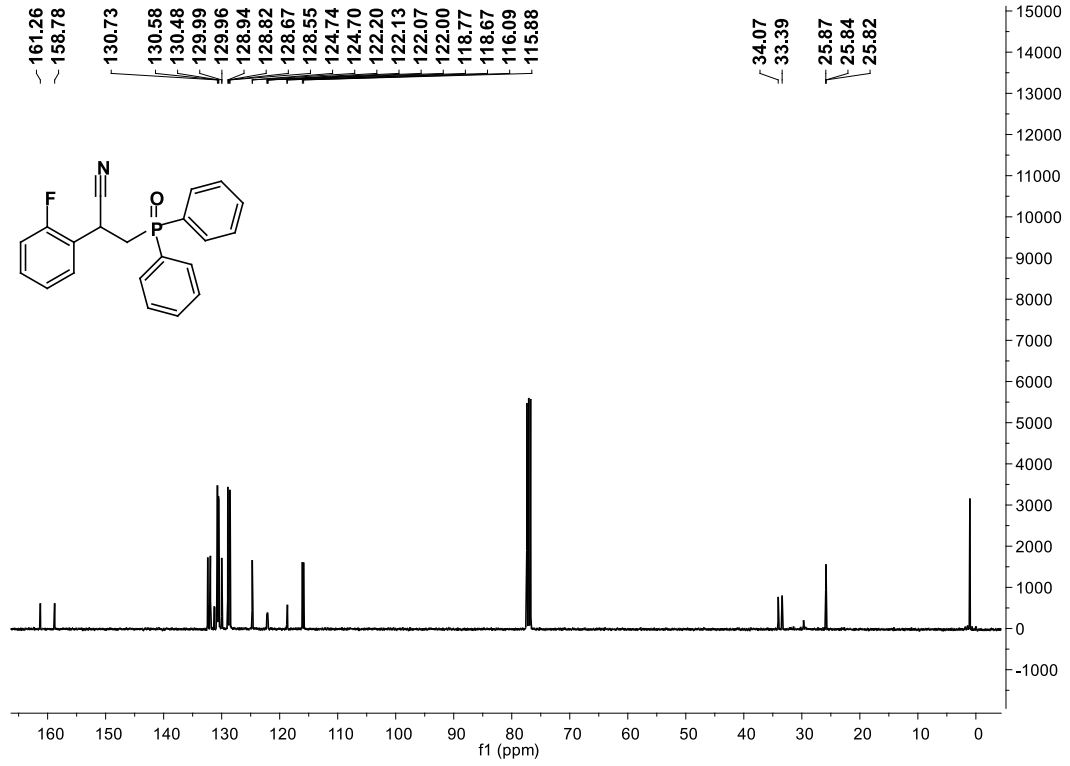

Cl
N
P
O
Cl
f1 (ppm)

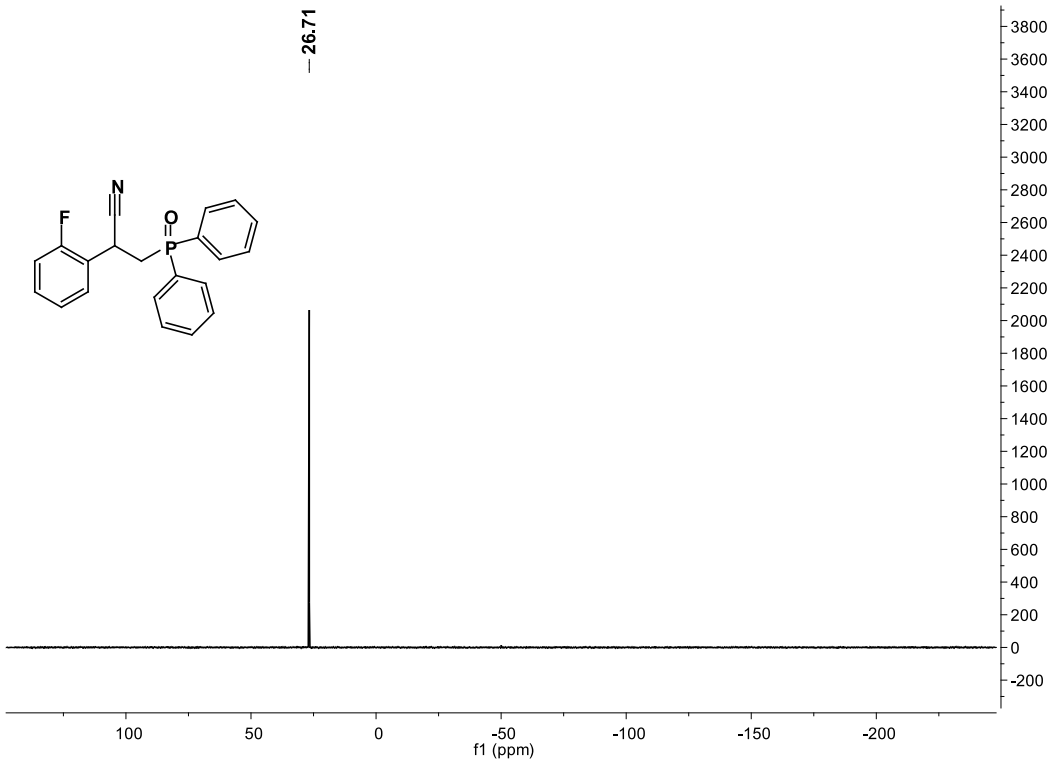

134.82
132.04
132.01
131.60
131.57
130.40
130.30
130.15
130.05
129.92
129.60
129.54
128.89
128.54
128.42
128.15
128.03
117.00
116.90
115.11
31.28
30.61
25.91
N
Cl
O
P
Cl
f1 (ppm)


27.17
N
Cl
O
P
Cl
f1 (ppm)

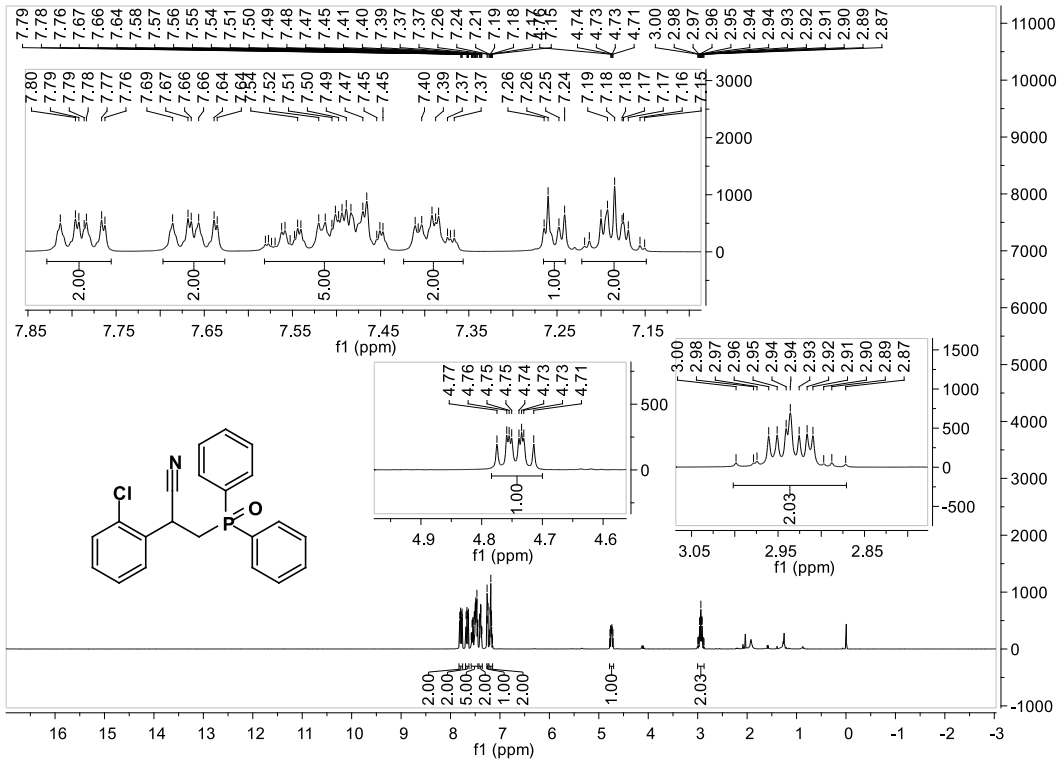
Compound 3m

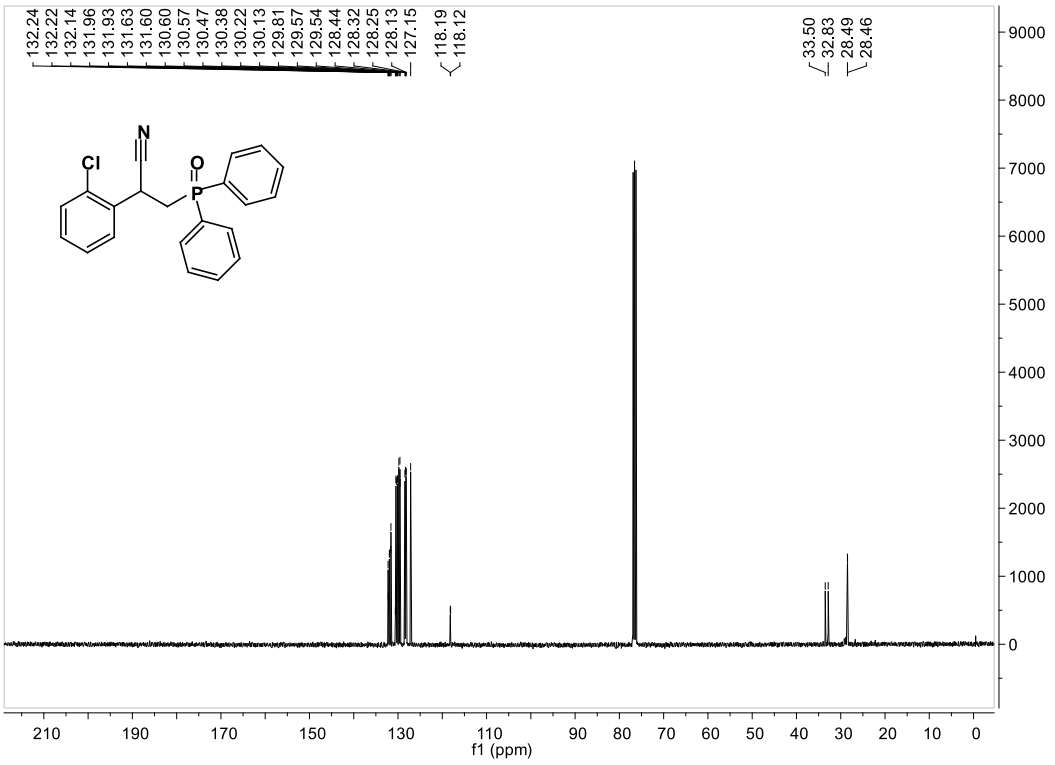

7.77 7.76 7.74 7.61 7.59 7.58 7.57 7.55 7.53 7.51 7.50 7.49 7.44 7.42 7.41 7.35 7.34 7.33 7.31 7.21 7.19 7.17 7.16 7.03 7.01 6.99 6.91 6.88 6.86
4.60 4.58 4.56 4.54
3.05 3.03 3.03 3.02 3.00 2.99 2.97 2.95 2.93 2.92 2.91 2.89 2.89 2.87
N
F
O
P
f1 (ppm)
2.01 5.01 1.00 3.00 1.00 1.00 0.41 0.58
2.00
1.00 2.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3

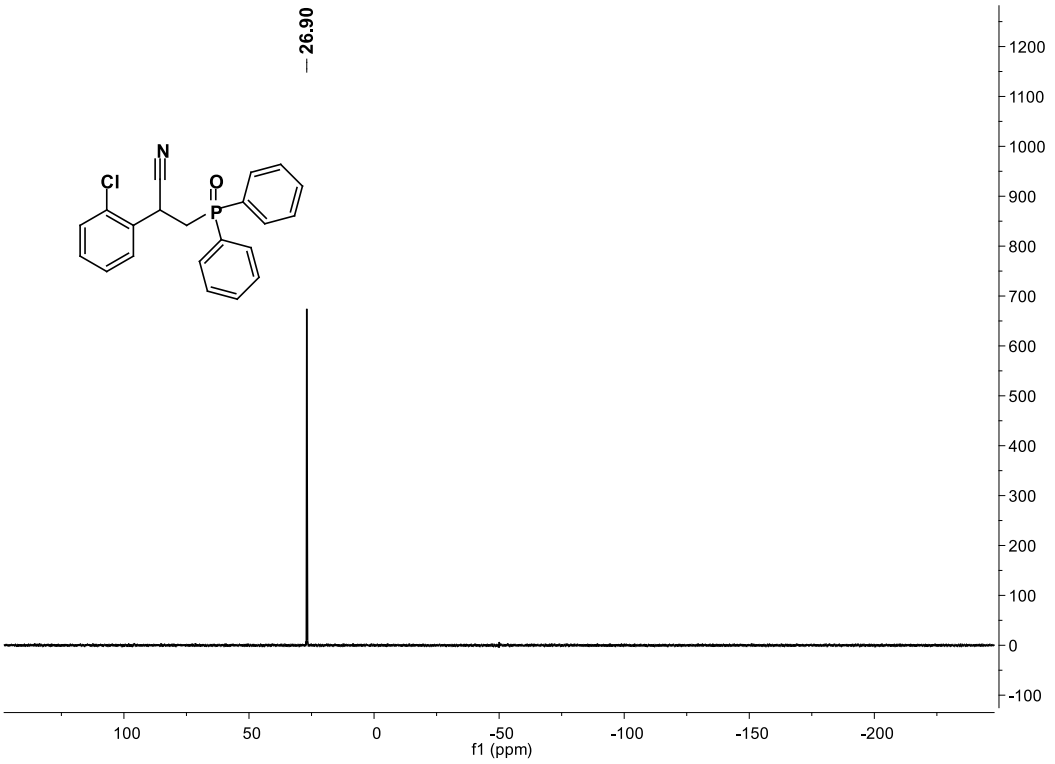

161.26 158.78 130.73 130.58 130.48 129.99 129.96 128.94 128.82 128.67 128.55 124.74 124.70 122.20 122.13 122.07 122.00 118.77 118.67 116.09 115.88
34.07 33.39 25.87 25.84 25.82
N
F
O
P
f1 (ppm)
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0



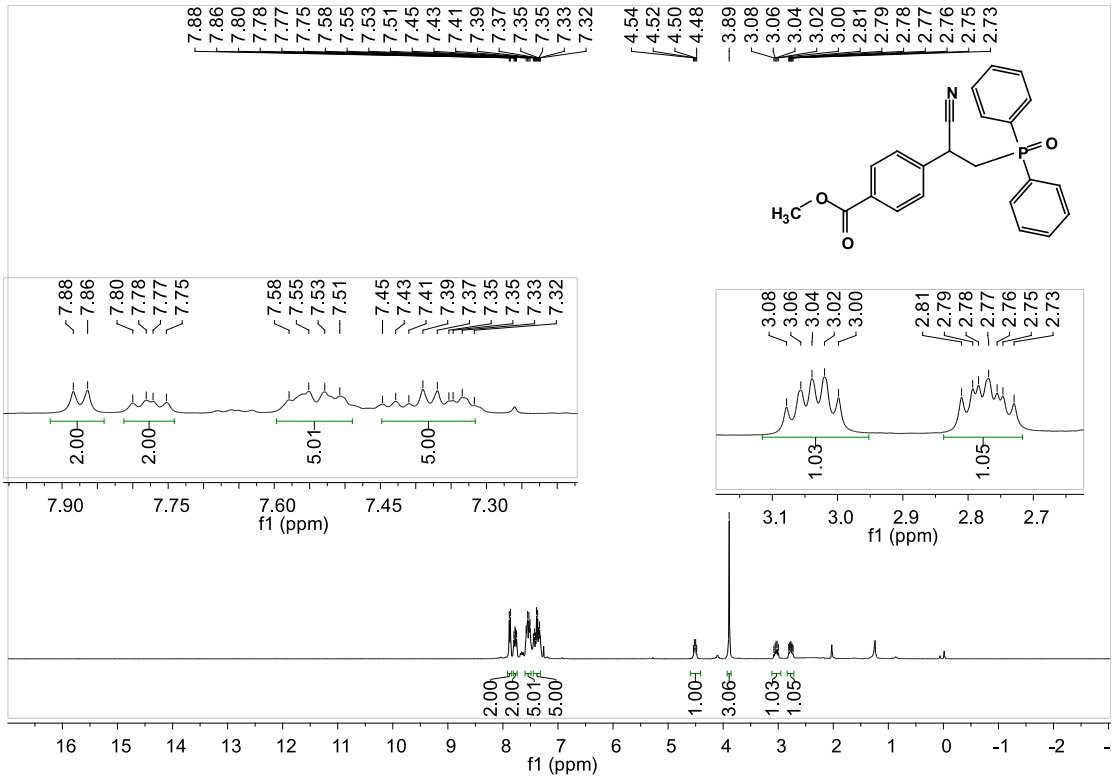
Compound 3n

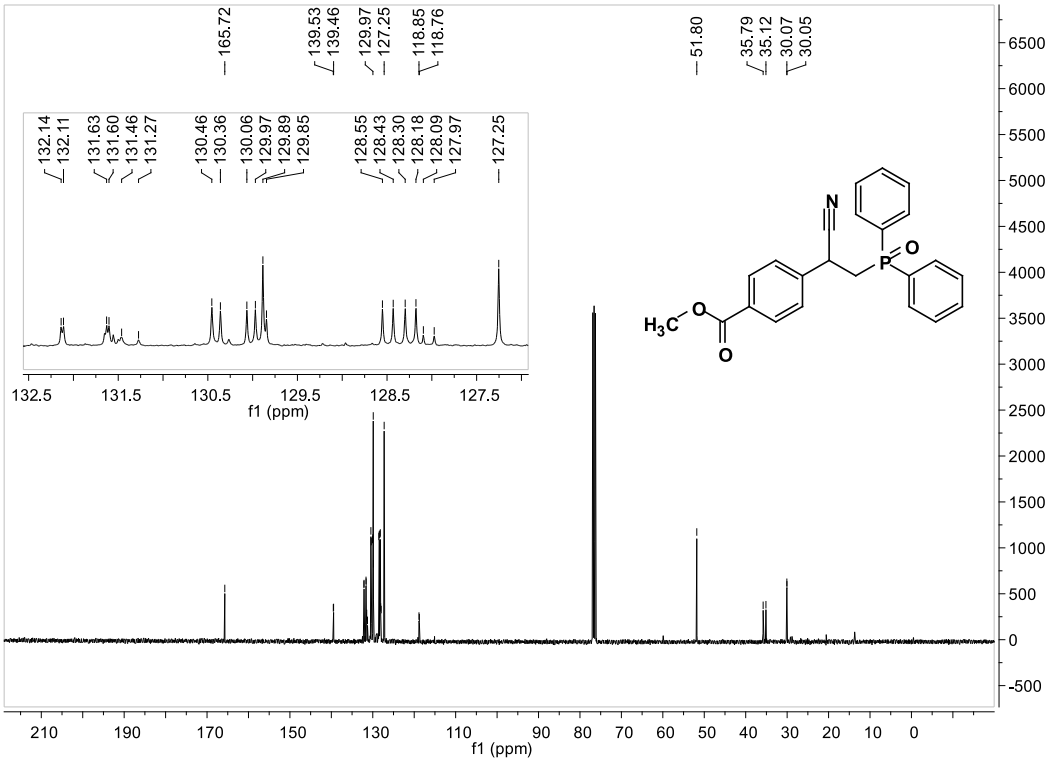


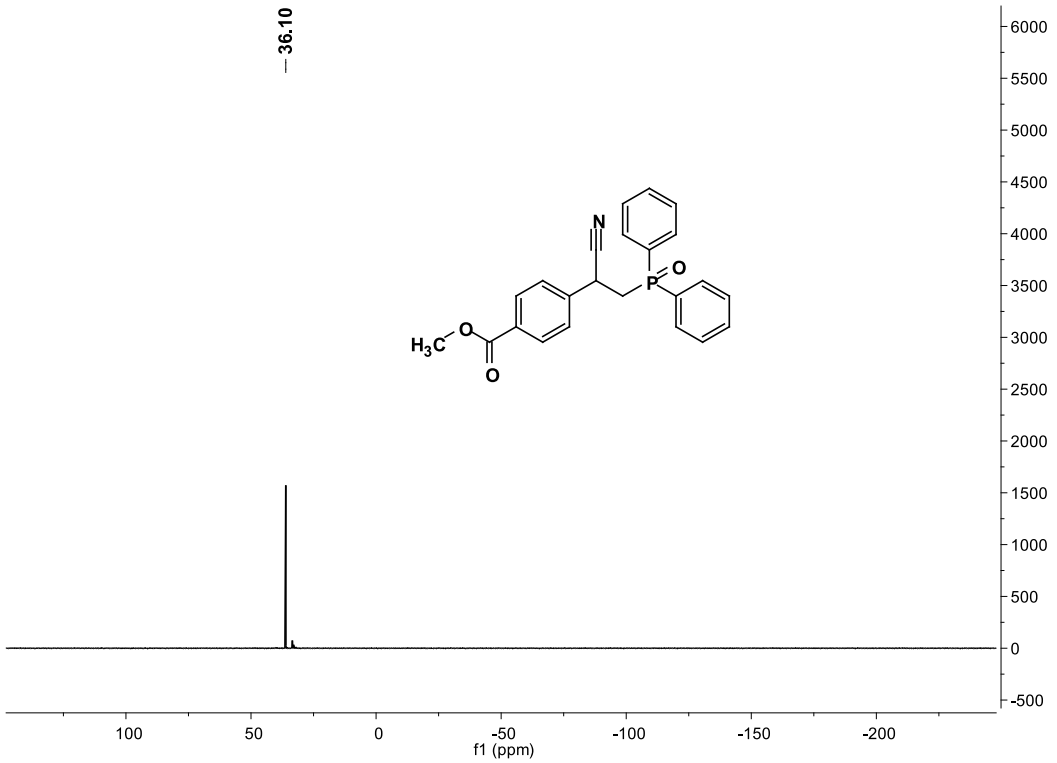

132.24
132.22
132.14
131.96
131.93
131.63
131.60
130.60
130.57
130.47
130.38
130.22
130.13
129.81
129.57
129.54
128.44
128.32
128.25
128.13
127.15
118.19
118.12
33.50
32.83
28.49
28.46
N
Cl
O
P
f1 (ppm)


26.90
N
Cl
O
P
f1 (ppm)

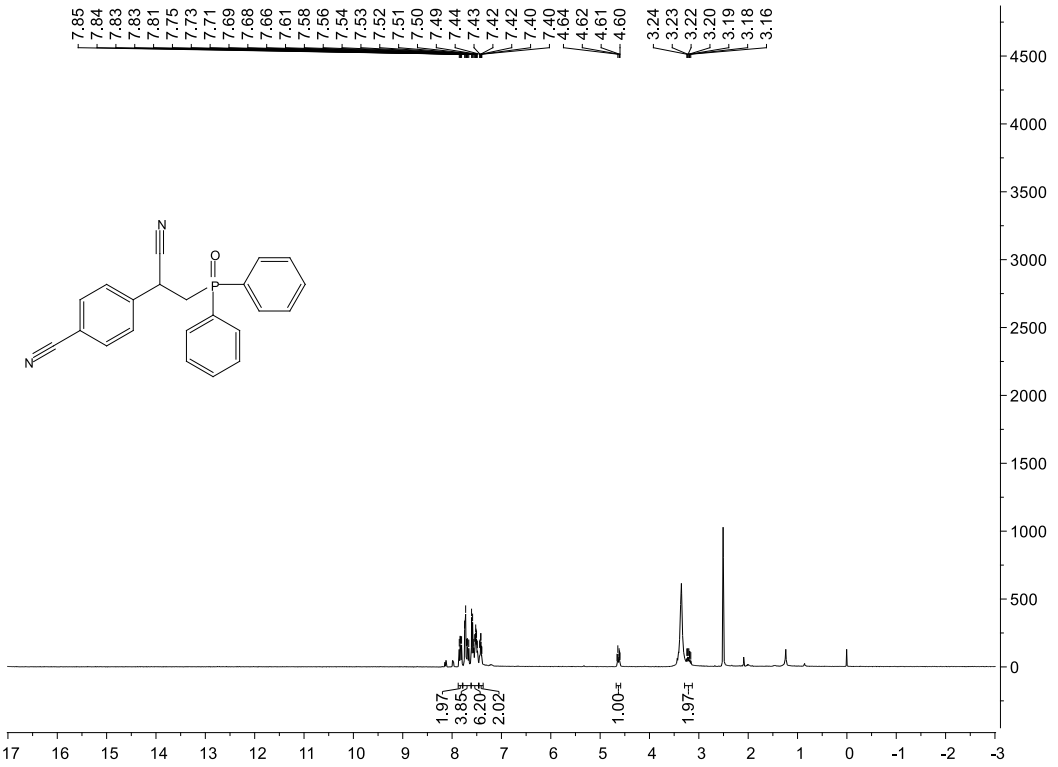
Compound 3o

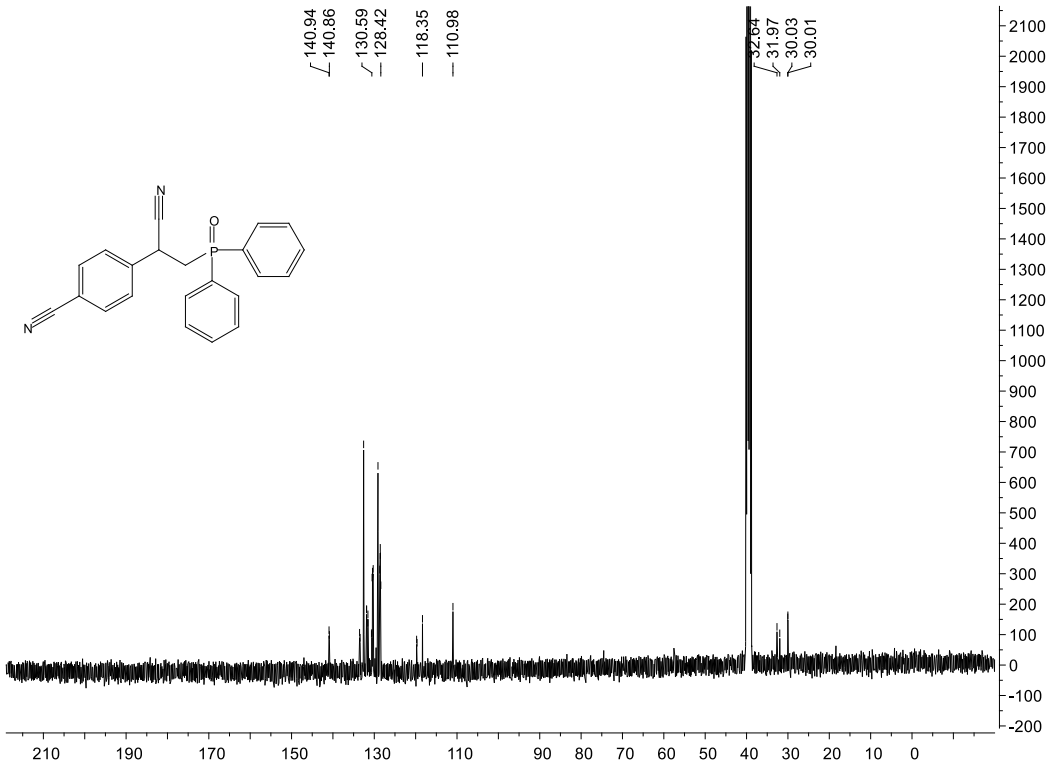

7.88 7.86 7.80 7.78 7.77 7.75 7.58 7.55 7.53 7.51 7.45 7.43 7.41 7.39 7.37 7.35 7.35 7.33 7.32
4.54 4.52 4.50 4.48
3.89
3.08 3.06 3.04 3.02 3.00 2.81 2.79 2.78 2.77 2.76 2.75 2.73
N
P
O
H3C
O
O
2.00 2.00 5.01 5.00
7.90 7.75 7.60 7.45 7.30
f1 (ppm)
1.03 1.05
3.1 3.0 2.9 2.8 2.7
f1 (ppm)
2.00 2.00 5.01 5.00 1.00 3.06 1.03 1.05
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2
f1 (ppm)

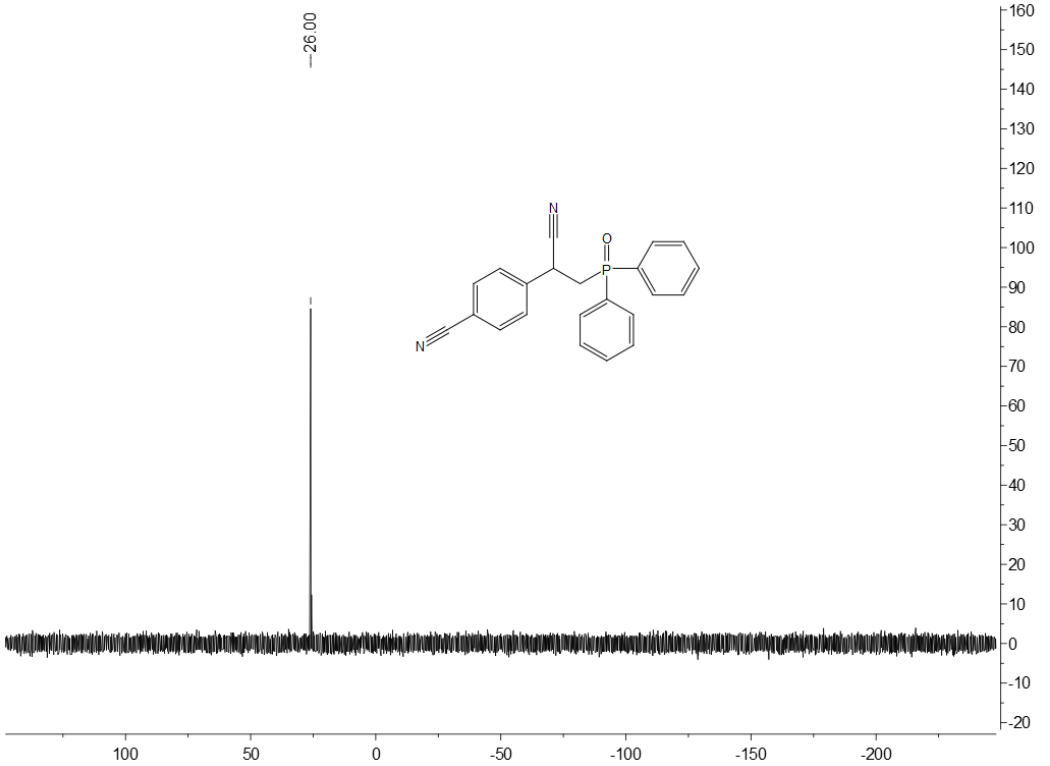

165.72
139.53 139.46
129.97 127.25
118.85 118.76
51.80
35.79 35.12 30.07 30.05
132.14 132.11 131.63 131.60 131.46 131.27 130.46 130.36 130.06 128.97 129.89 129.85 128.55 128.43 128.30 128.18 128.09 127.97 127.25
132.5 131.5 130.5 129.5 128.5 127.5
f1 (ppm)
N
P
O
H3C
O
O
6500 6000 5500 5000 4500 4000 3500 3000 2500 2000 1500 1000 500 0 -500
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


36.10
N
O
P
O
H3C
O
O
6000
5500
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
-500
100
50
0
-50
-100
-150
-200
f1 (ppm)

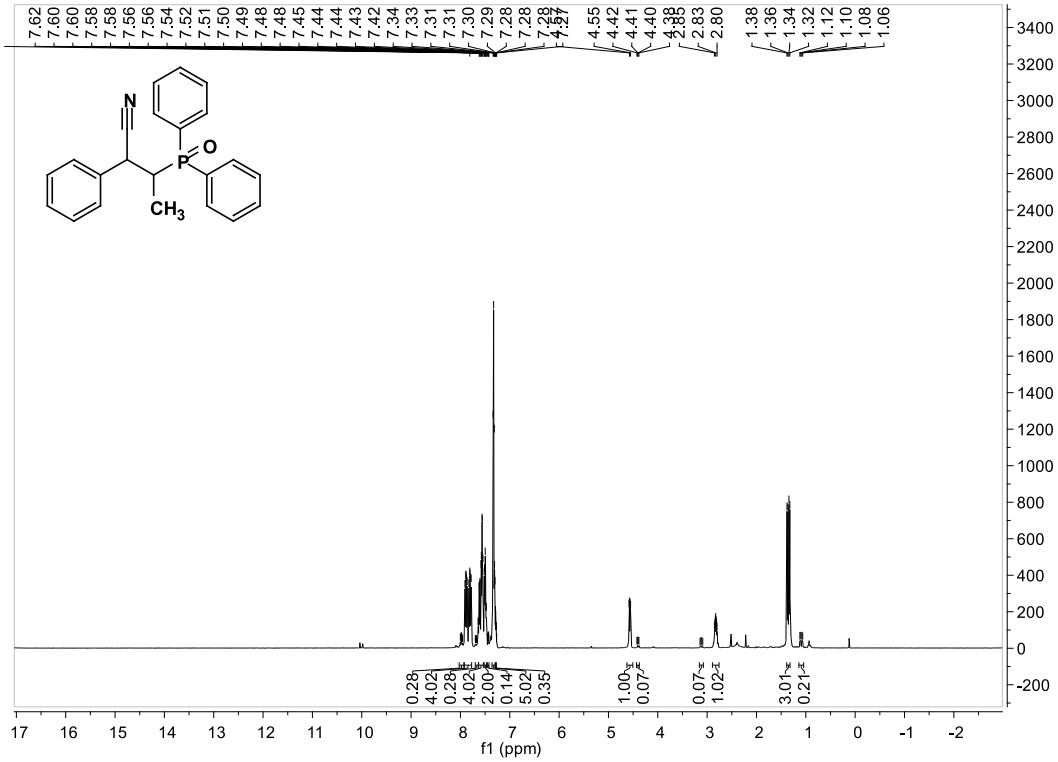
Compound 3p

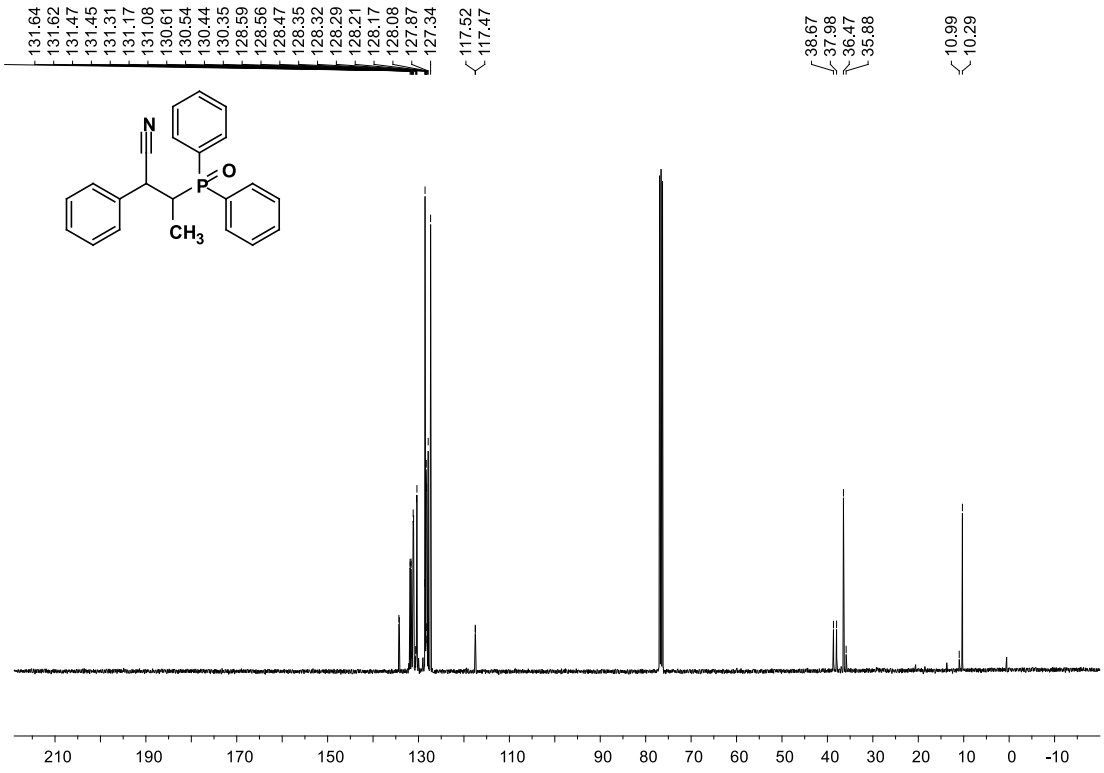

7.85
7.84
7.83
7.83
7.81
7.75
7.73
7.71
7.69
7.68
7.66
7.61
7.58
7.56
7.54
7.53
7.52
7.51
7.50
7.49
7.44
7.43
7.42
7.42
7.40
7.40
4.64
4.62
4.61
4.60
3.24
3.23
3.22
3.20
3.19
3.18
3.16
N
O
P
N
4500
4000
3500
3000
2500
2000
1500
1000
500
0
1.97
3.85
6.20
2.02
1.00
1.97
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3

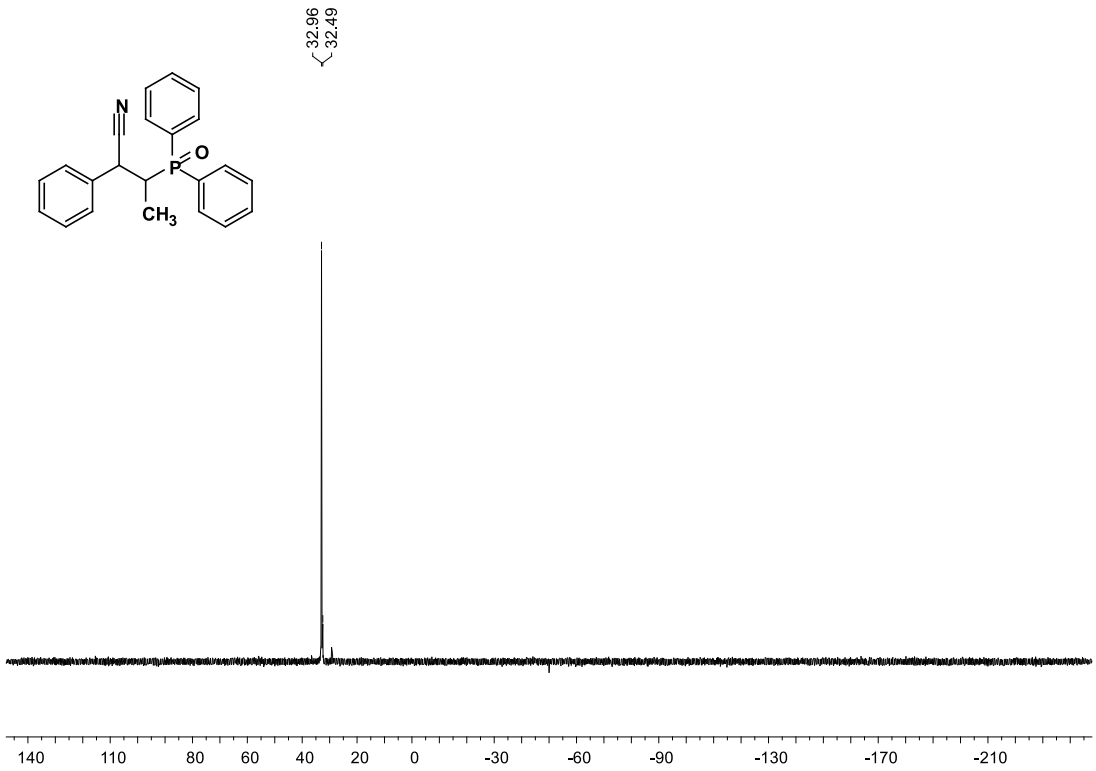

140.94
140.86
130.59
128.42
118.35
110.98
32.64
31.97
30.03
30.01
N
O
P
N


26.00
N
O
P
N

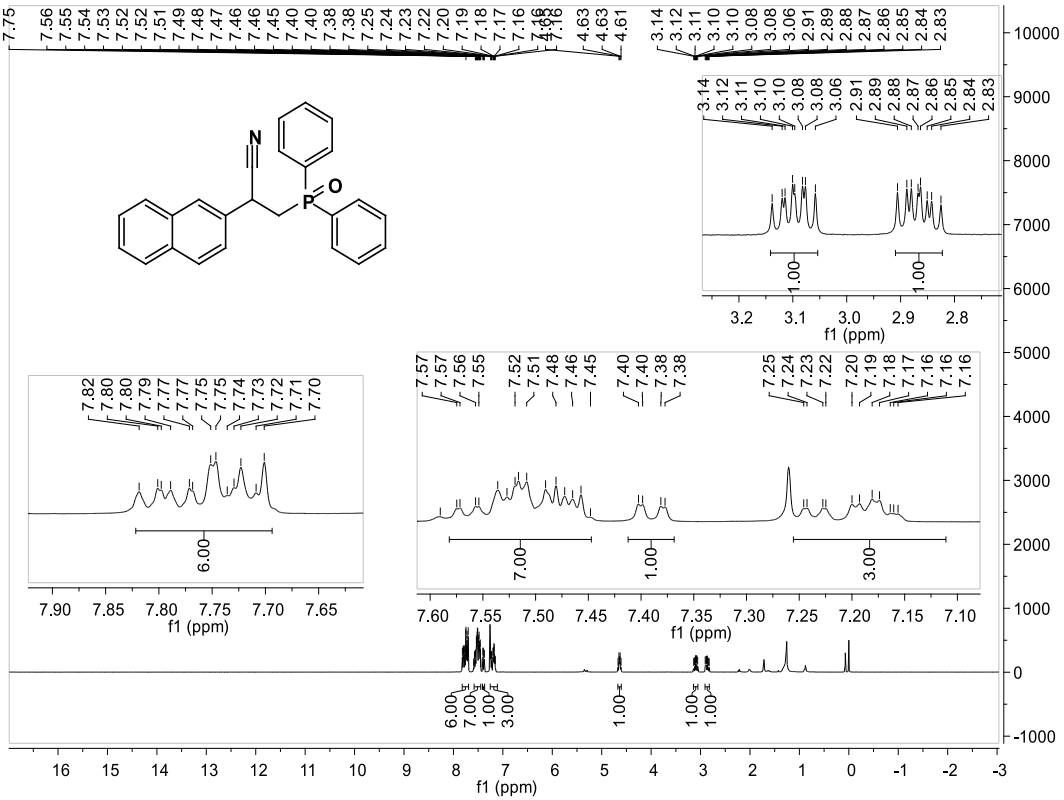
Compound 3q

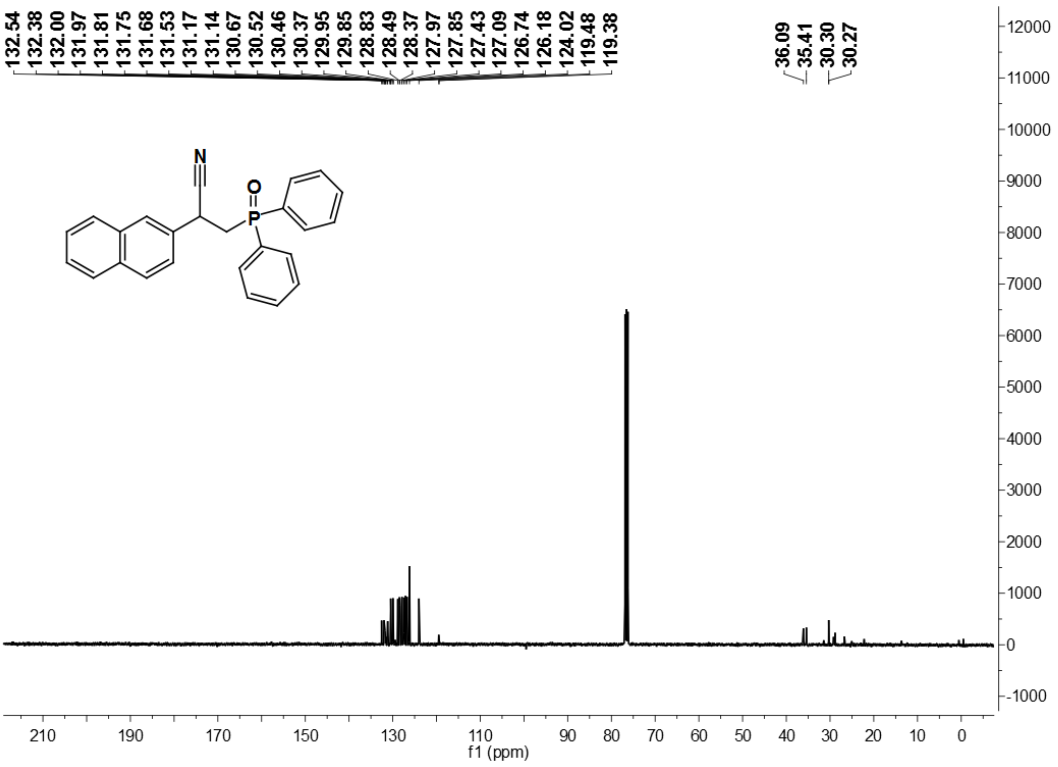

7.62 7.60 7.60 7.58 7.58 7.56 7.56 7.54 7.52 7.51 7.50 7.49 7.48 7.48 7.45 7.44 7.44 7.43 7.42 7.34 7.33 7.31 7.31 7.30 7.29 7.28 7.28 7.28 4.55 4.42 4.41 4.40 2.85 2.83 2.80 1.38 1.36 1.34 1.32 1.12 1.10 1.08 1.06
N
O
P
CH3
0.28 4.02 0.28 4.02 2.00 0.14 5.02 0.35 1.00 0.07 0.07 1.02 3.01 0.21
f1 (ppm)

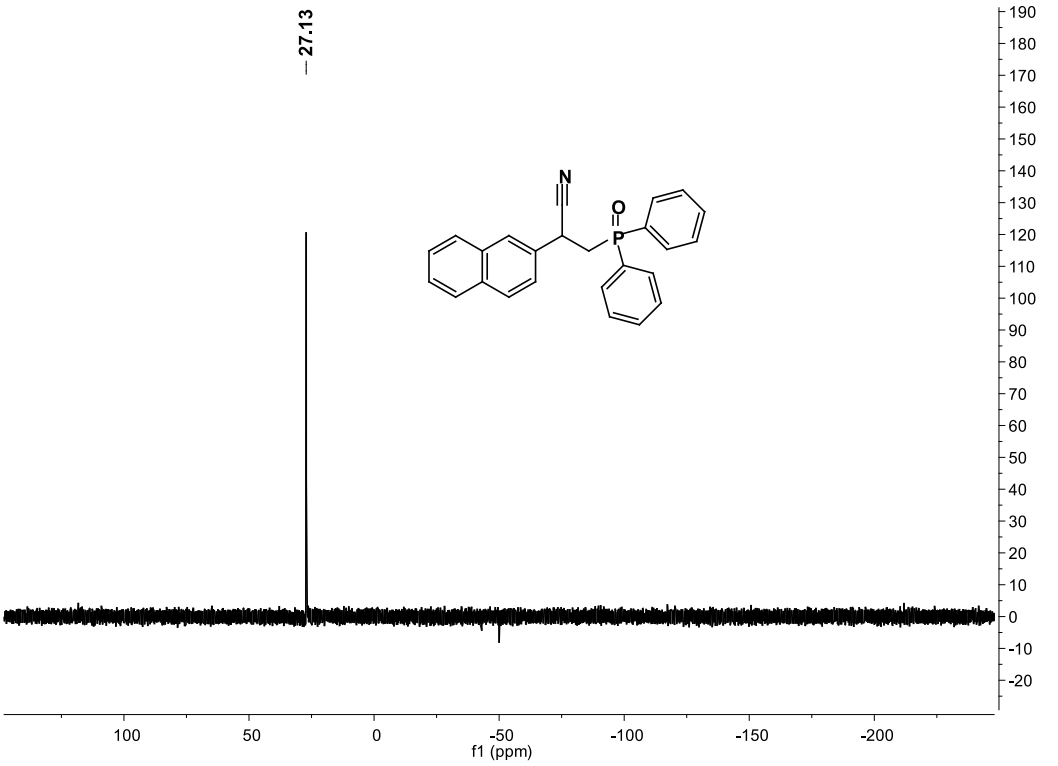

131.64 131.62 131.47 131.45 131.31 131.17 131.08 130.61 130.54 130.44 130.35 128.59 128.56 128.47 128.35 128.32 128.29 128.21 128.17 128.08 127.87 127.34 117.52 117.47 38.67 37.98 36.47 35.88 10.99 10.29
N
O
P
CH3


32.96
32.49
N
O
P
CH3
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210

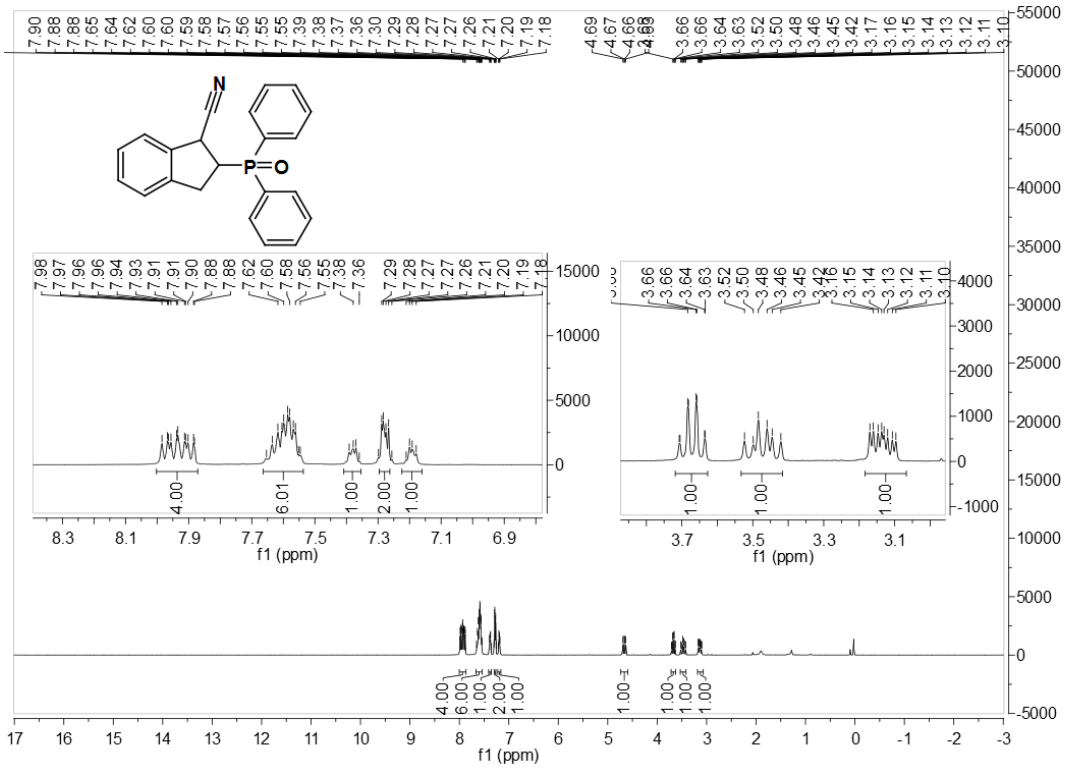
Compound 3r

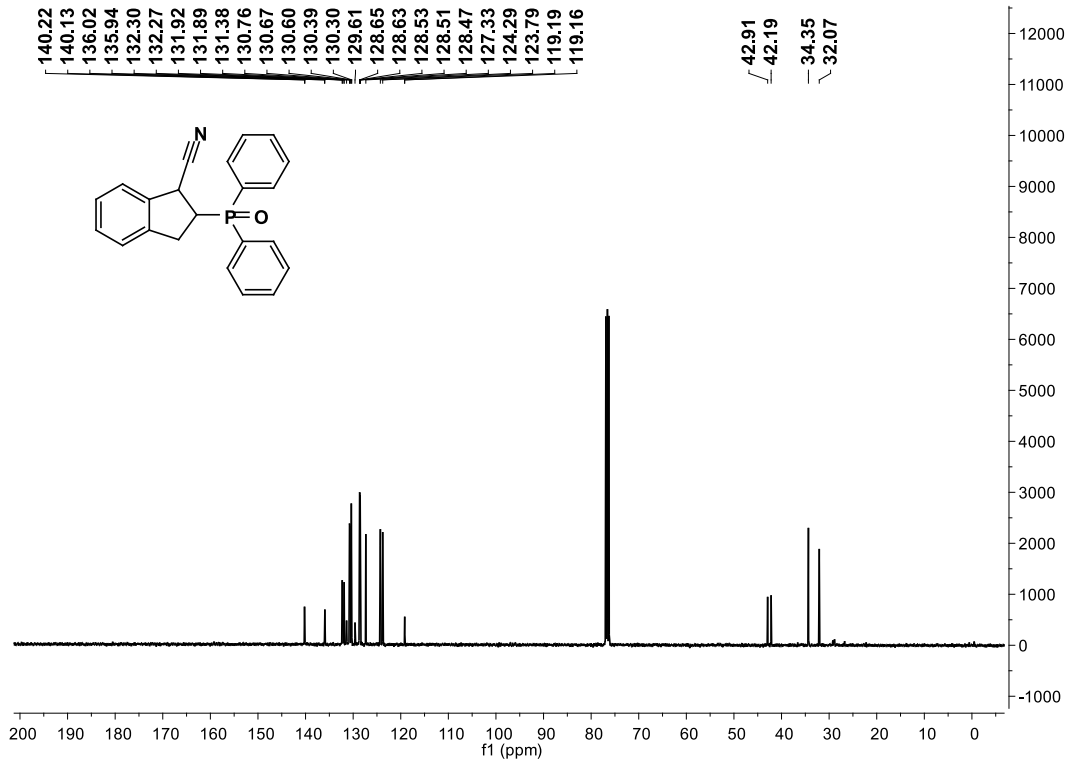

N
O
P
f1 (ppm)
6.00
7.00
1.00
3.00
1.00
1.00
1.00

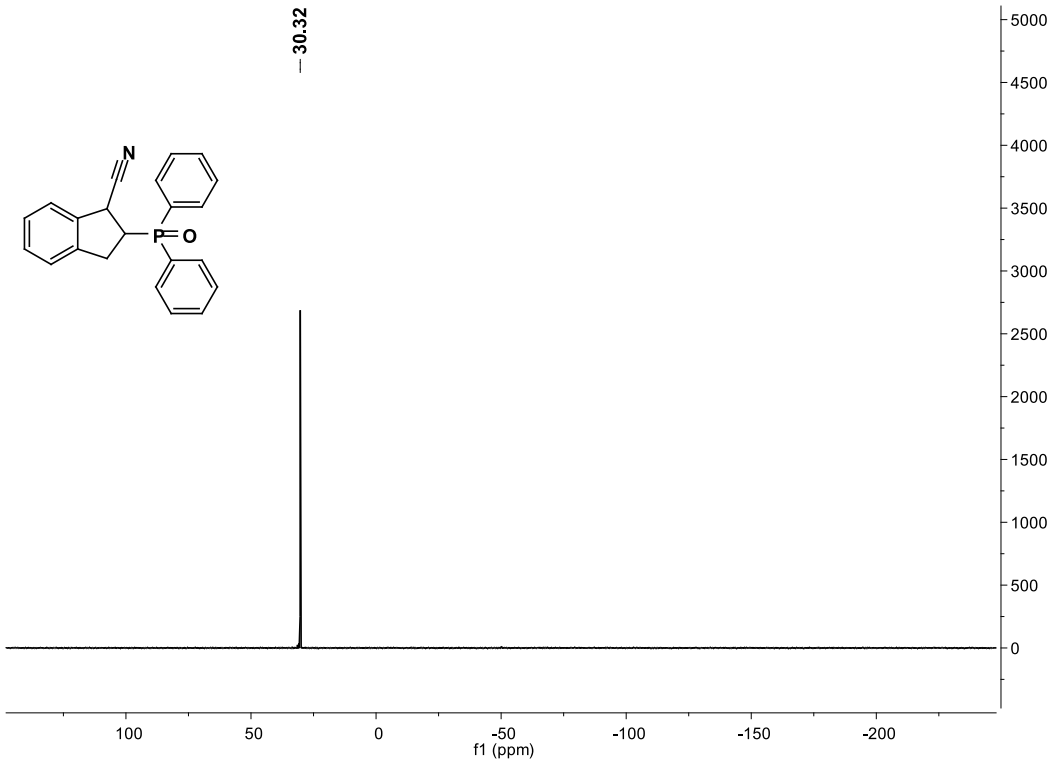

132.54
132.38
132.00
131.97
131.81
131.75
131.68
131.53
131.17
131.14
130.67
130.52
130.46
130.37
129.95
129.85
128.83
128.49
128.37
127.97
127.85
127.43
127.09
126.74
126.18
124.02
119.48
119.38
36.09
35.41
30.30
30.27
N
O
P
f1 (ppm)


27.13
N
O
P
f1 (ppm)

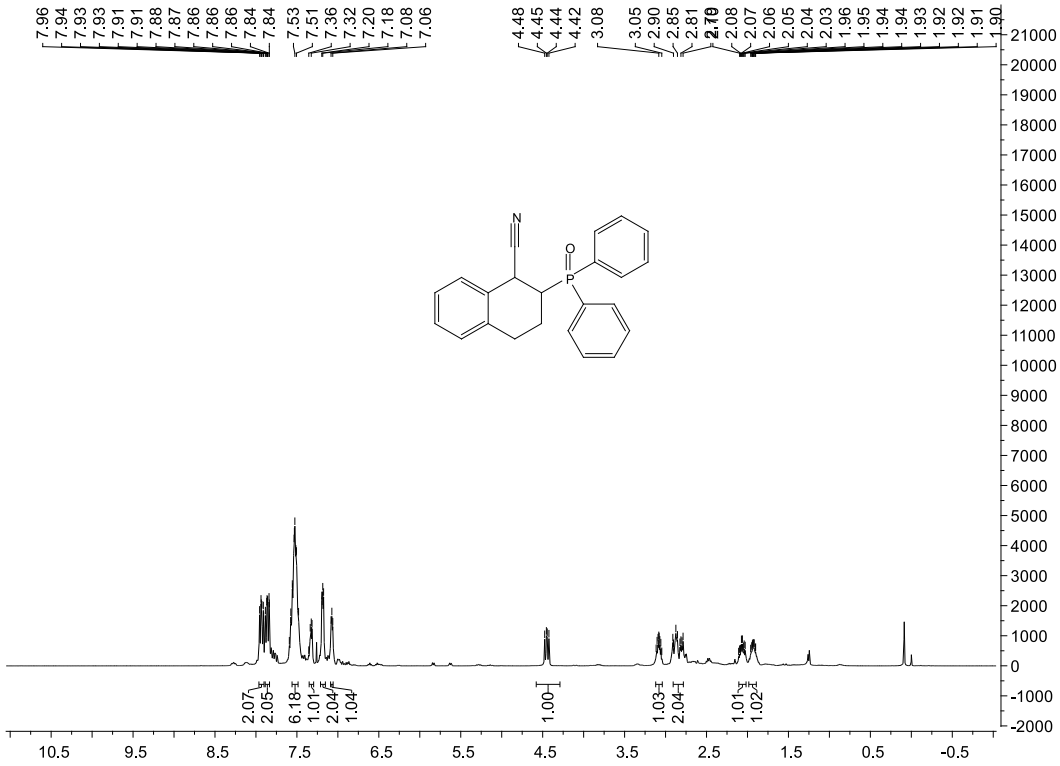
Compound 3s

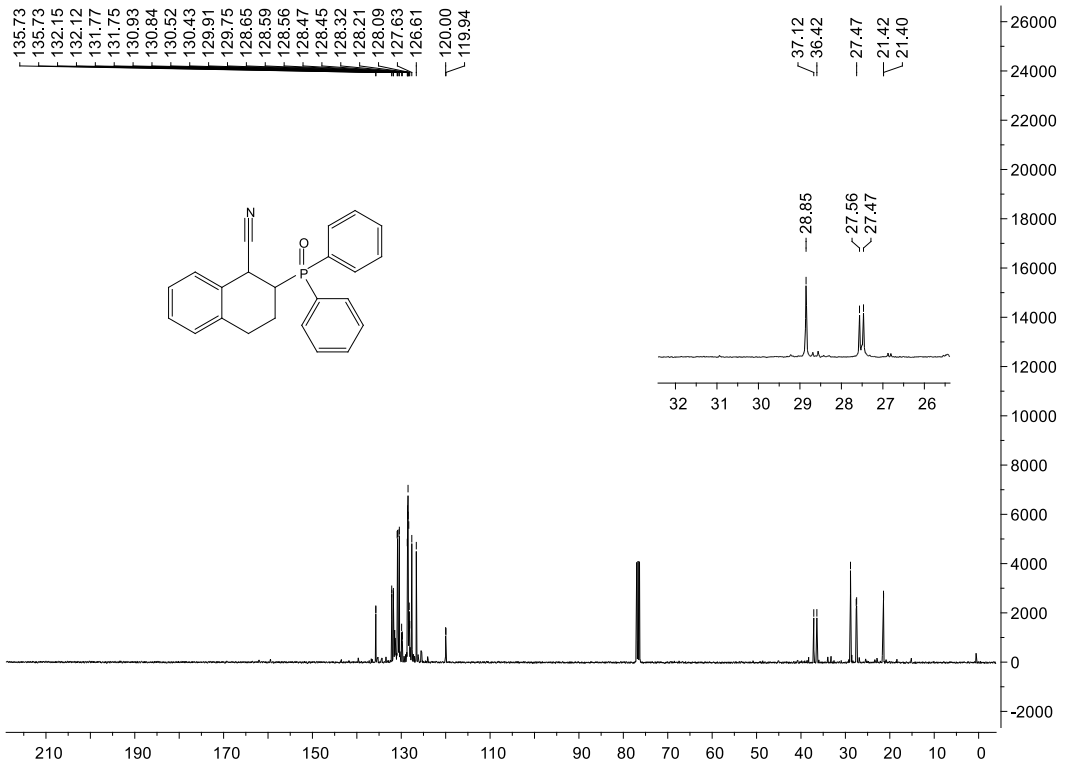

N
P=O
f1 (ppm)

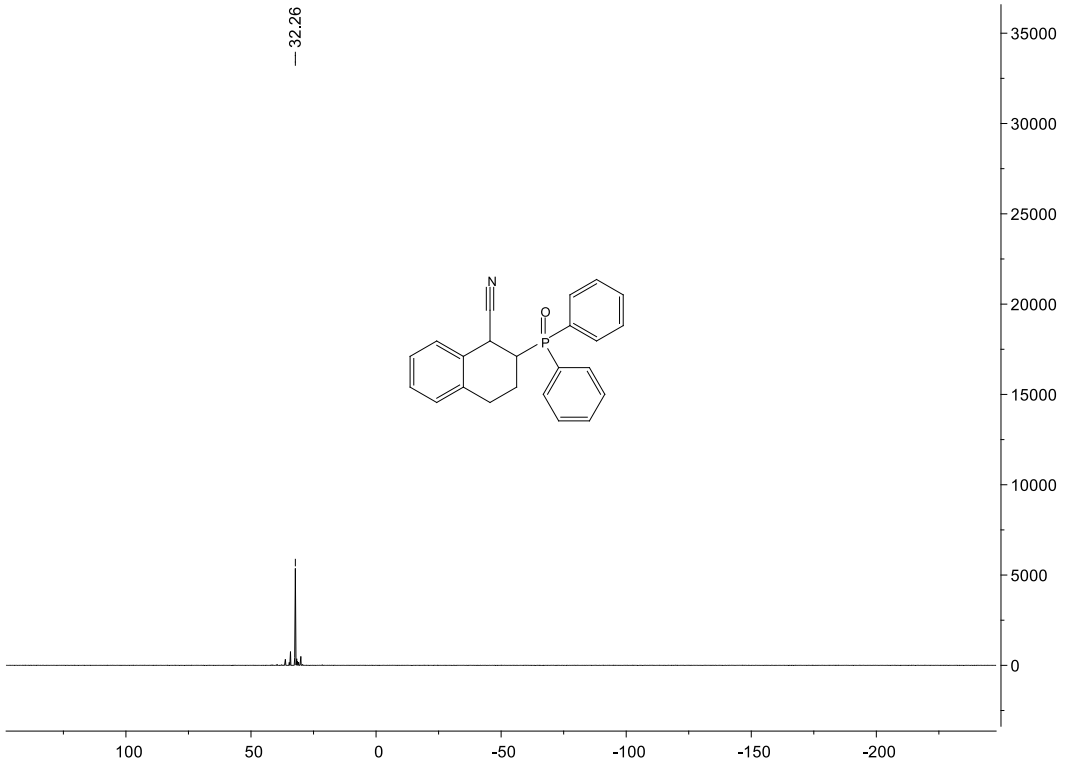

140.22
140.13
136.02
135.94
132.30
132.27
131.92
131.89
131.38
130.76
130.67
130.60
130.39
130.30
129.61
128.65
128.63
128.53
128.51
128.47
127.33
124.29
123.79
119.19
119.16
42.91
42.19
34.35
32.07
N
P=O
f1 (ppm)


30.32
N
P
O
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
100
50
0
-50
-100
-150
-200
f1 (ppm)

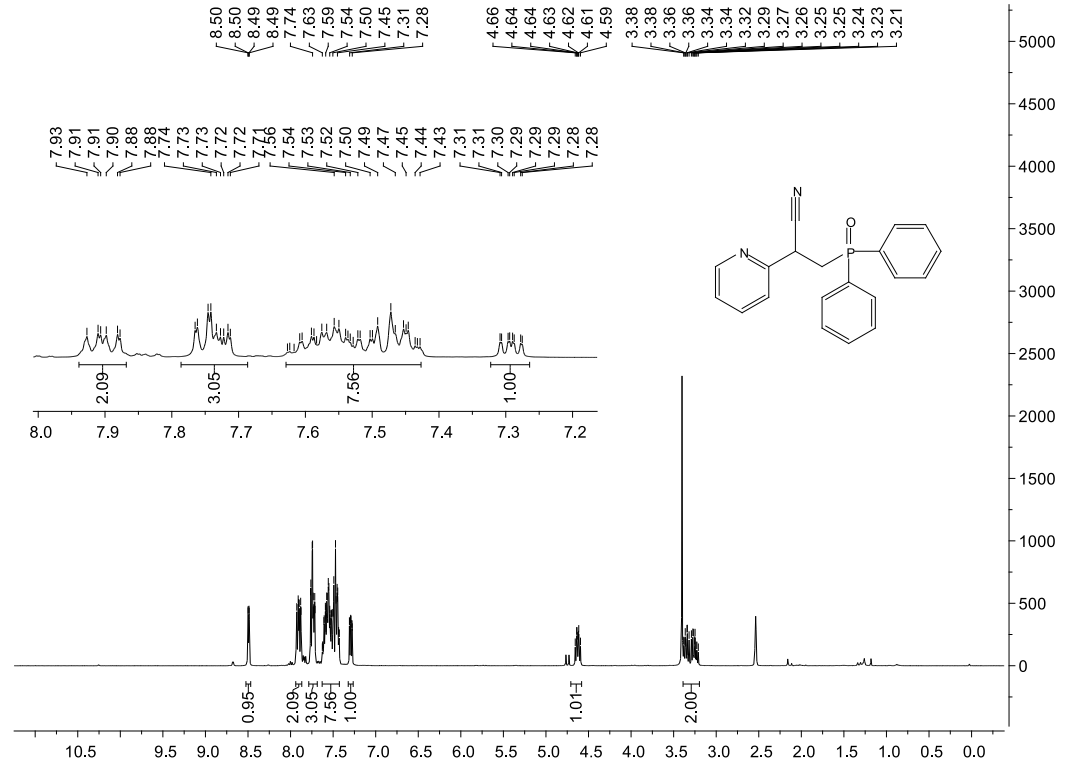
Compound 3t

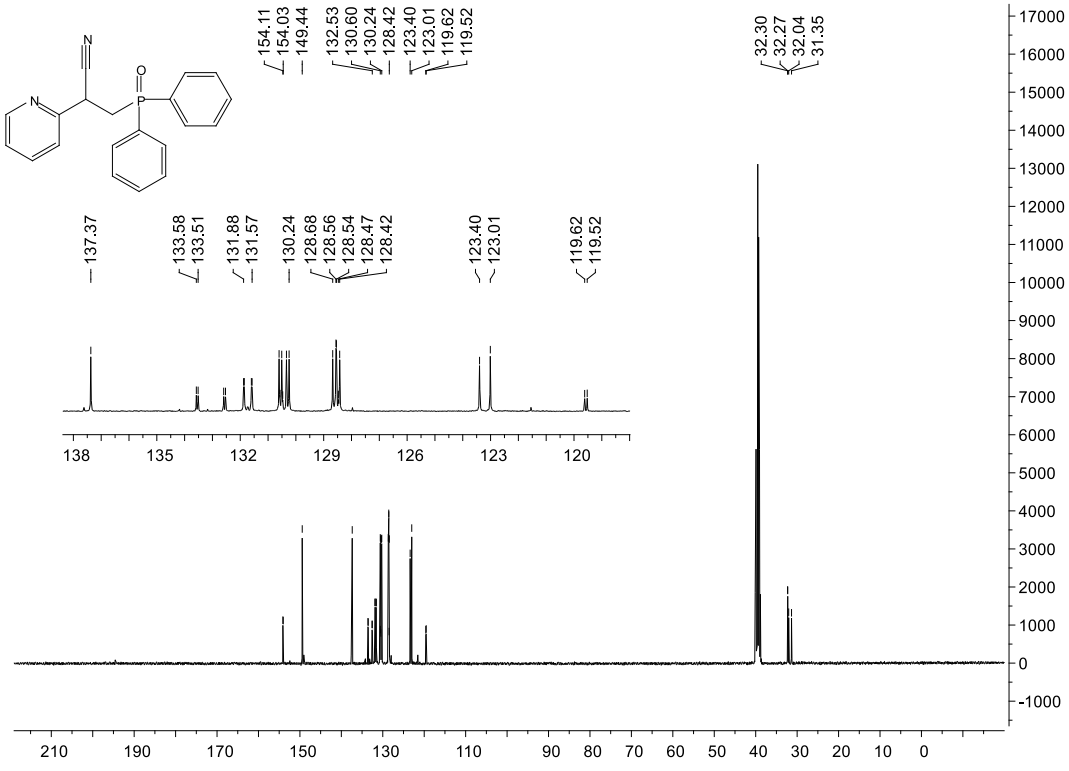

7.96
7.94
7.93
7.93
7.91
7.91
7.88
7.87
7.86
7.86
7.86
7.84
7.84
7.53
7.51
7.36
7.32
7.20
7.18
7.08
7.06
4.48
4.45
4.44
4.42
3.08
3.05
2.90
2.85
2.81
2.08
2.07
2.06
2.05
2.04
2.03
1.96
1.95
1.94
1.94
1.93
1.92
1.92
1.91
1.90
N
O
P
2.07
2.05
6.18
1.01
2.04
1.04
1.00
1.03
2.04
1.01
1.02
21000
20000
19000
18000
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
-2000
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5

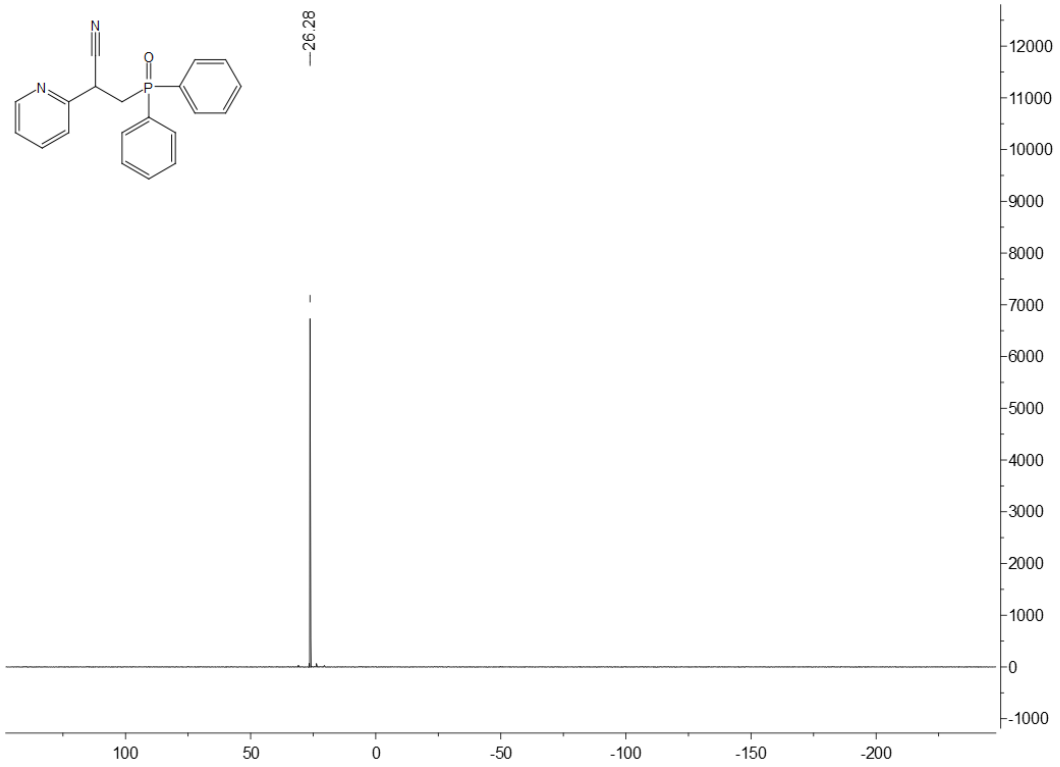

135.73
135.73
132.15
132.12
131.77
131.75
130.93
130.84
130.52
130.43
129.91
129.75
128.65
128.59
128.56
128.47
128.45
128.32
128.21
128.09
127.63
126.61
120.00
119.94
37.12
36.42
27.47
21.42
21.40
28.85
27.56
27.47
N
O
P


32.26
N
O
P

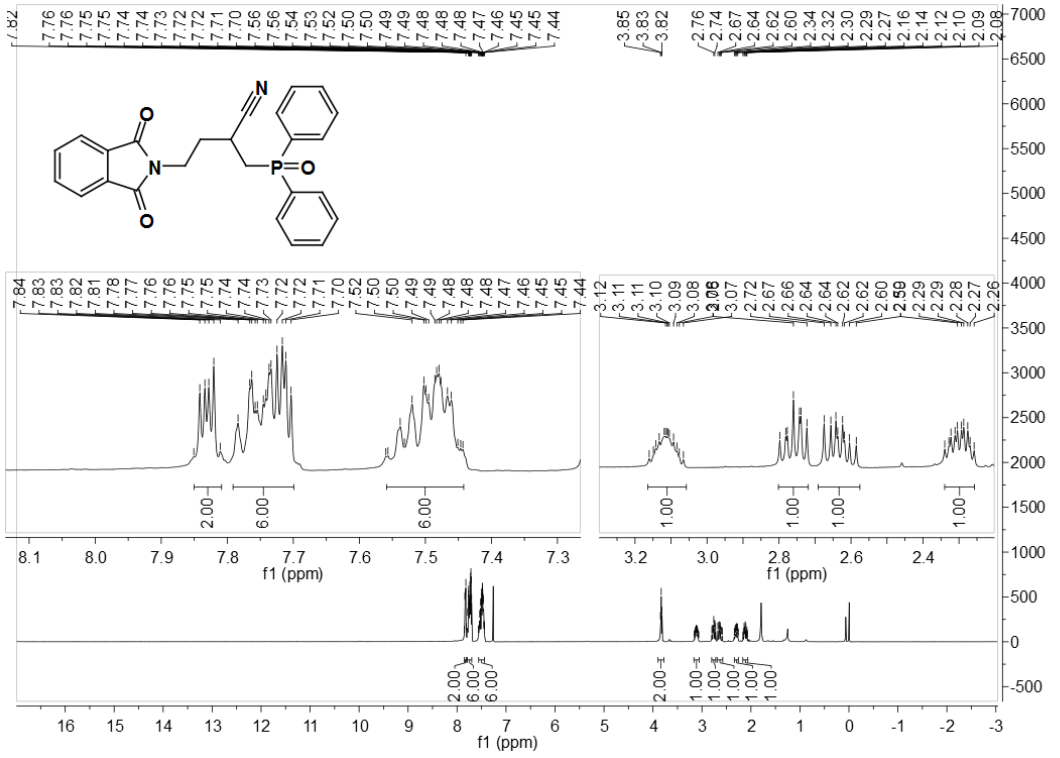
Compound 3u

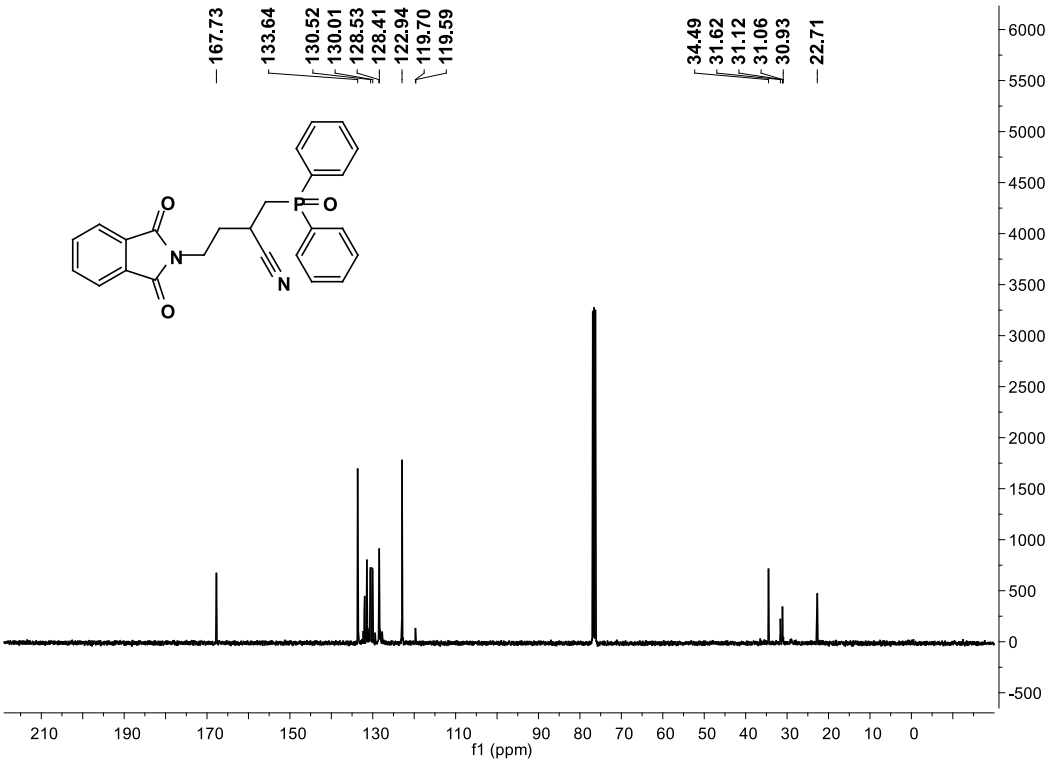

8.50
8.50
8.49
8.49
7.74
7.63
7.59
7.54
7.50
7.45
7.31
7.28
4.66
4.64
4.64
4.63
4.62
4.61
4.59
3.38
3.38
3.36
3.36
3.34
3.34
3.32
3.29
3.27
3.26
3.25
3.25
3.24
3.23
3.21
7.93
7.91
7.91
7.90
7.88
7.88
7.74
7.73
7.73
7.72
7.72
7.71
7.56
7.54
7.53
7.52
7.50
7.49
7.47
7.45
7.44
7.43
7.31
7.31
7.30
7.29
7.29
7.29
7.28
7.28
N
O
P
N
2.09
3.05
7.56
1.00
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
0.95
2.09
3.05
7.56
1.00
1.01
2.00
10.5
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
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0

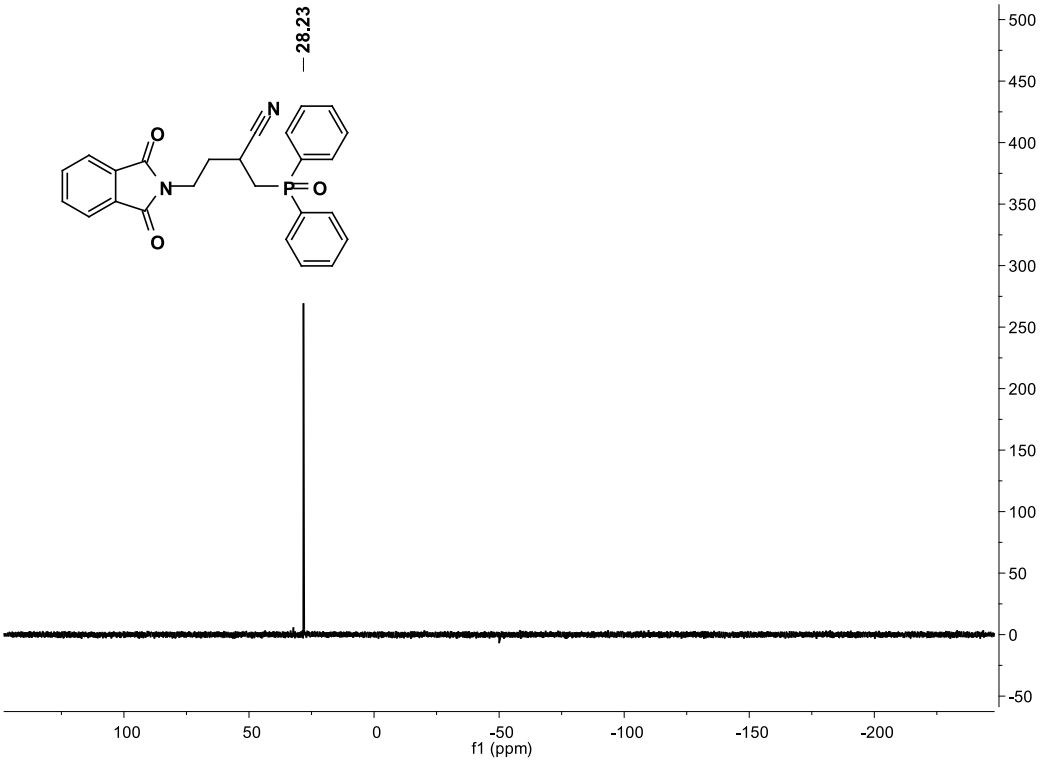

N
O
P
N
154.11
154.03
149.44
132.53
130.60
130.24
128.42
123.40
123.01
119.62
119.52
32.30
32.27
32.04
31.35
137.37
133.58
133.51
131.88
131.57
130.24
128.68
128.56
128.54
128.47
128.42
123.40
123.01
119.62
119.52
138
135
132
129
126
123
120
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
17000
16000
15000
14000
13000
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000


26.28
N
O
P
N
12000
11000
10000
9000
8000
7000
6000
5000
4000
3000
2000
1000
0
-1000
100
50
0
-50
-100
-150
-200

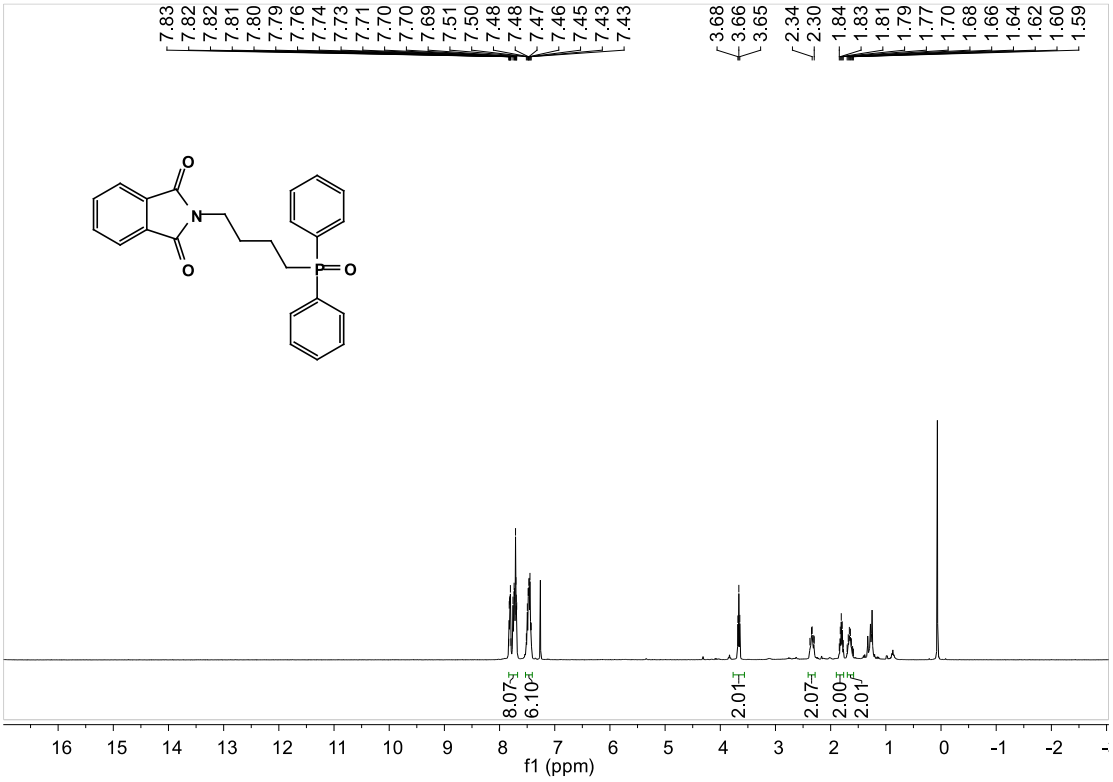
Compound 5a

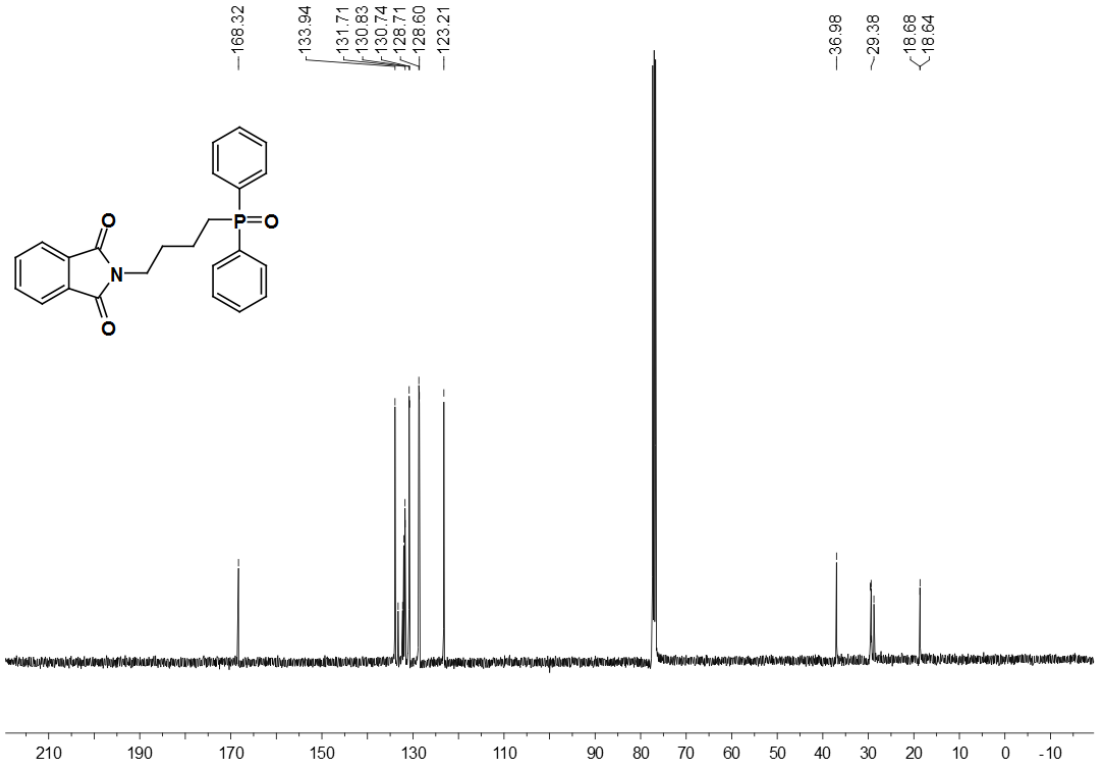

N
O
N
O
P=O
2.00
6.00
6.00
1.00
1.00
1.00
1.00
f1 (ppm)
f1 (ppm)
f1 (ppm)

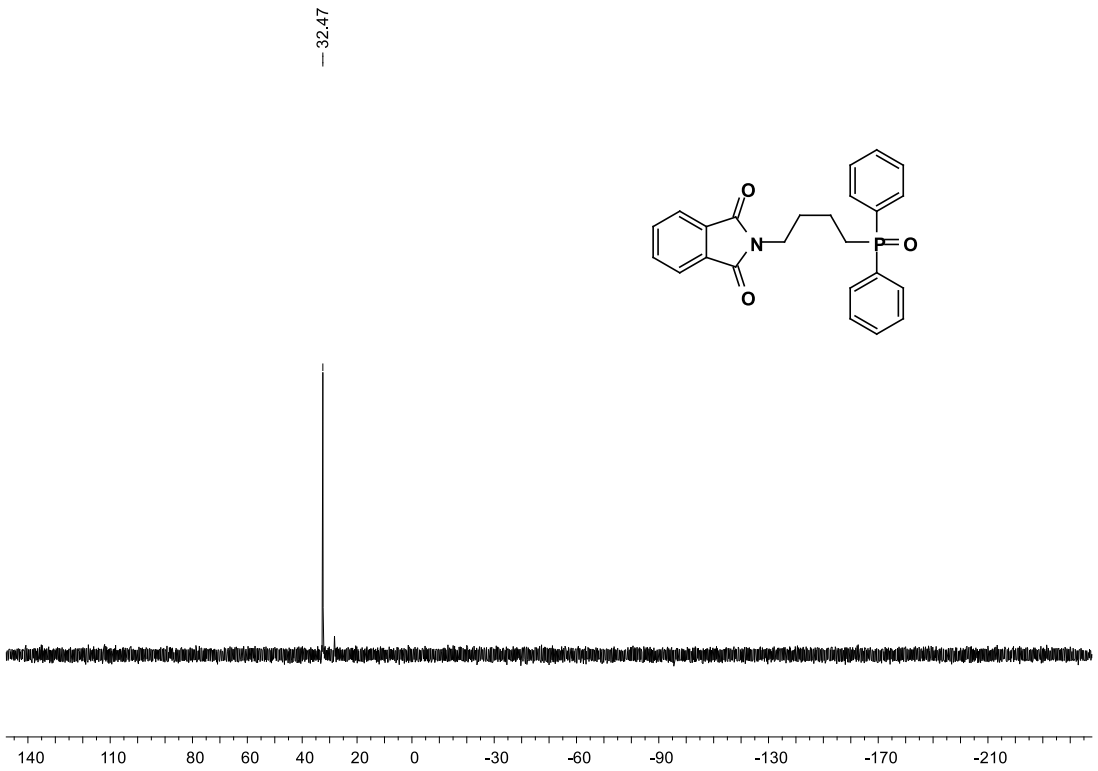

167.73
133.64
130.52
130.01
128.53
128.41
122.94
119.70
119.59
34.49
31.62
31.12
31.06
30.93
22.71
O
N
O
P
O
N
f1 (ppm)


28.23
N
O
N
O
P
O
O
f1 (ppm)

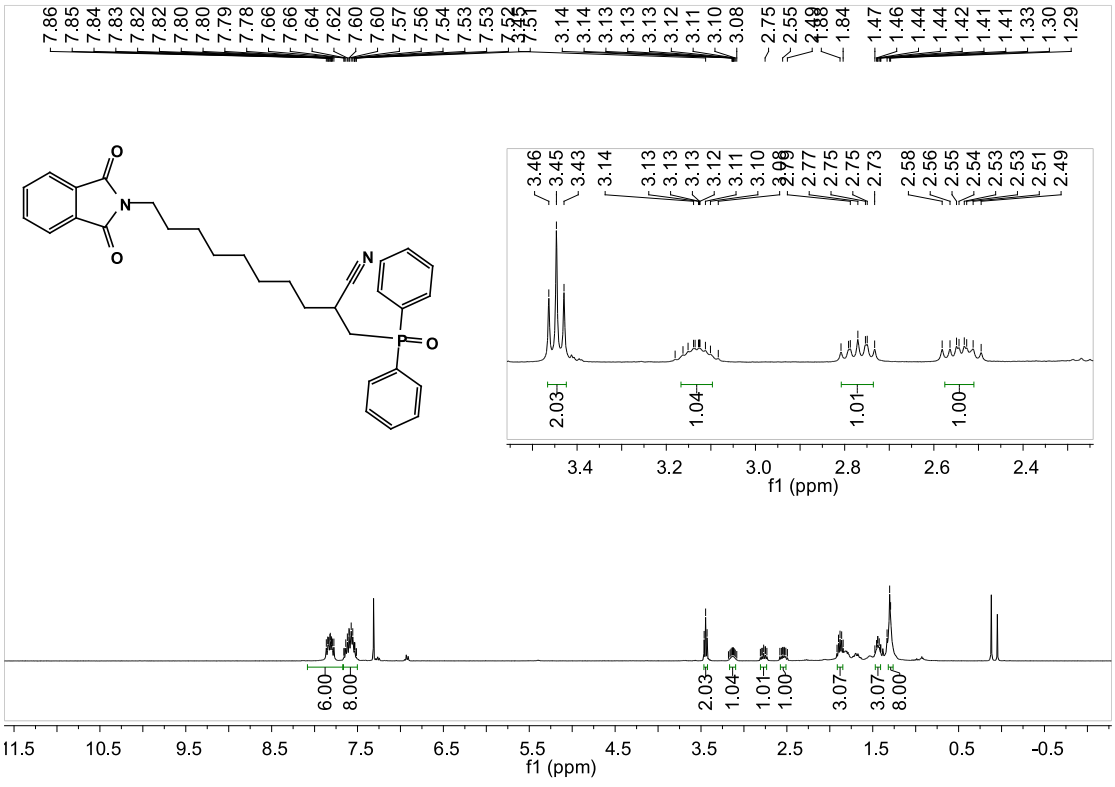
Compound 5a'

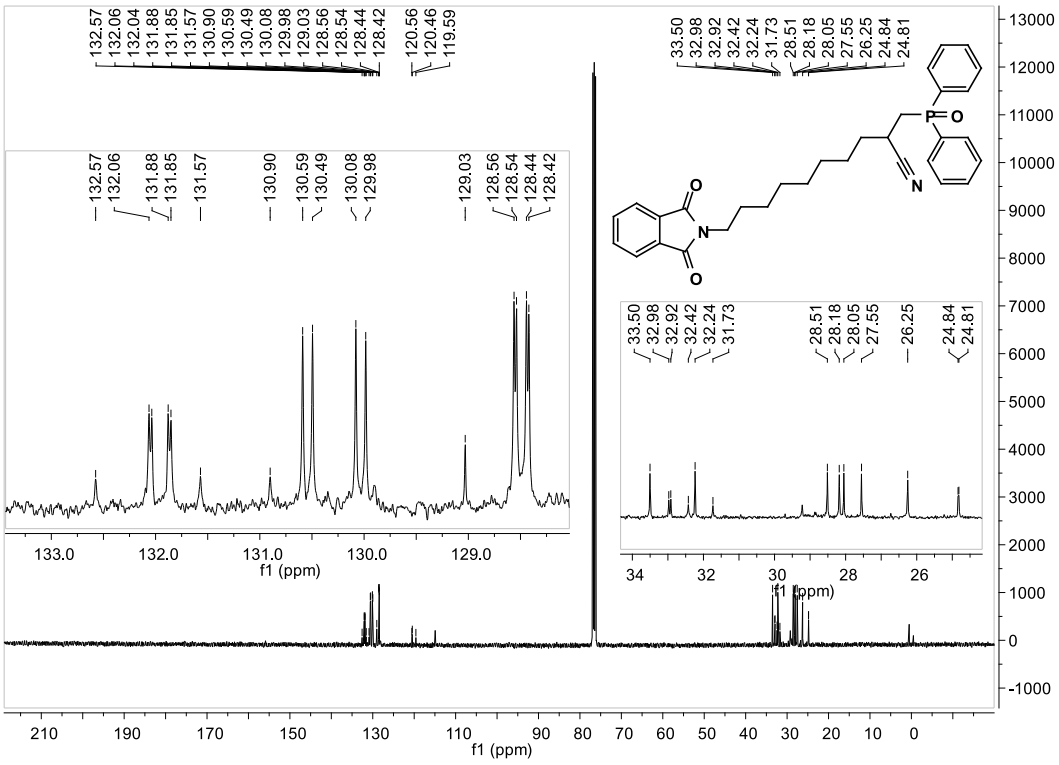

7.83
7.82
7.82
7.81
7.80
7.79
7.76
7.74
7.73
7.71
7.70
7.70
7.69
7.51
7.50
7.48
7.48
7.47
7.46
7.45
7.43
7.43
3.68
3.66
3.65
2.34
2.30
1.84
1.83
1.81
1.79
1.77
1.70
1.68
1.66
1.64
1.62
1.60
1.59
O
N
O
P
O
8.07
6.10
2.01
2.07
2.00
2.01
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2
f1 (ppm)

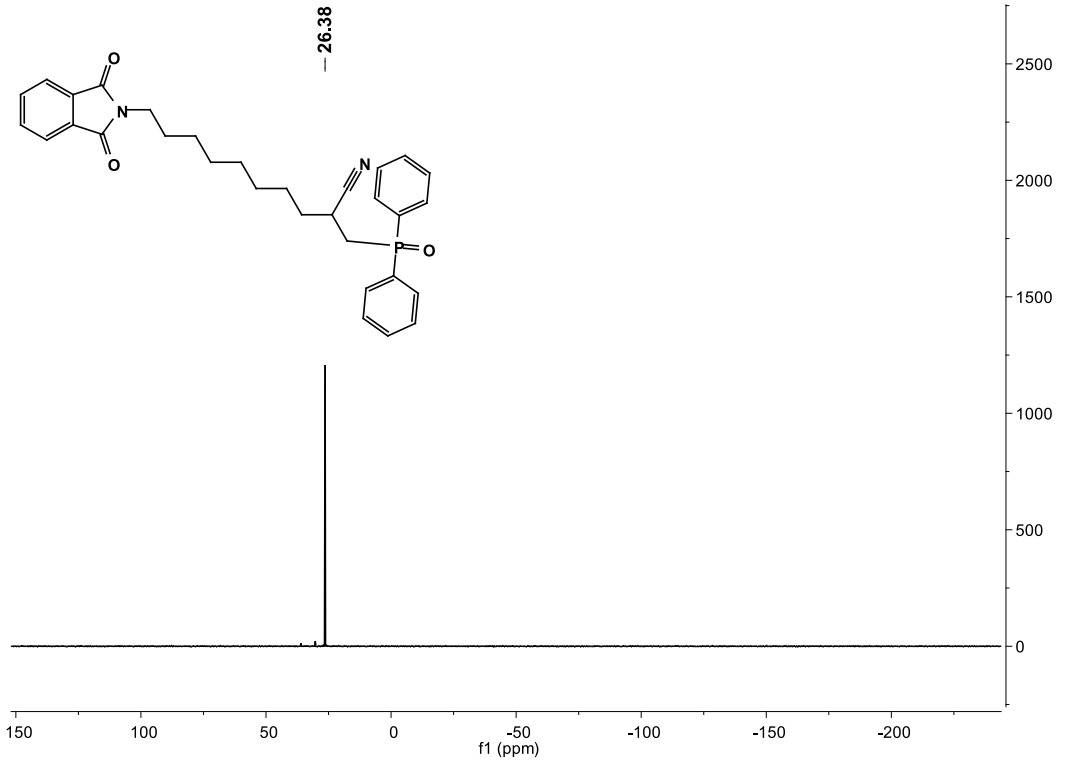

168.32
133.94
131.71
130.83
130.74
128.71
128.60
123.21
36.98
29.38
18.68
18.64
O
N
O
P
O
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10


32.47
O
N
P
O
O
O
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210

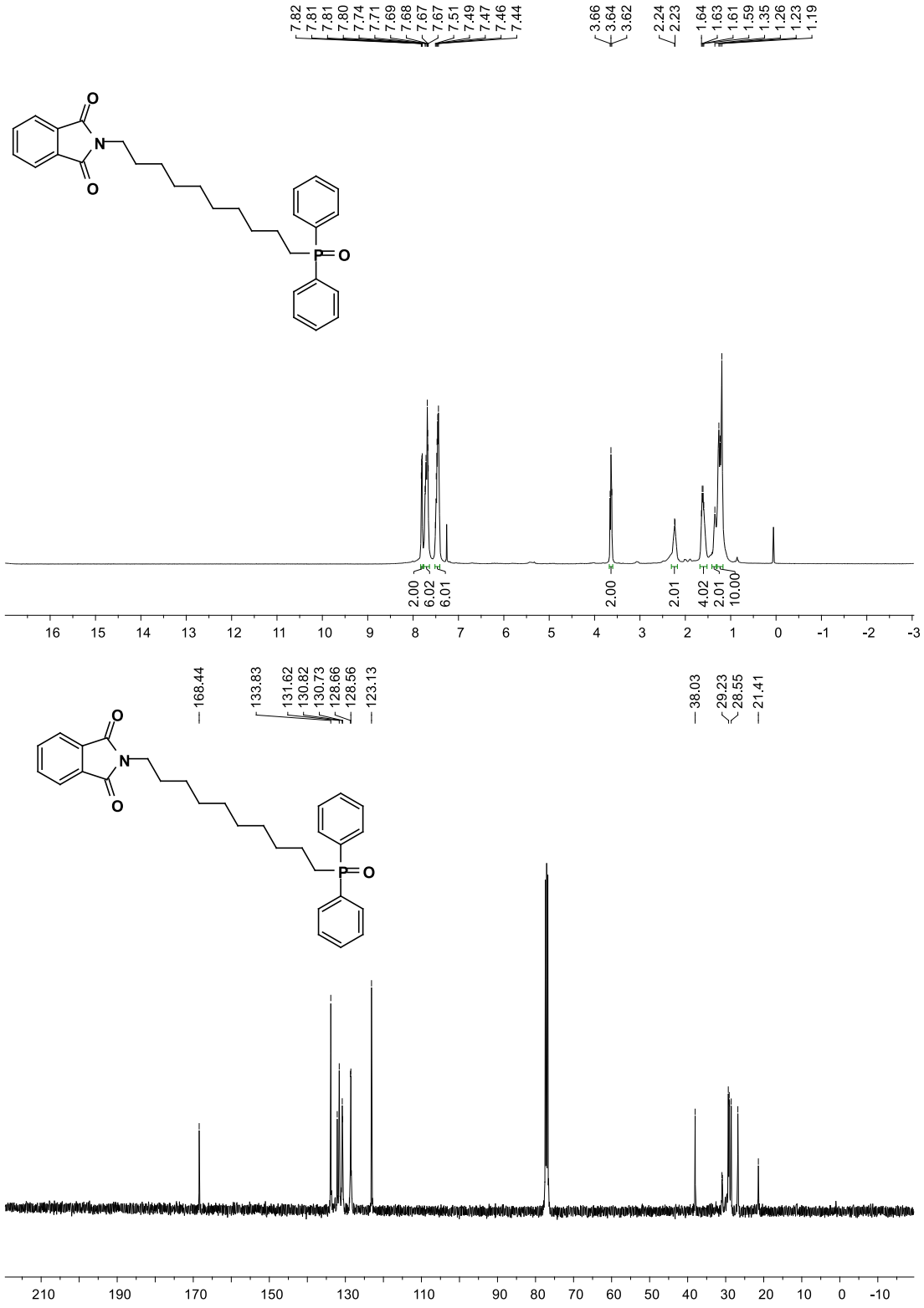
Compound 5b

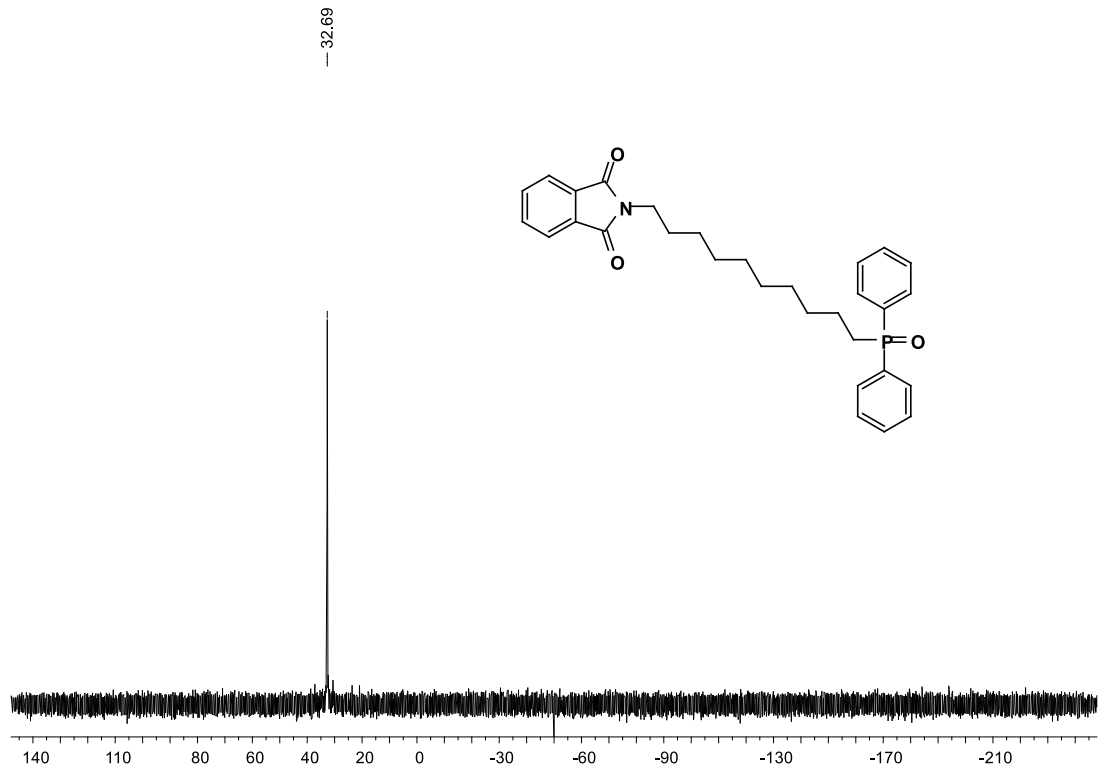

7.86
7.85
7.84
7.83
7.82
7.82
7.80
7.80
7.79
7.78
7.66
7.66
7.64
7.62
7.60
7.60
7.57
7.56
7.54
7.53
7.53
3.14
3.14
3.13
3.13
3.13
3.12
3.11
3.10
3.08
2.75
2.55
1.84
1.47
1.46
1.44
1.44
1.42
1.41
1.41
1.33
1.30
1.29
O
N
O
N
P
O
3.46
3.45
3.43
3.14
3.13
3.13
3.13
3.12
3.11
3.10
2.77
2.75
2.75
2.73
2.58
2.56
2.55
2.54
2.53
2.53
2.51
2.49
2.03
1.04
1.01
1.00
3.4
3.2
3.0
2.8
2.6
2.4
f1 (ppm)
6.00
8.00
2.03
1.04
1.01
1.00
3.07
3.07
8.00
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)


132.57
132.06
132.04
131.88
131.85
131.57
130.90
130.59
130.49
130.08
129.98
129.03
128.56
128.54
128.44
128.42
120.56
120.46
119.59
33.50
32.98
32.92
32.42
32.24
31.73
28.51
28.18
28.05
27.55
26.25
24.84
24.81
O
N
O
P
O
N
f1 (ppm)
f1 (ppm)

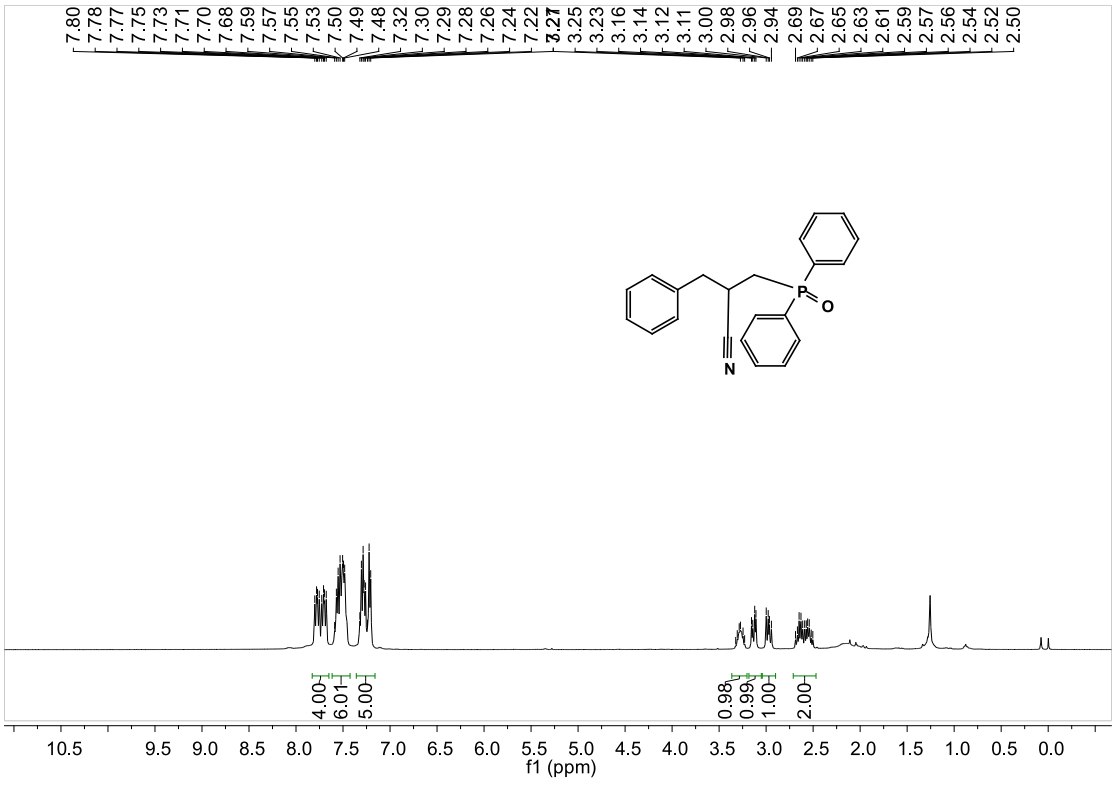

26.38
O
N
O
N
P
O
f1 (ppm)

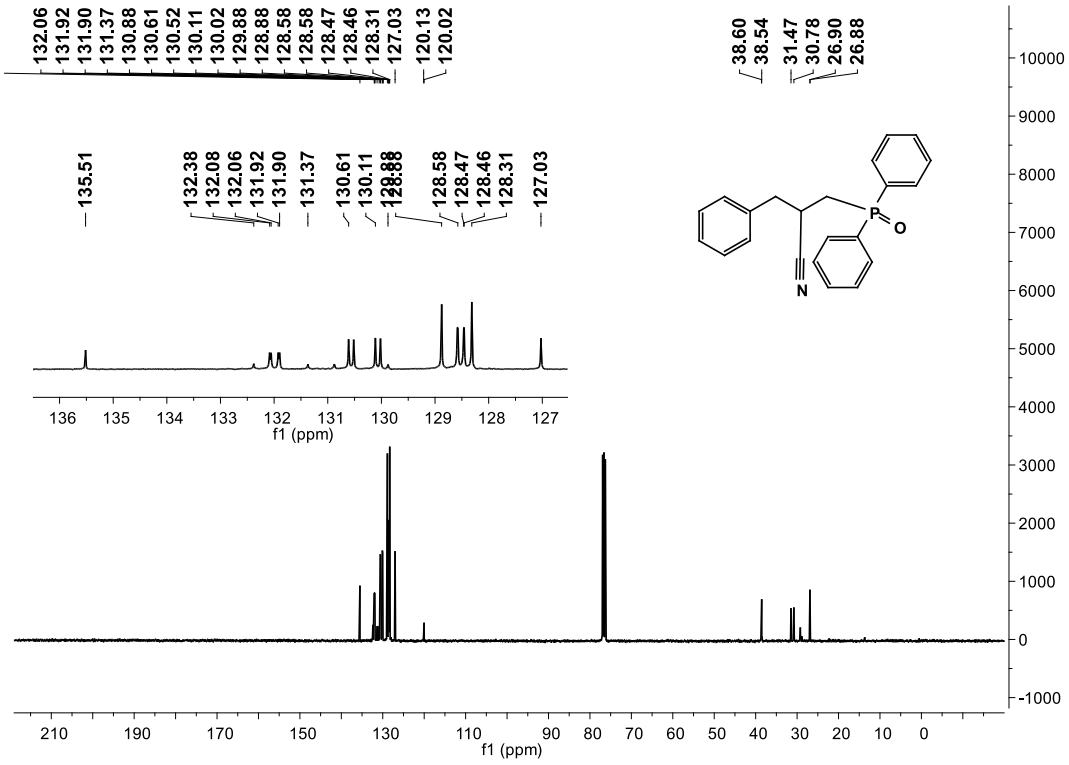
Compound 5b’

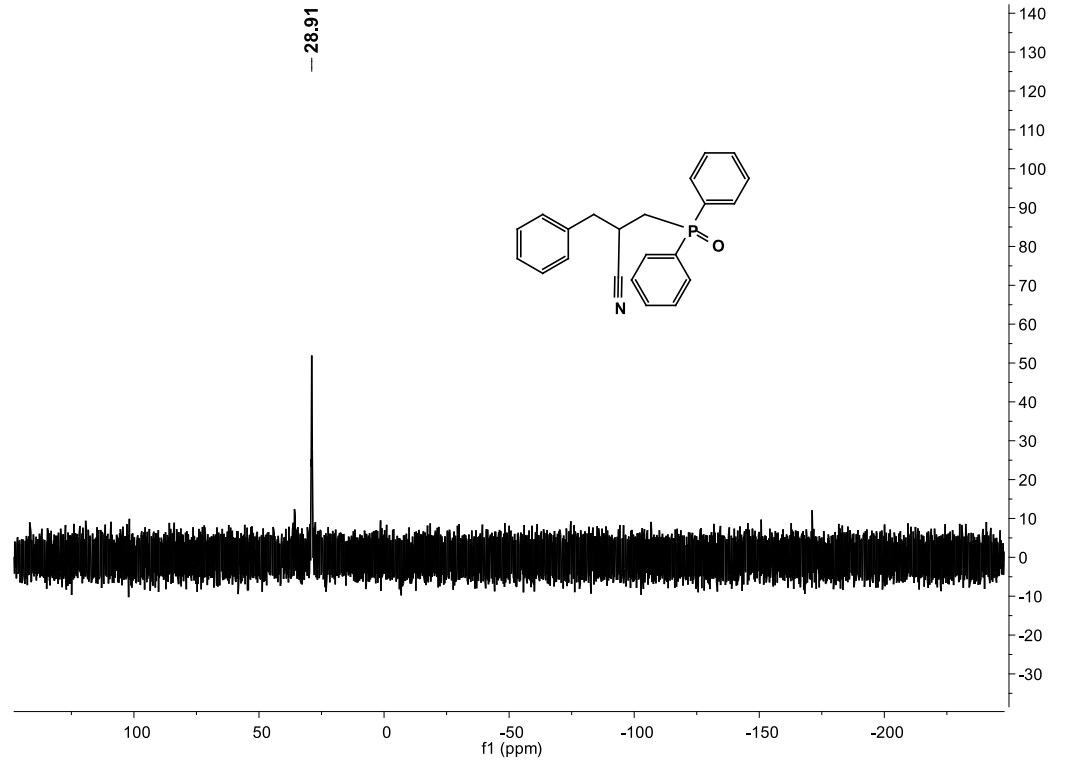

7.82
7.81
7.81
7.80
7.74
7.71
7.69
7.68
7.67
7.67
7.51
7.49
7.47
7.46
7.44
3.66
3.64
3.62
2.24
2.23
1.64
1.63
1.61
1.59
1.35
1.26
1.23
1.19
O
N
O
P
O
2.00
6.02
6.01
2.00
2.01
4.02
2.01
10.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
168.44
133.83
131.62
130.82
130.73
128.66
128.56
123.13
38.03
29.23
28.55
21.41
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10


32.69
O
N
O
P
O
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210

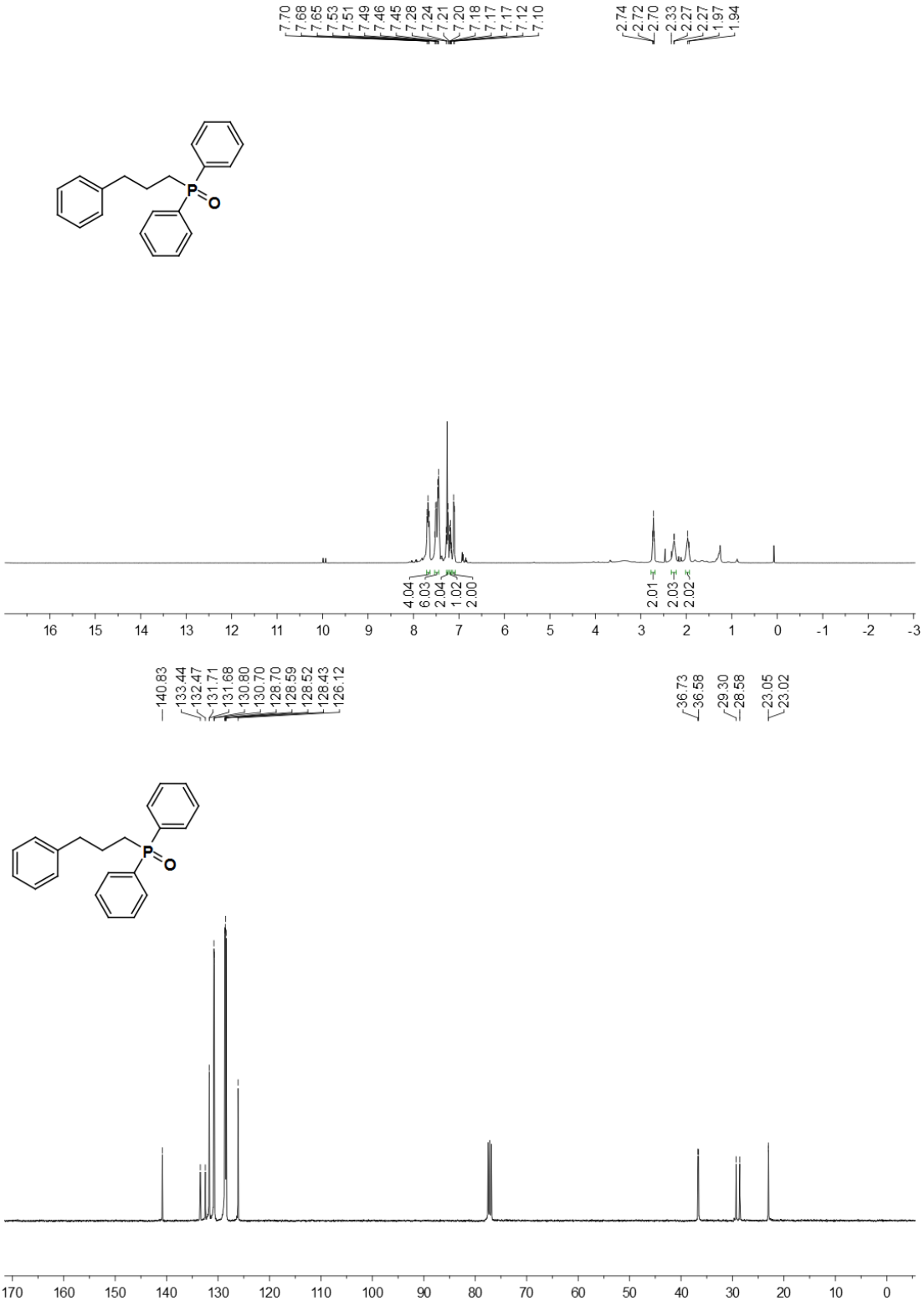
Compound 5c

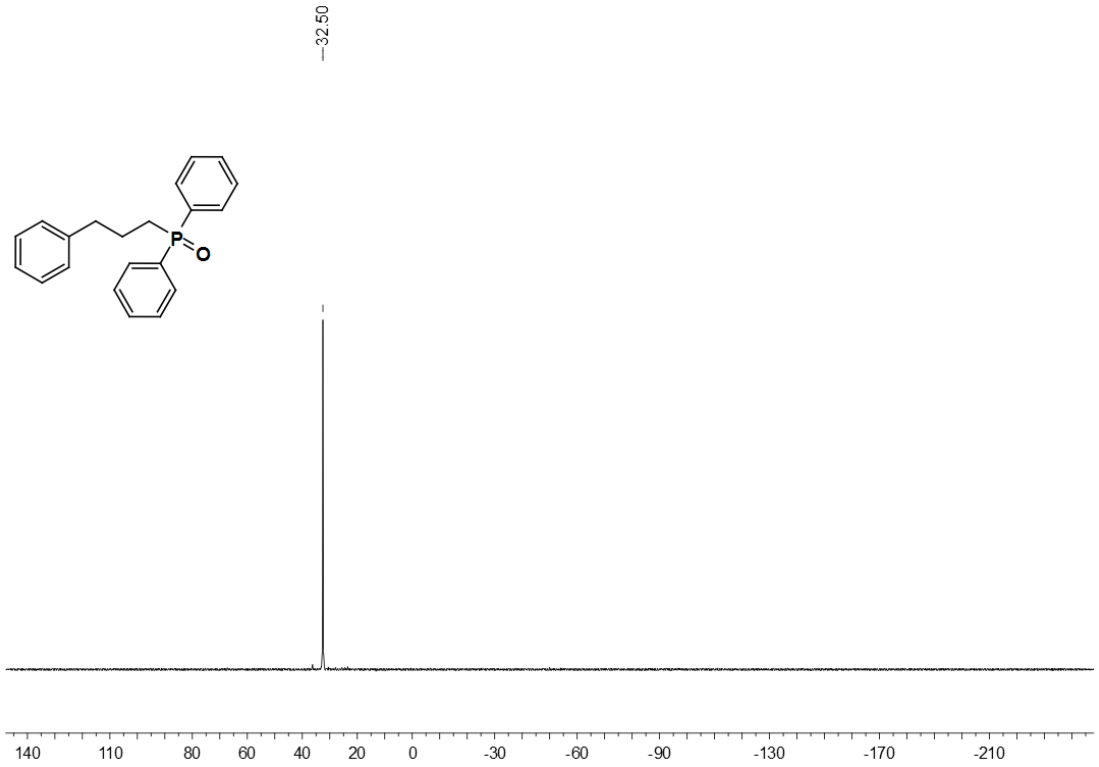

7.80
7.78
7.77
7.75
7.73
7.71
7.70
7.68
7.59
7.57
7.55
7.53
7.50
7.49
7.48
7.32
7.30
7.29
7.28
7.26
7.24
7.22
3.27
3.25
3.23
3.16
3.14
3.12
3.11
3.00
2.98
2.96
2.94
2.69
2.67
2.65
2.63
2.61
2.59
2.57
2.56
2.54
2.52
2.50
P
O
N
4.00
6.01
5.00
0.98
0.99
1.00
2.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


132.06
131.92
131.90
131.37
130.88
130.61
130.52
130.11
130.02
129.88
128.88
128.58
128.58
128.47
128.46
128.31
127.03
120.13
120.02
38.60
38.54
31.47
30.78
26.90
26.88
135.51
132.38
132.08
132.06
131.92
131.90
131.37
130.61
130.11
128.58
128.47
128.46
128.31
127.03
P
O
N
f1 (ppm)
f1 (ppm)

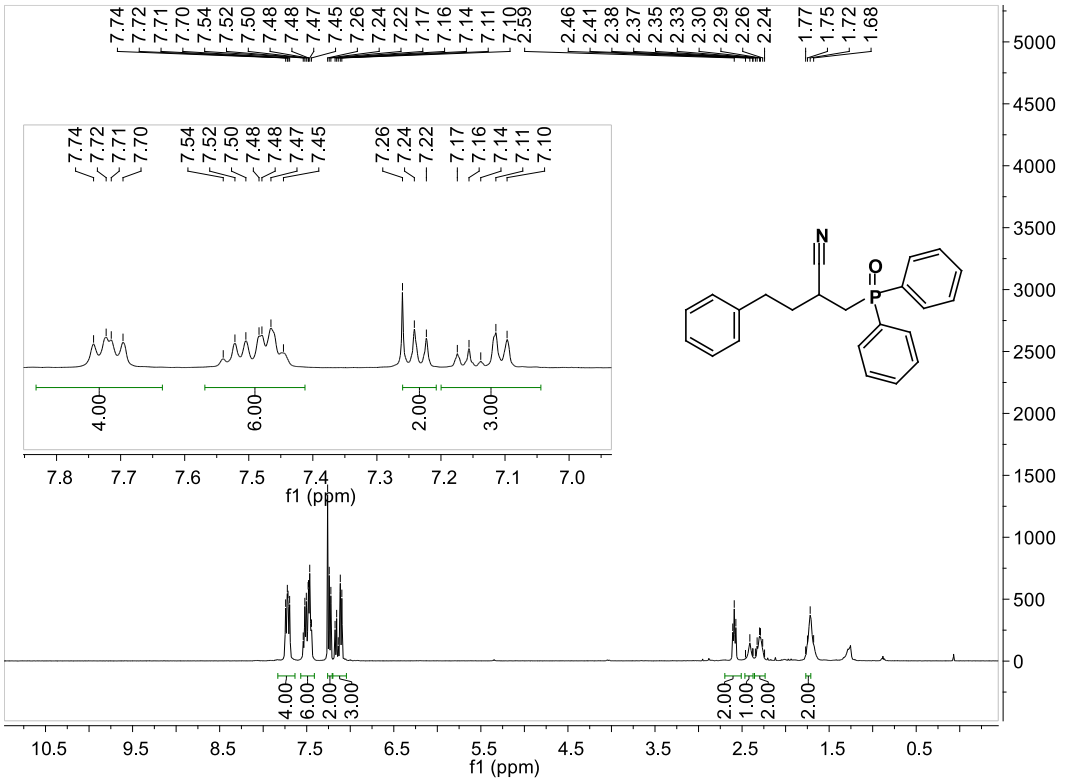

28.91
P
O
N
f1 (ppm)

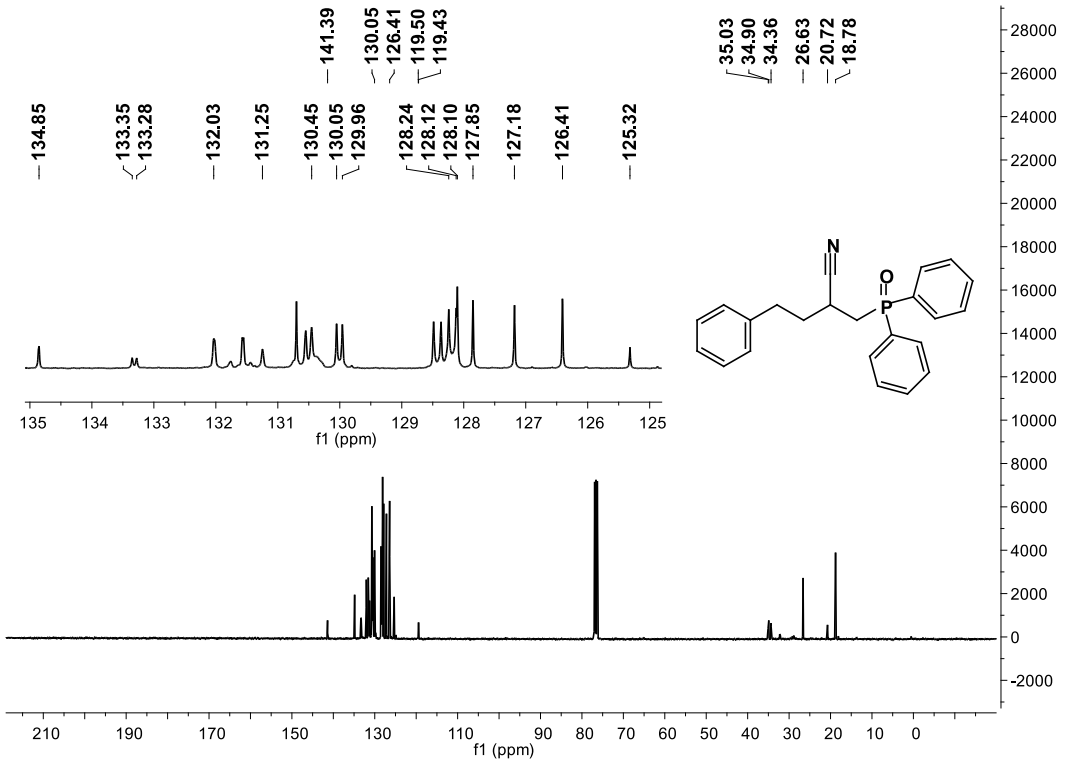
Compound 5c'

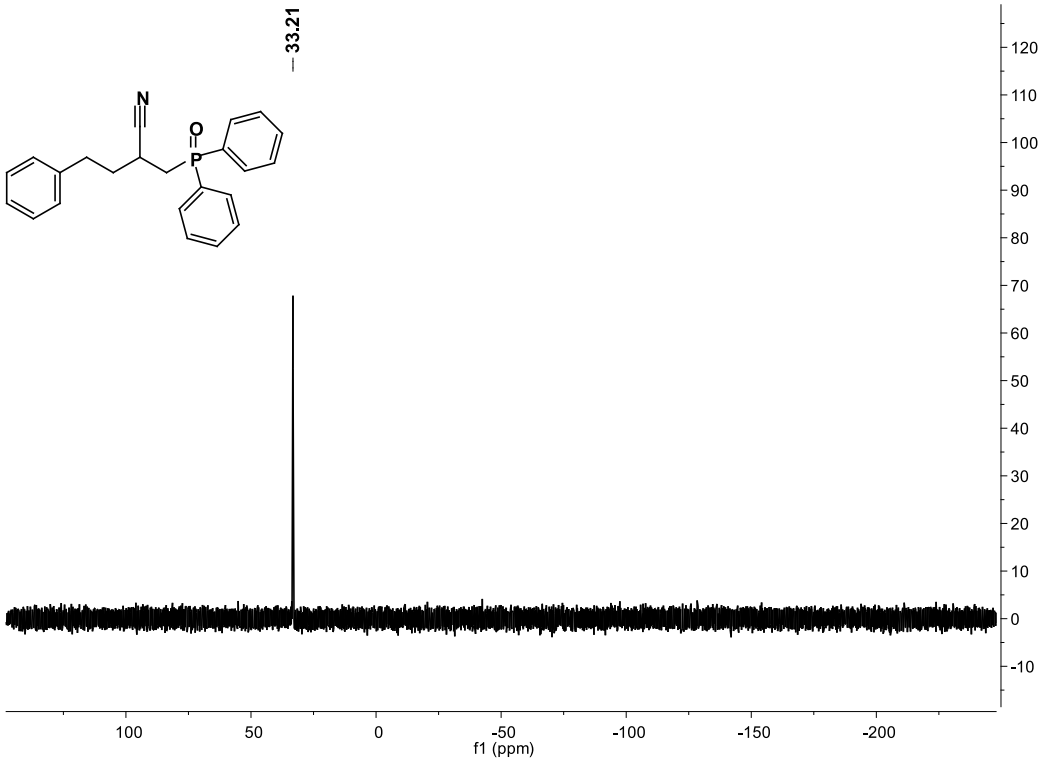

7.70
7.68
7.65
7.53
7.51
7.49
7.46
7.45
7.28
7.24
7.21
7.20
7.18
7.17
7.17
7.12
7.10
2.74
2.72
2.70
2.33
2.27
2.27
1.97
1.94
P
O
4.04
6.03
2.04
1.02
2.00
2.01
2.03
2.02
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3
140.83
133.44
132.47
131.71
131.68
130.80
130.70
128.70
128.59
128.52
128.43
126.12
36.73
36.58
29.30
28.58
23.05
23.02
P
O
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0


32.50
P
O
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210

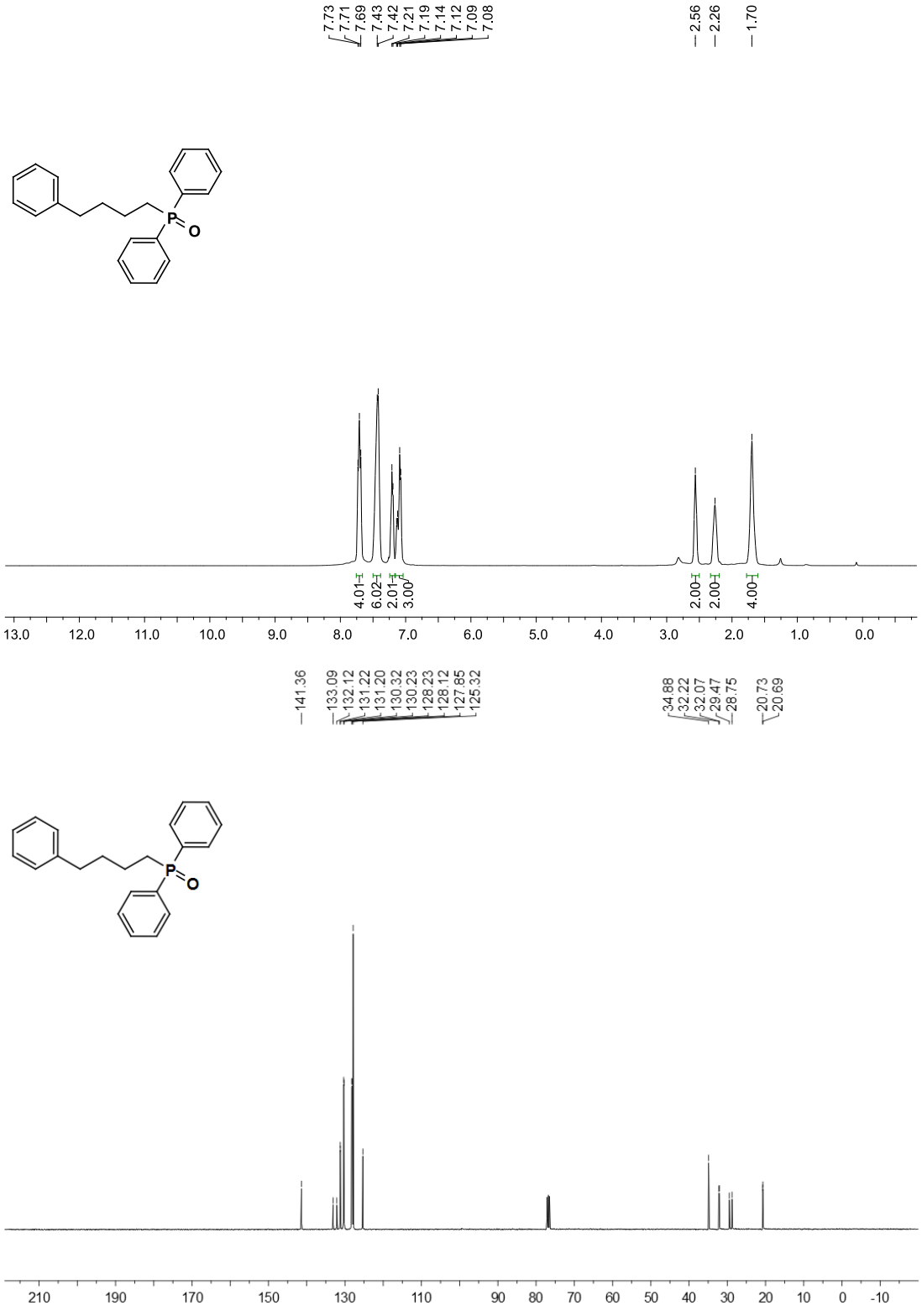
Compound 5d

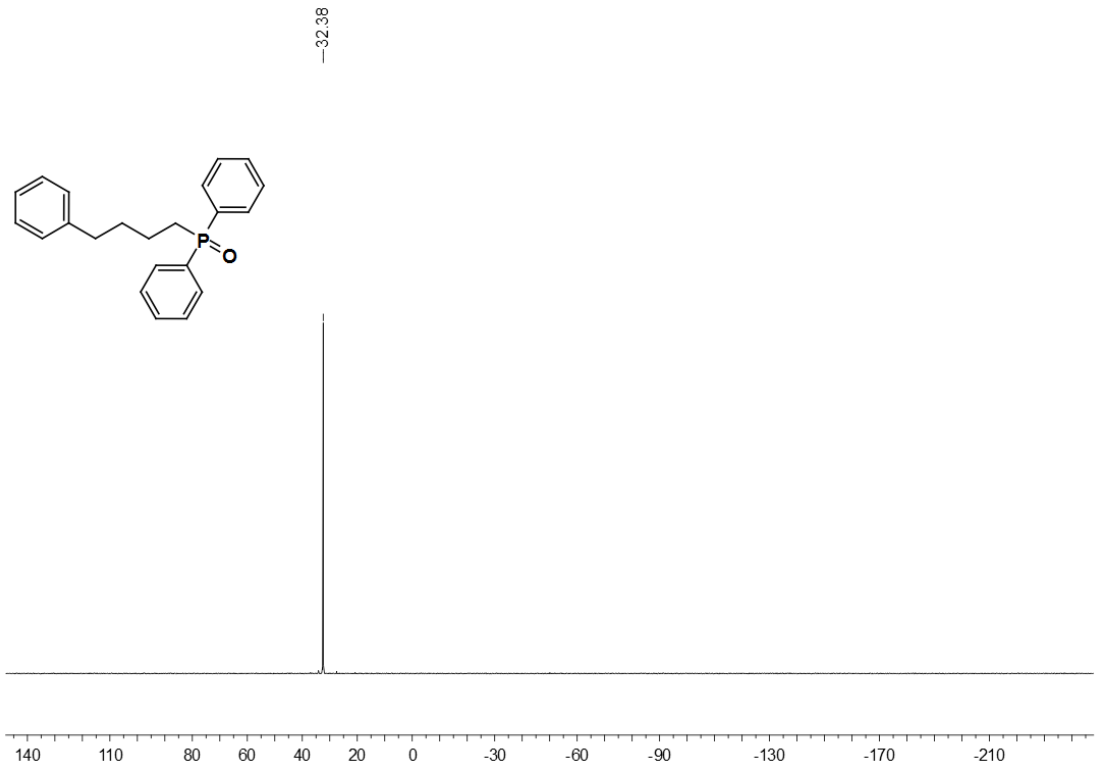

7.74
7.72
7.71
7.70
7.54
7.52
7.50
7.48
7.48
7.47
7.45
7.26
7.24
7.22
7.17
7.16
7.14
7.11
7.10
2.59
2.46
2.41
2.38
2.37
2.35
2.33
2.30
2.29
2.26
2.24
1.77
1.75
1.72
1.68
N
O
P
4.00
6.00
2.00
3.00
f1 (ppm)
5000
4500
4000
3500
3000
2500
2000
1500
1000
500
0
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5


134.85
133.35
133.28
132.03
131.25
130.45
130.05
129.96
128.24
128.12
128.10
127.85
127.18
126.41
125.32
141.39
130.05
126.41
119.50
119.43
35.03
34.90
34.36
26.63
20.72
18.78
N
O
P
f1 (ppm)

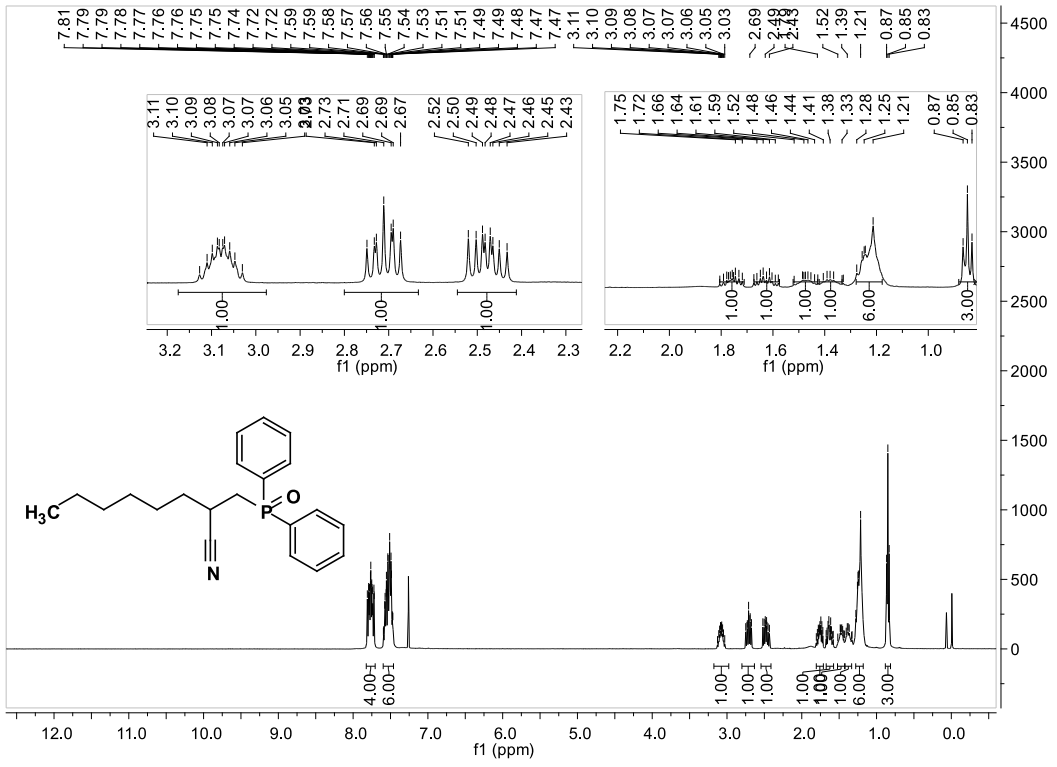

33.21
N
O
P
f1 (ppm)

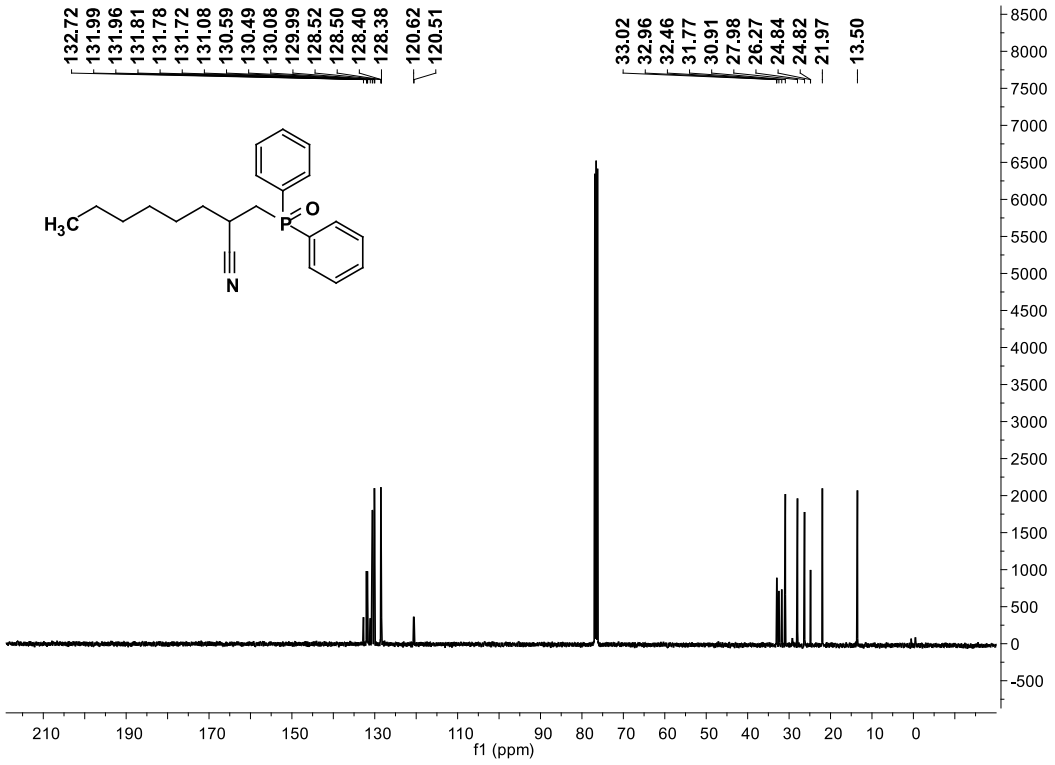
Compound 5d’

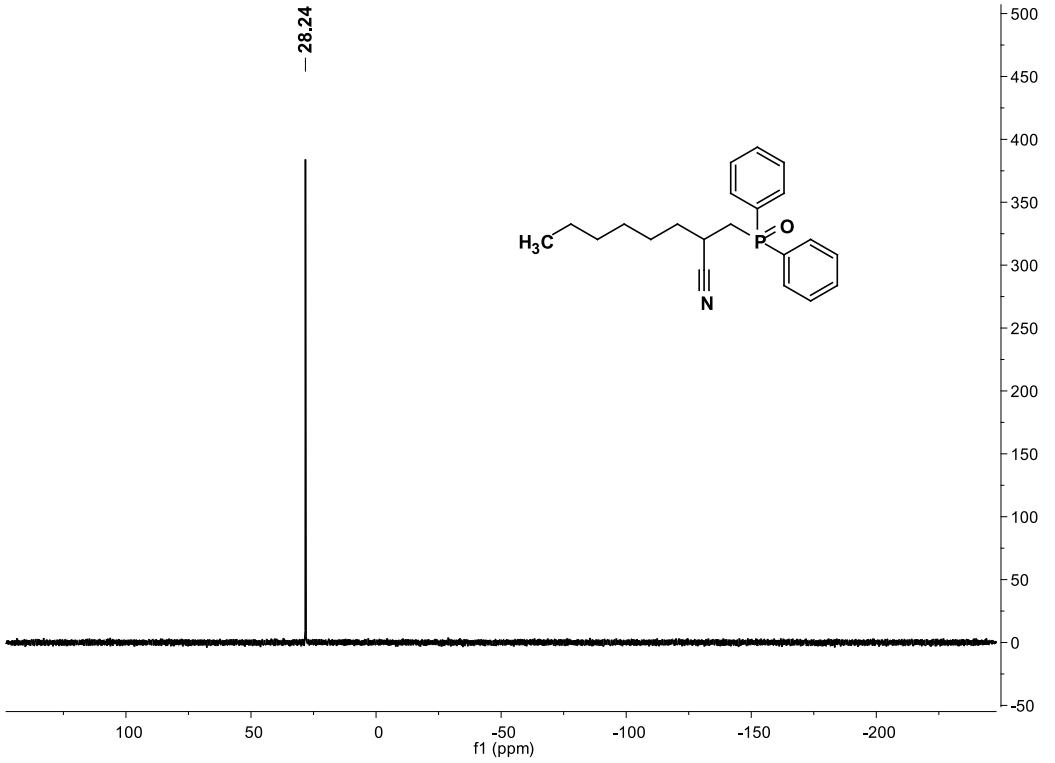

7.73
7.71
7.69
7.43
7.42
7.21
7.19
7.14
7.12
7.09
7.08
2.56
2.26
1.70
O
4.01
6.02
2.01
3.00
2.00
2.00
4.00
13.0
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
141.36
133.09
132.12
131.22
131.20
130.32
130.23
128.23
128.12
127.85
125.32
34.88
32.22
32.07
29.47
28.75
20.73
20.69
O
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10


32.38
P
O
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210

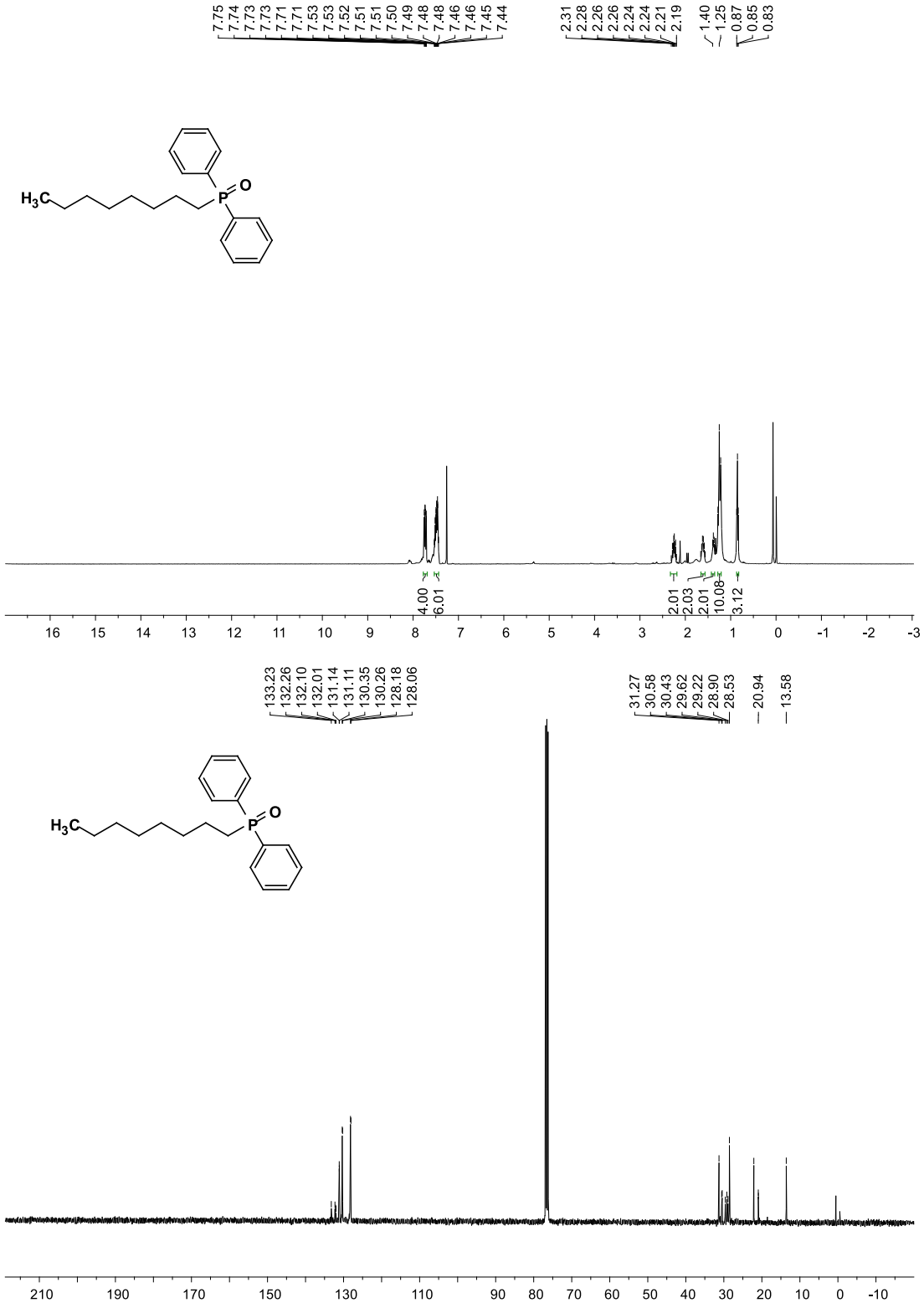
Compound 5e

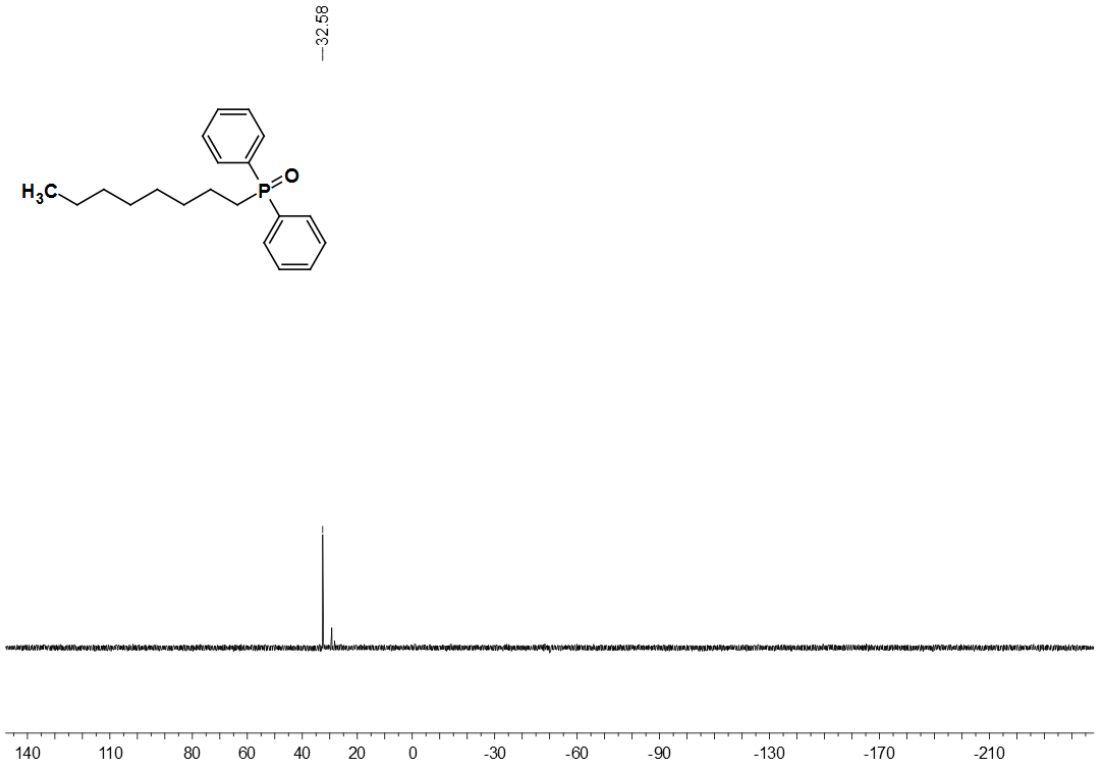

H3C
P
O
N
f1 (ppm)
12.0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0


132.72
131.99
131.96
131.81
131.78
131.72
131.08
130.59
130.49
130.08
129.99
128.52
128.50
128.40
128.38
120.62
120.51
33.02
32.96
32.46
31.77
30.91
27.98
26.27
24.84
24.82
21.97
13.50
H3C
P
O
N
f1 (ppm)

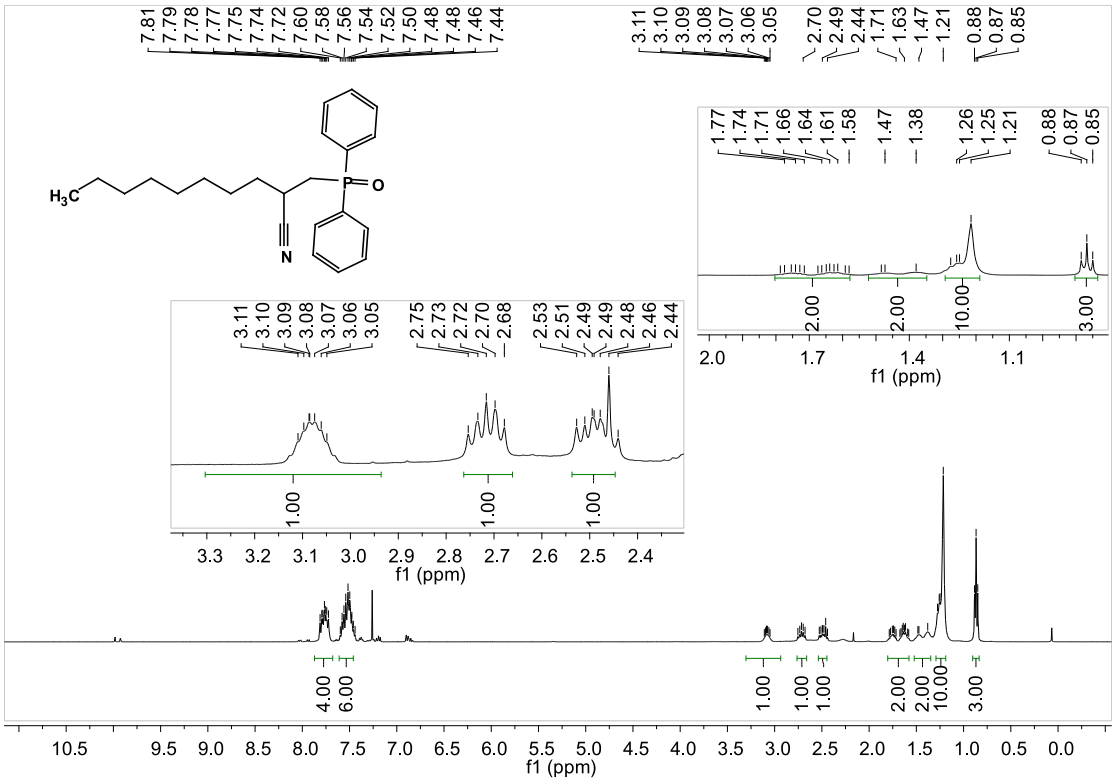

28.24
H3C
P
O
N
f1 (ppm)

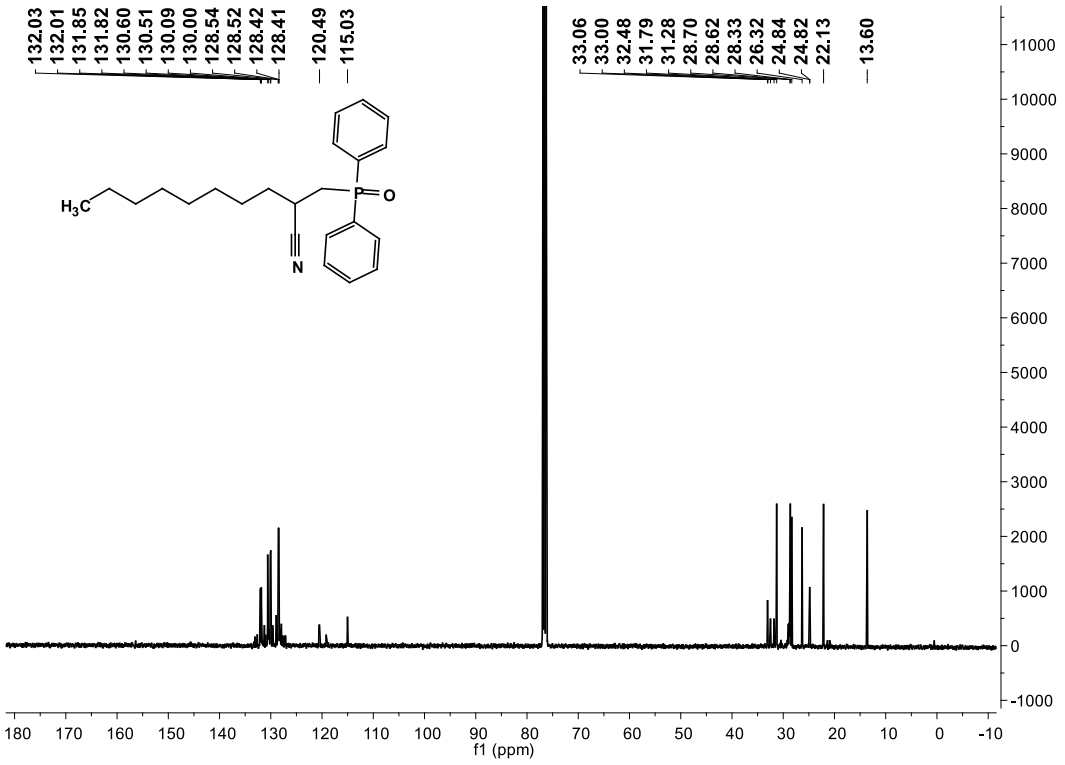
Compound 5e'

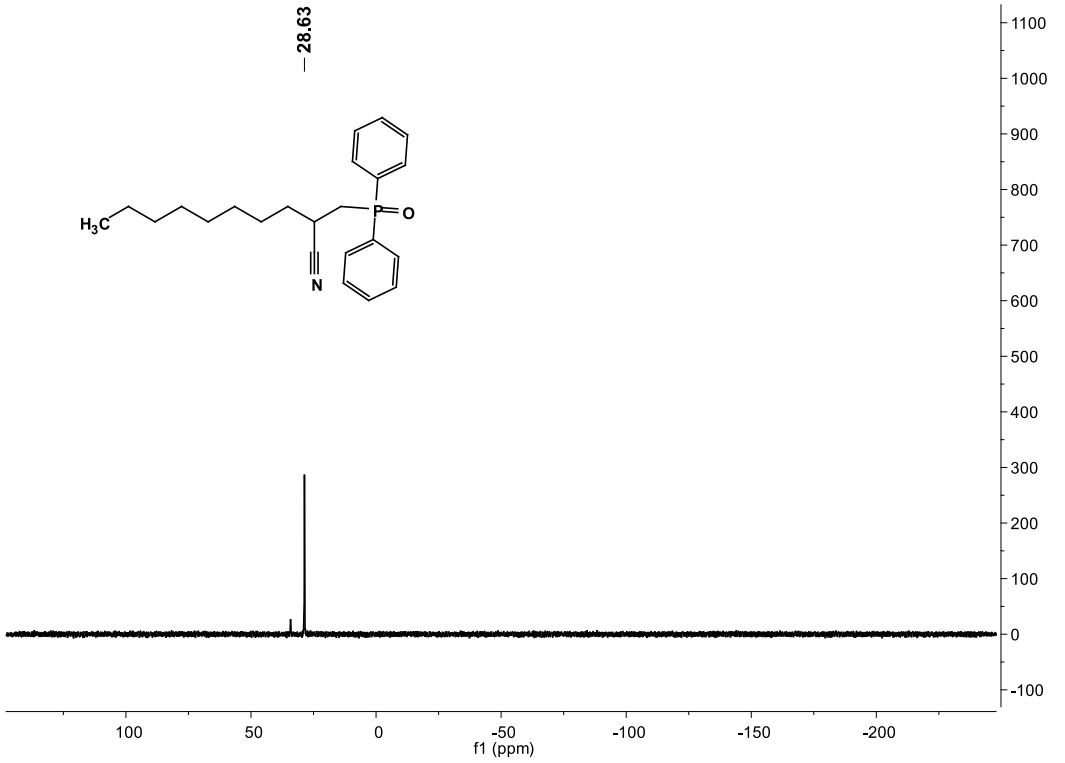

7.75
7.74
7.73
7.73
7.71
7.71
7.53
7.53
7.52
7.51
7.51
7.50
7.49
7.48
7.48
7.46
7.46
7.45
7.44
2.31
2.28
2.26
2.26
2.24
2.24
2.21
2.19
1.40
1.25
0.87
0.85
0.83
H3C
P
O
4.00
6.01
2.01
2.03
2.01
10.08
3.12
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
133.23
132.26
132.10
132.01
131.14
131.11
130.35
130.26
128.18
128.06
31.27
30.58
30.43
29.62
29.22
28.90
28.53
20.94
13.58
H3C
P
O
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10


32.58
H3C
P
O
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210

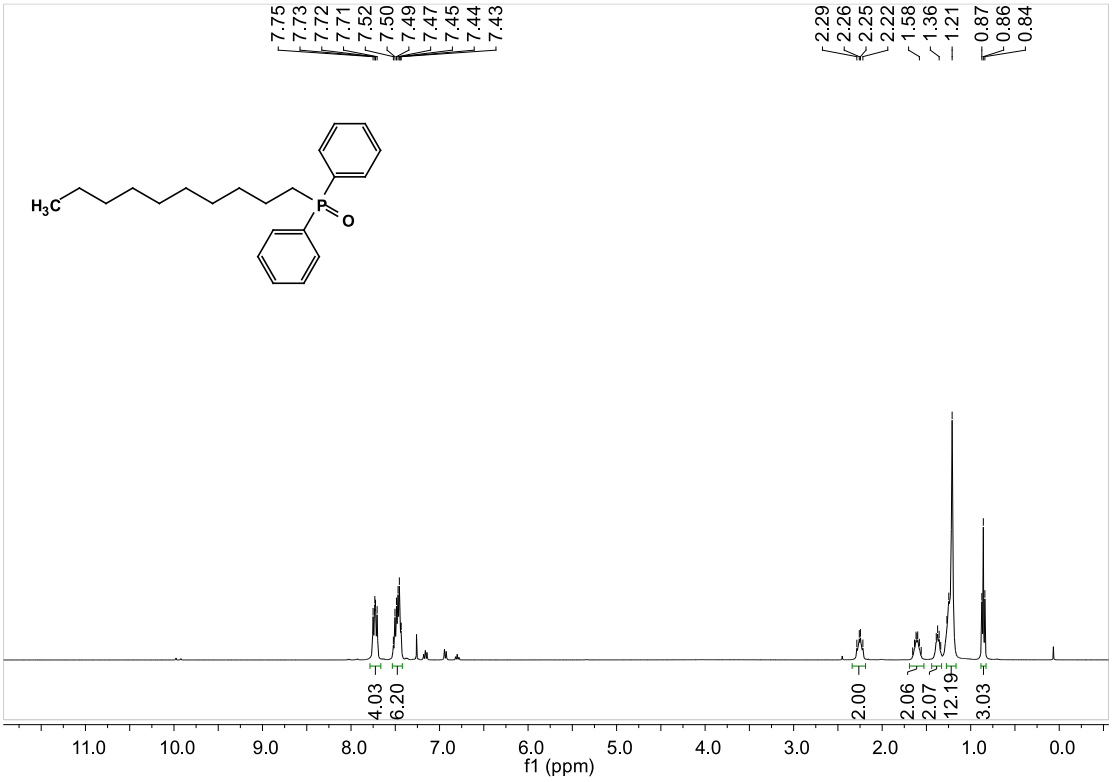
Compound 5f

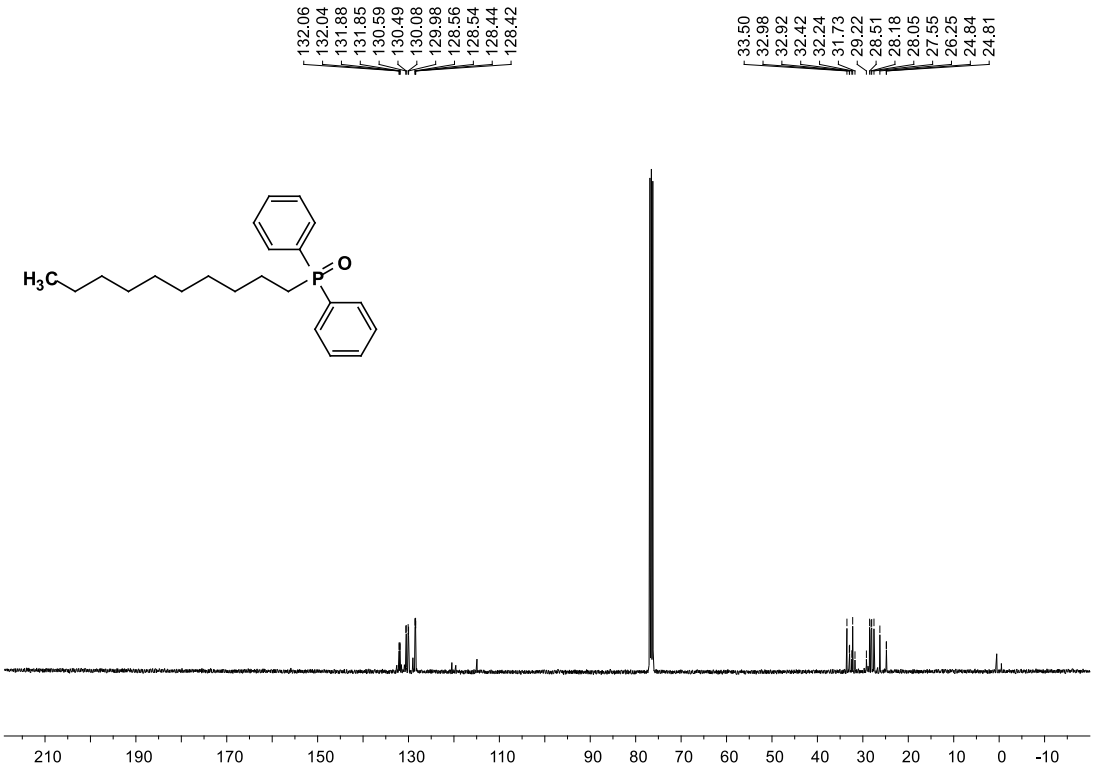

H3C
P
O
N
f1 (ppm)

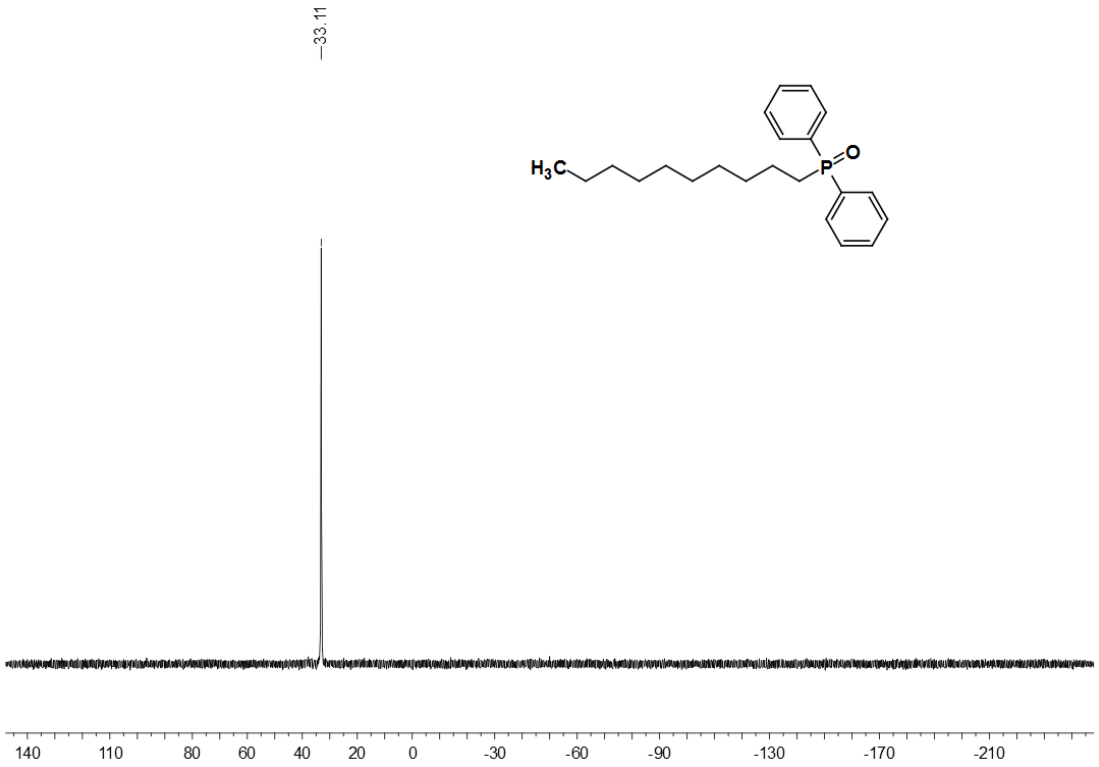

132.03
132.01
131.85
131.82
130.60
130.51
130.09
130.00
128.54
128.52
128.42
128.41
120.49
115.03
33.06
33.00
32.48
31.79
31.28
28.70
28.62
28.33
26.32
24.84
24.82
22.13
13.60
H3C
P
O
N
f1 (ppm)


28.63
H3C
P
O
N
f1 (ppm)

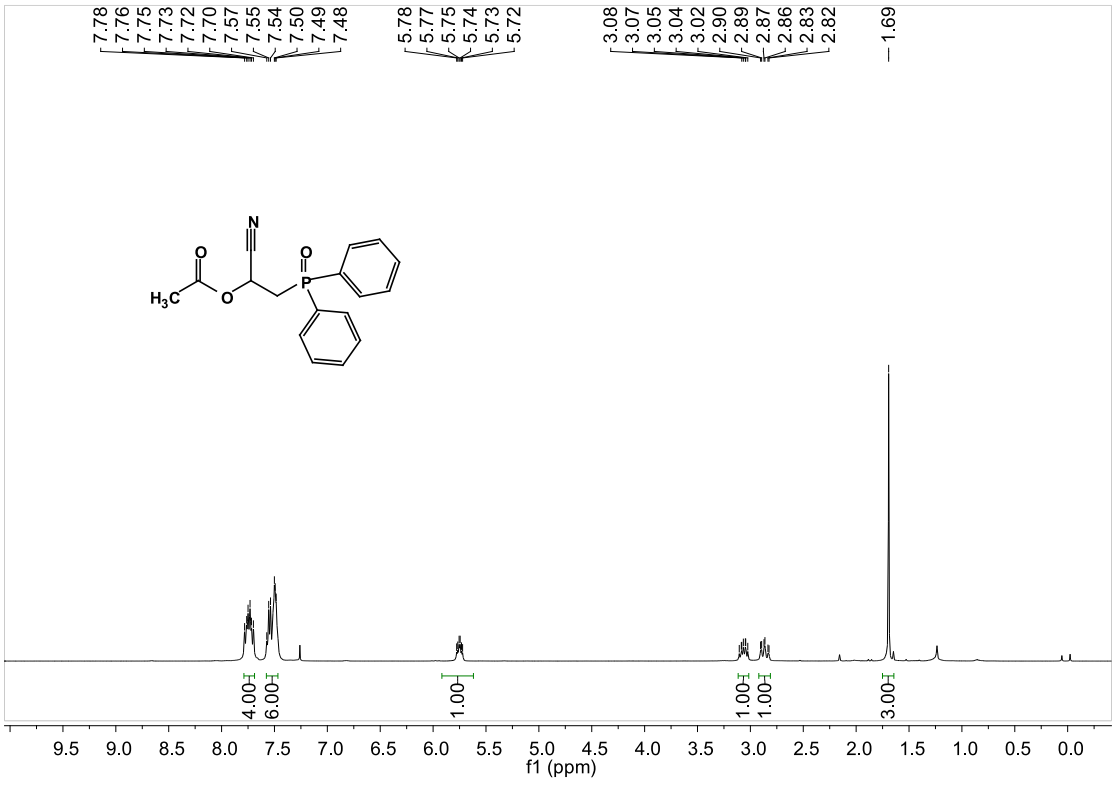
Compound 5f'

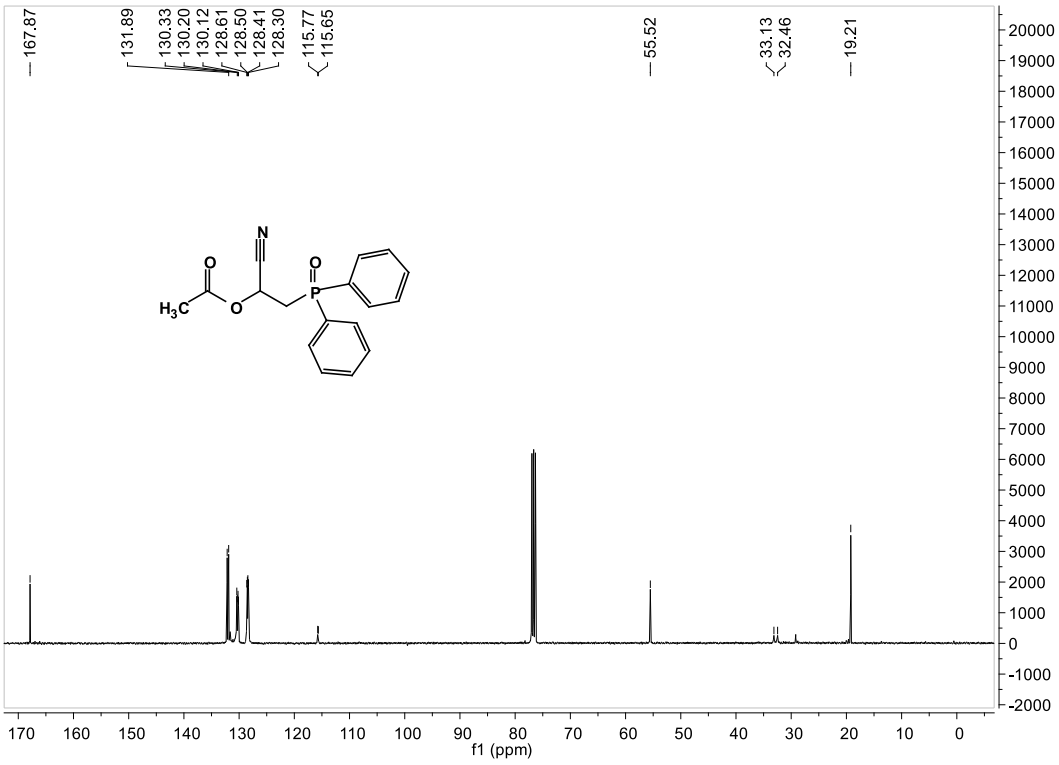

7.75
7.73
7.72
7.71
7.52
7.50
7.49
7.47
7.45
7.44
7.43
2.29
2.26
2.25
2.22
1.58
1.36
1.21
0.87
0.86
0.84
H3C
P
O
4.03
6.20
2.00
2.06
2.07
12.19
3.03
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

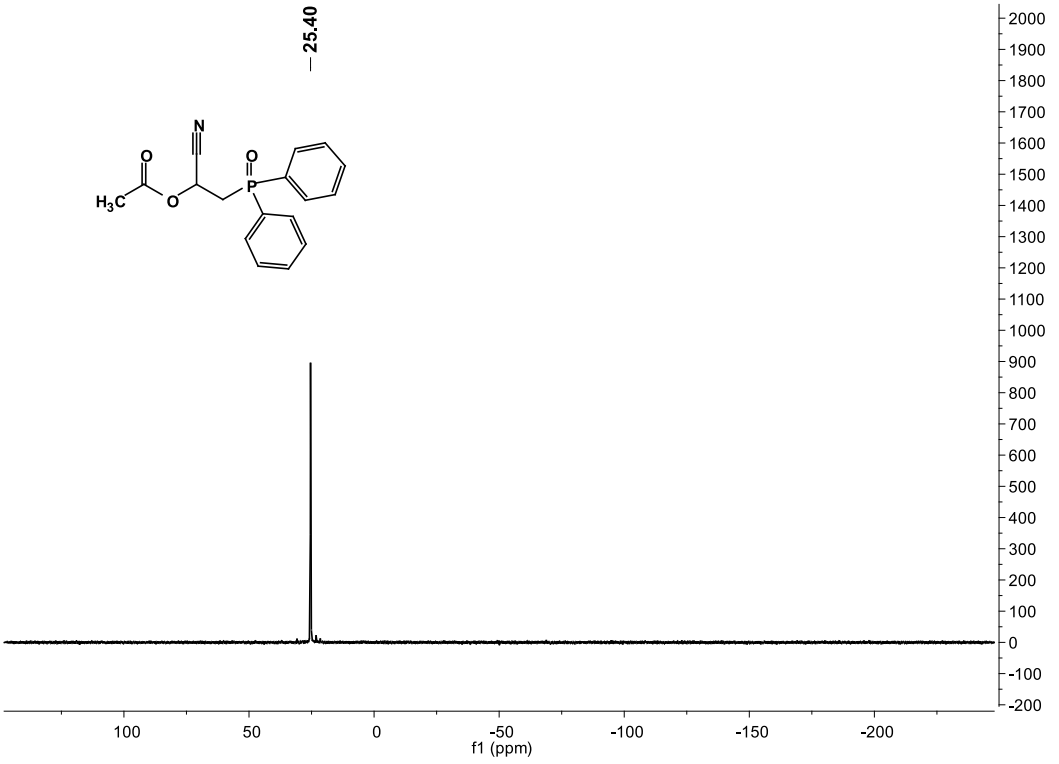

132.06
132.04
131.88
131.85
130.59
130.49
130.08
129.98
128.56
128.54
128.44
128.42
33.50
32.98
32.92
32.42
32.24
31.73
29.22
28.51
28.18
28.05
27.55
26.25
24.84
24.81
H3C
P
O
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10


33.11
H3C
P
O
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210

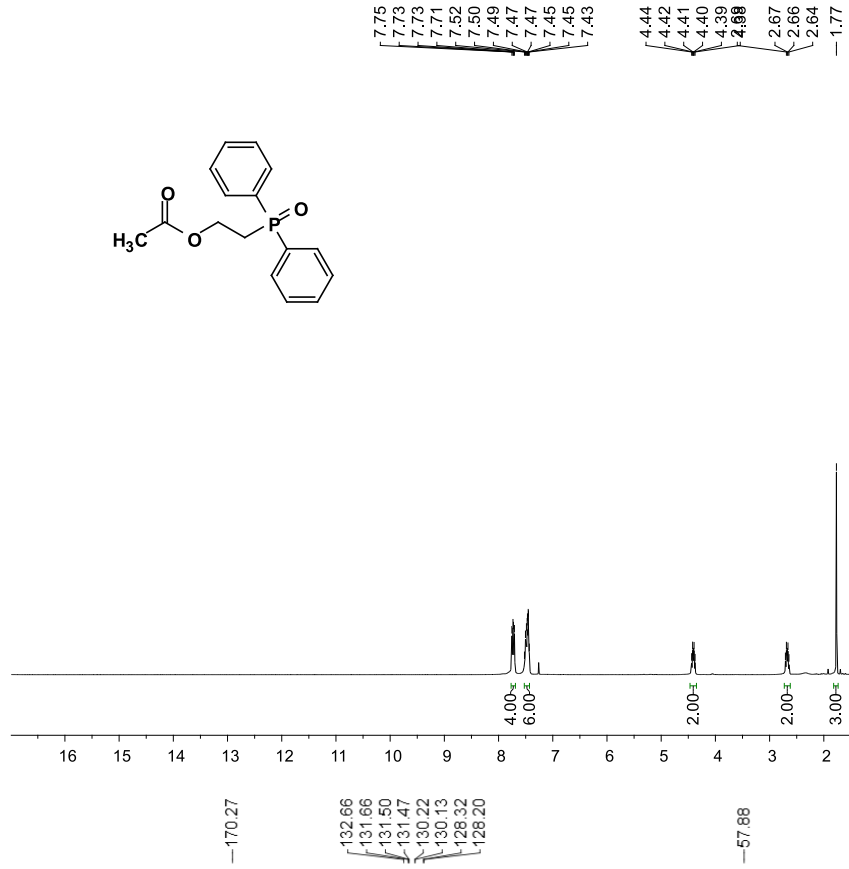
Compound 5g

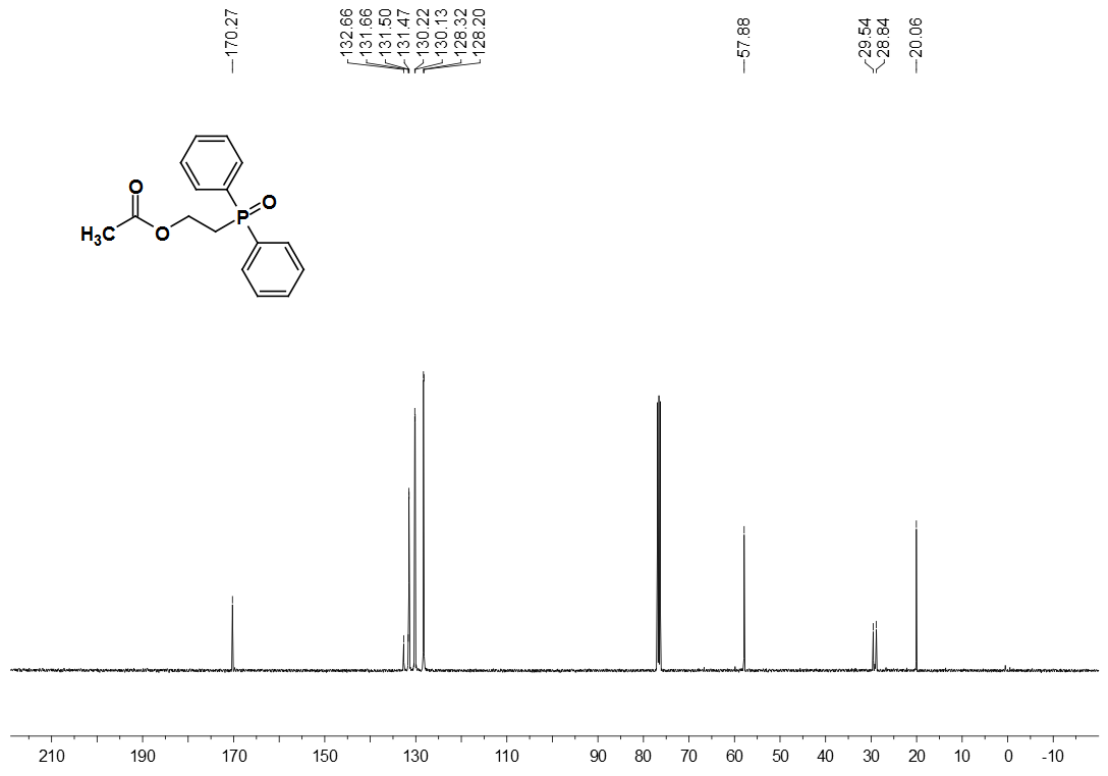

7.78
7.76
7.75
7.73
7.72
7.70
7.57
7.55
7.54
7.50
7.49
7.48
5.78
5.77
5.75
5.74
5.73
5.72
3.08
3.07
3.05
3.04
3.02
2.90
2.89
2.87
2.86
2.83
2.82
1.69
N
O
O
H3C
O
P
4.00
6.00
1.00
1.00
1.00
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

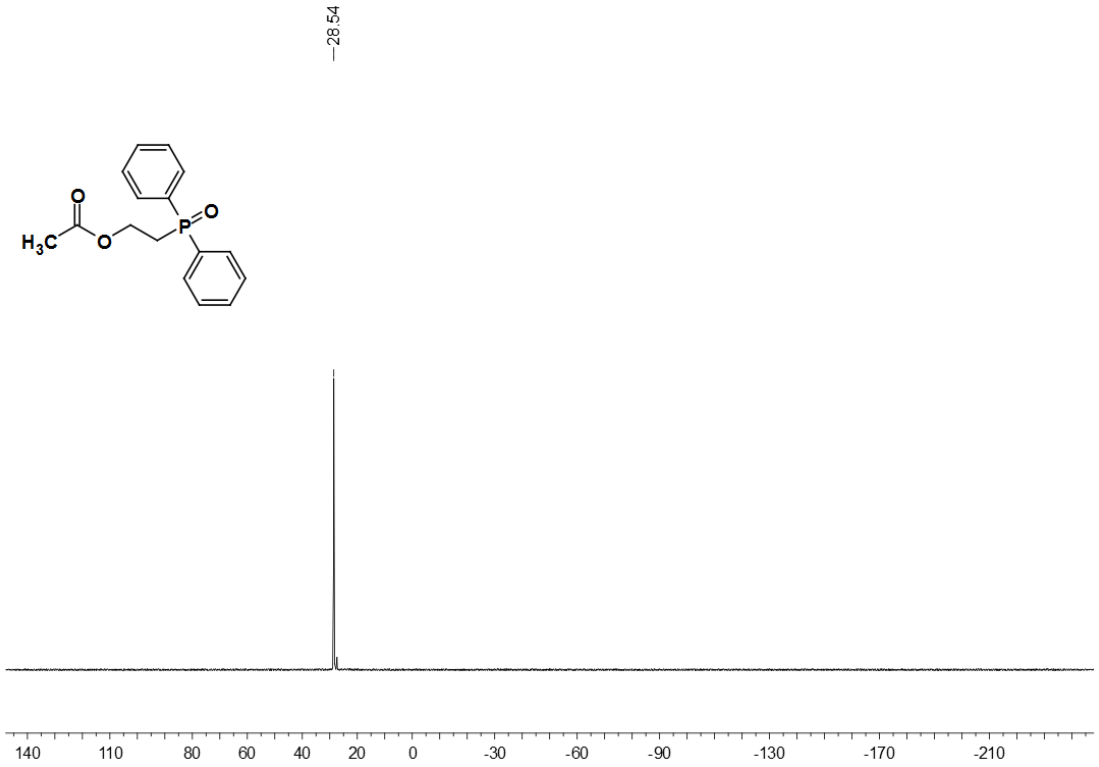

167.87
131.89
130.33
130.20
130.12
128.61
128.50
128.41
128.30
115.77
115.65
55.52
33.13
32.46
19.21
N
O
O
P
H3C
O
f1 (ppm)


25.40
N
O
O
P
H3C
O
f1 (ppm)

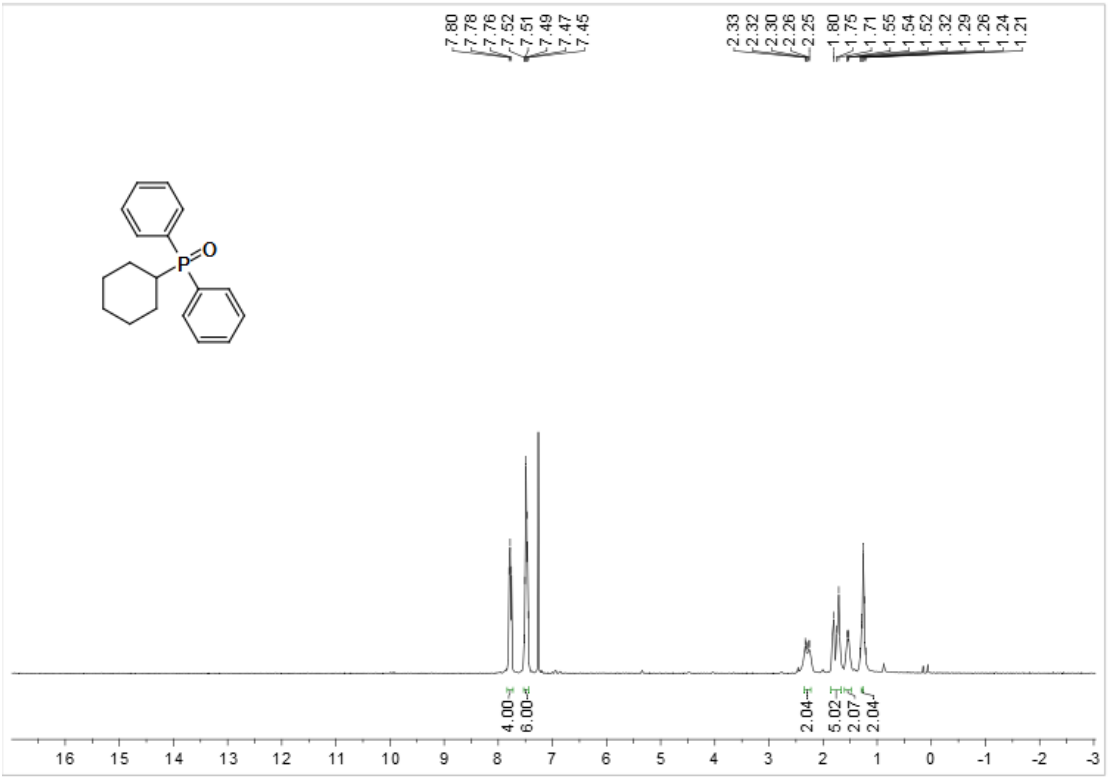
Compound 5g'

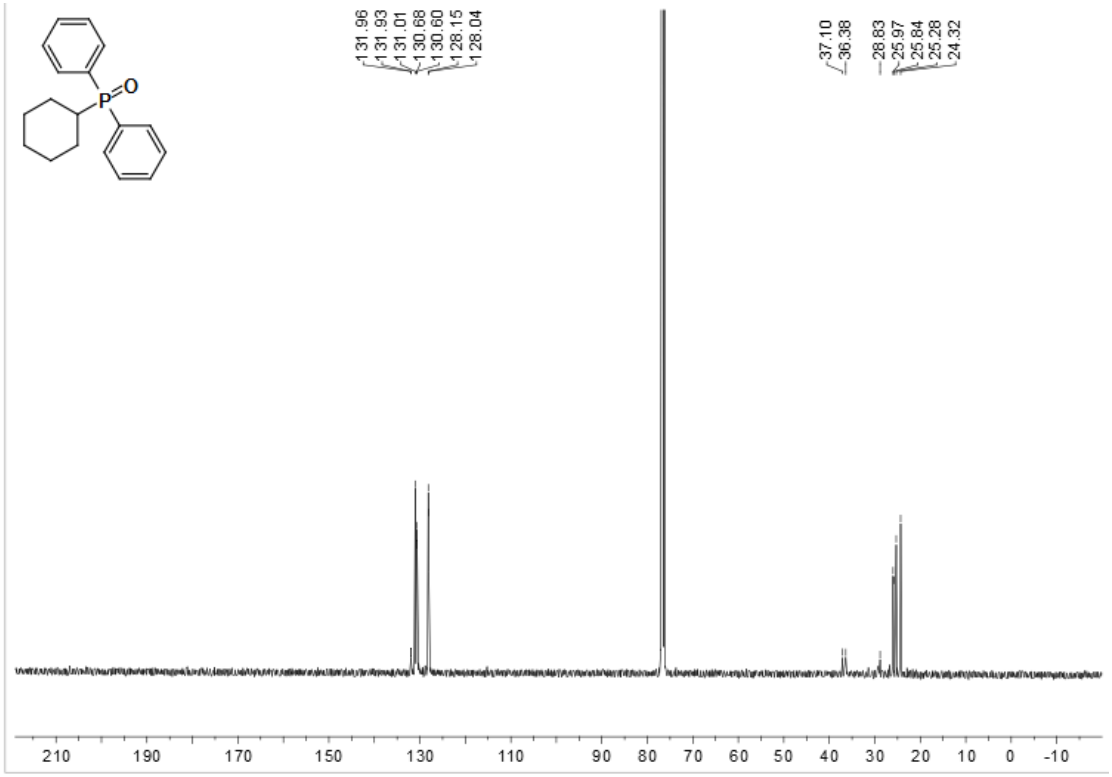

7.75
7.73
7.73
7.71
7.52
7.50
7.49
7.47
7.47
7.45
7.45
7.43
4.44
4.42
4.41
4.40
4.39
2.67
2.66
2.64
1.77
O
O
P
O
H3C
4.00
6.00
2.00
2.00
3.00
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 -3

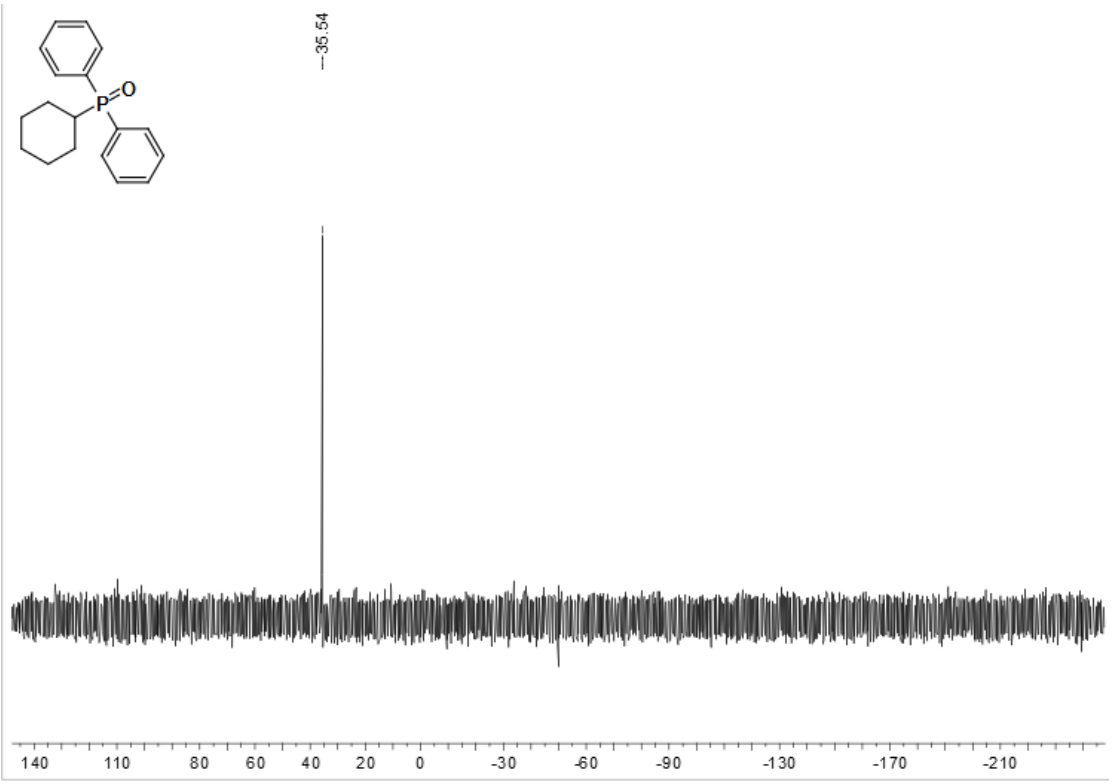

170.27
132.66
131.66
131.50
131.47
130.22
130.13
128.32
128.20
57.88
29.54
28.84
20.06
O
O
P
O
H3C
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10


28.54
O
H3C
O
P
O
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210

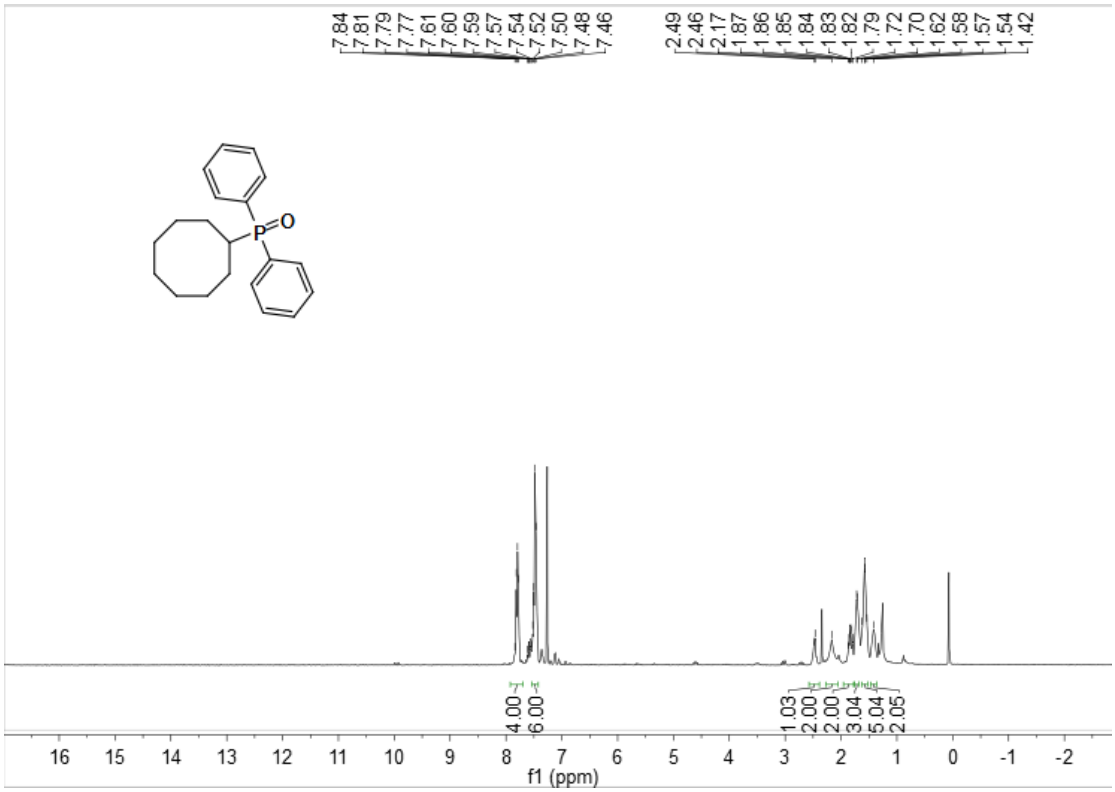
Compound 5h'

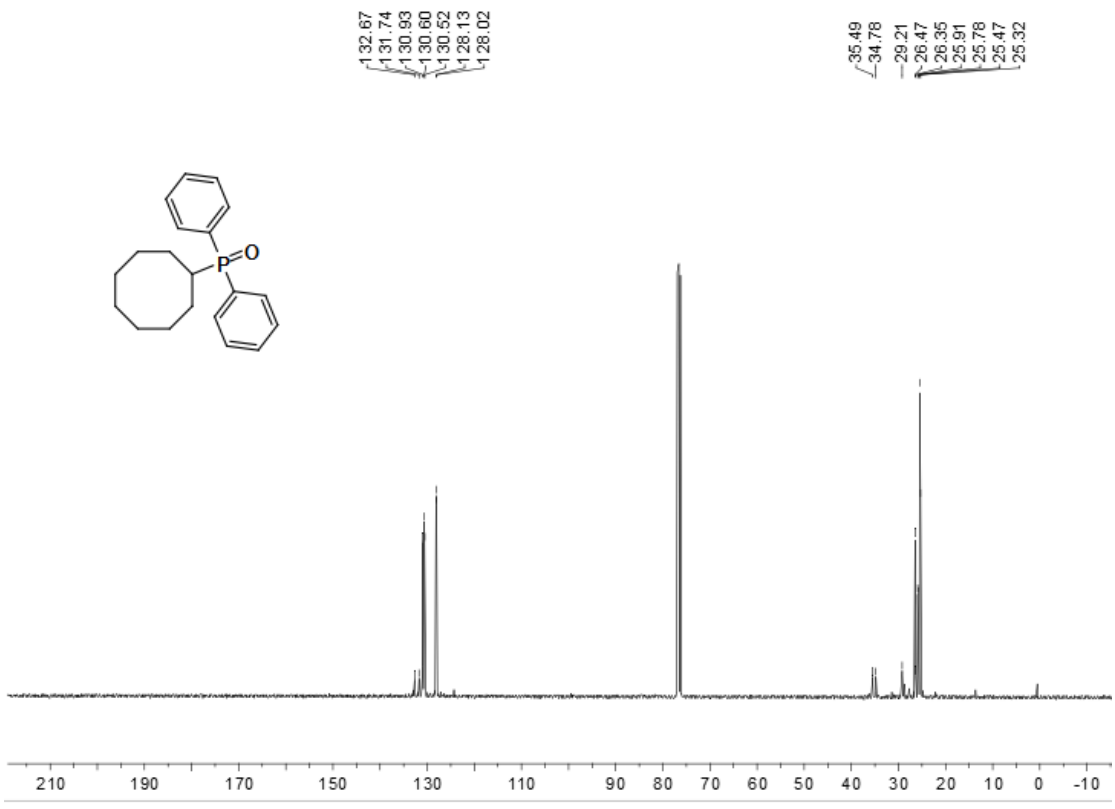

7.80
7.78
7.76
7.52
7.51
7.49
7.47
7.45
2.33
2.32
2.30
2.26
2.25
1.80
1.75
1.71
1.55
1.54
1.52
1.32
1.29
1.26
1.24
1.21
P
O
4.00
6.00
2.04
5.02
2.07
2.04
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3

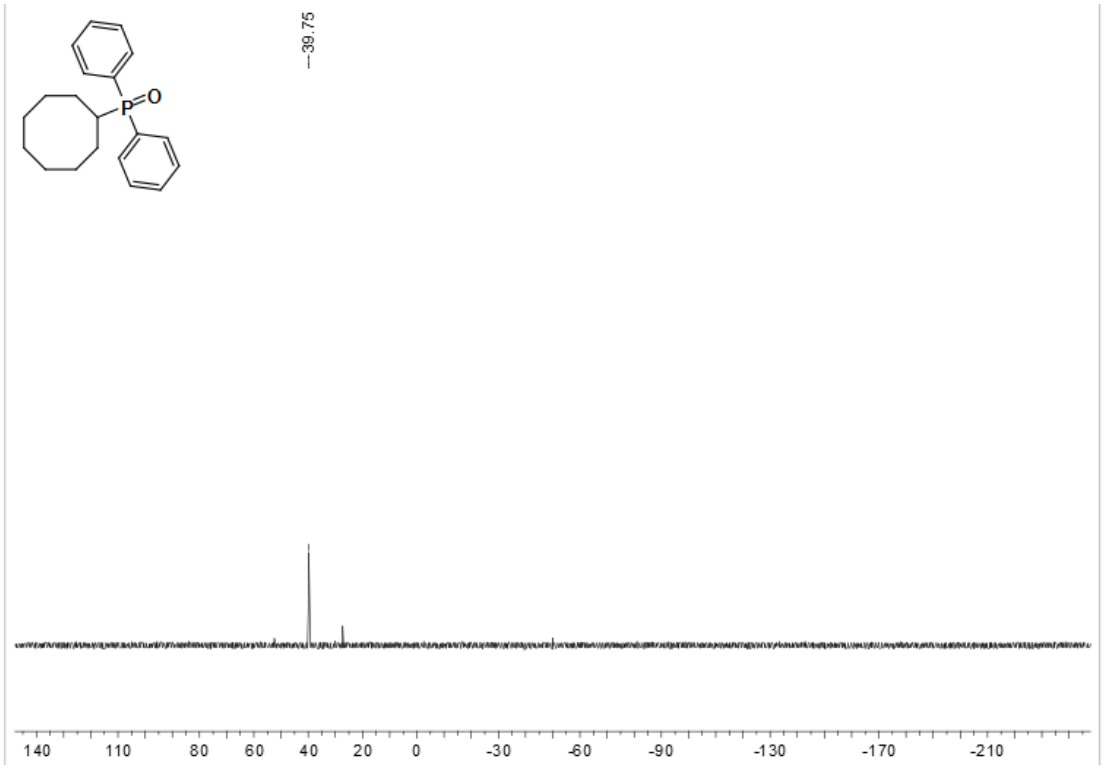

131.96
131.93
131.01
130.68
130.60
128.15
128.04
37.10
36.38
28.83
25.97
25.84
25.28
24.32
O
P
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10


35.54
O
P
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210

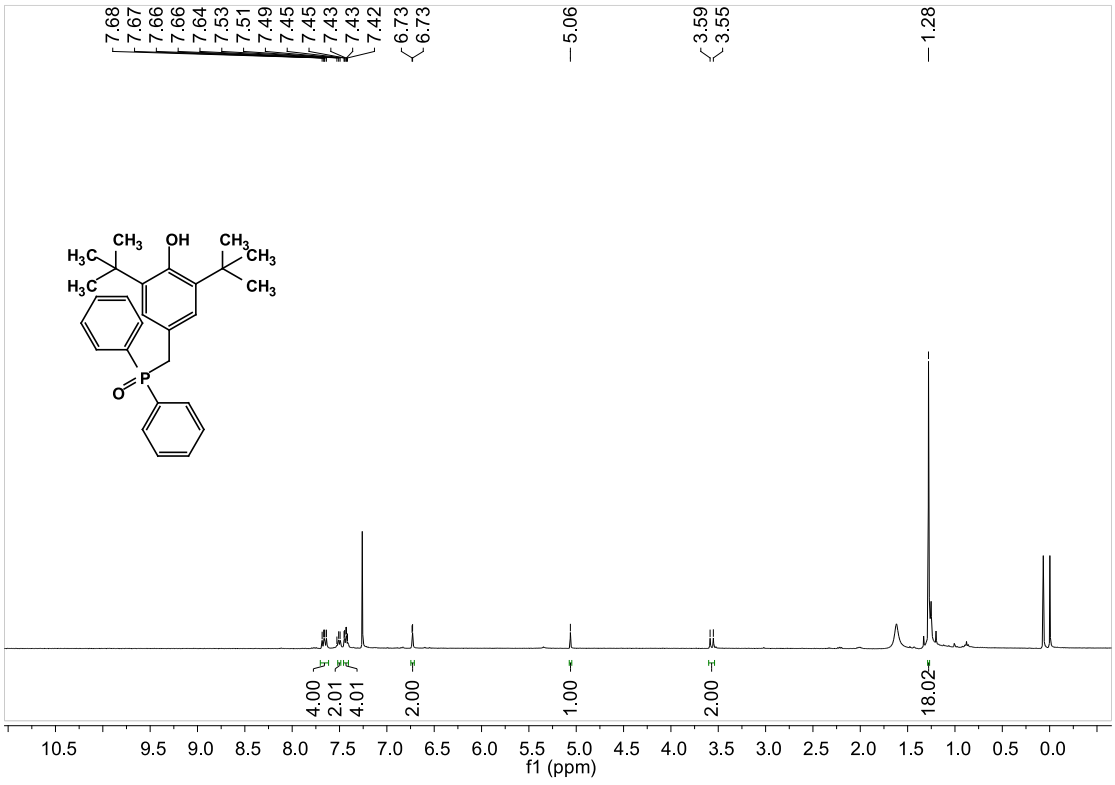
Compound 5i'

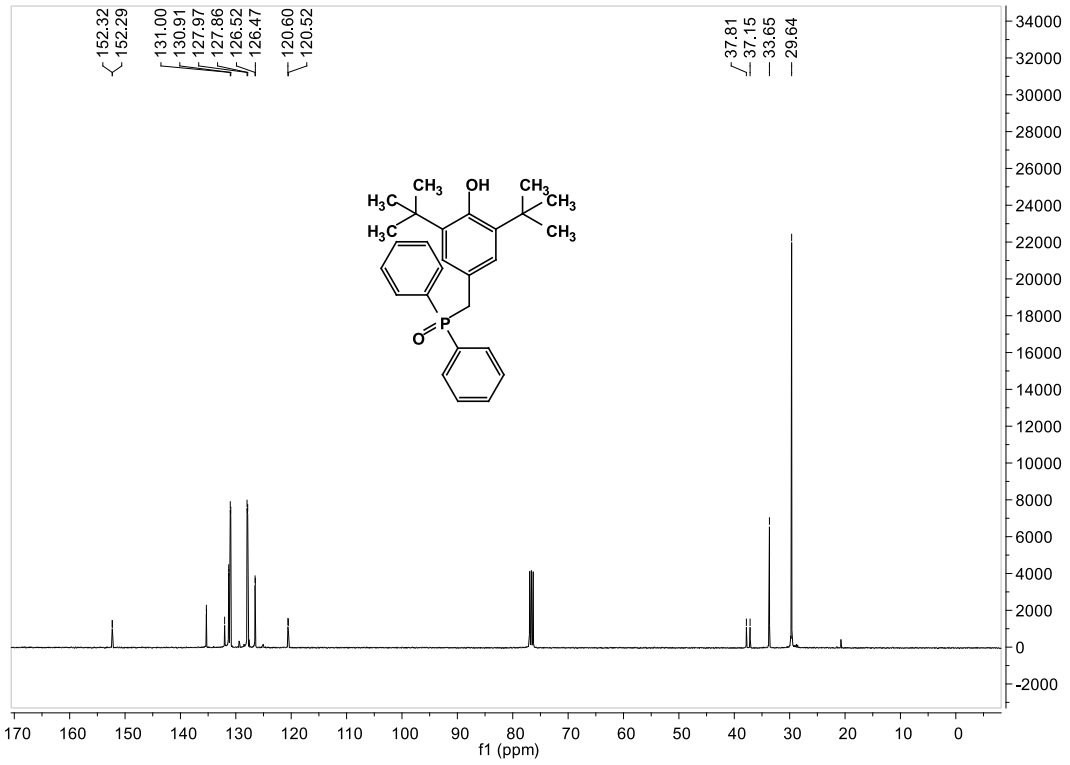

7.84
7.81
7.79
7.77
7.61
7.60
7.59
7.57
7.54
7.52
7.50
7.48
7.46
2.49
2.46
2.17
1.87
1.86
1.85
1.84
1.83
1.82
1.79
1.72
1.70
1.62
1.58
1.57
1.54
1.42
P
O
4.00
6.00
1.03
2.00
2.00
3.04
5.04
2.05
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2
f1 (ppm)

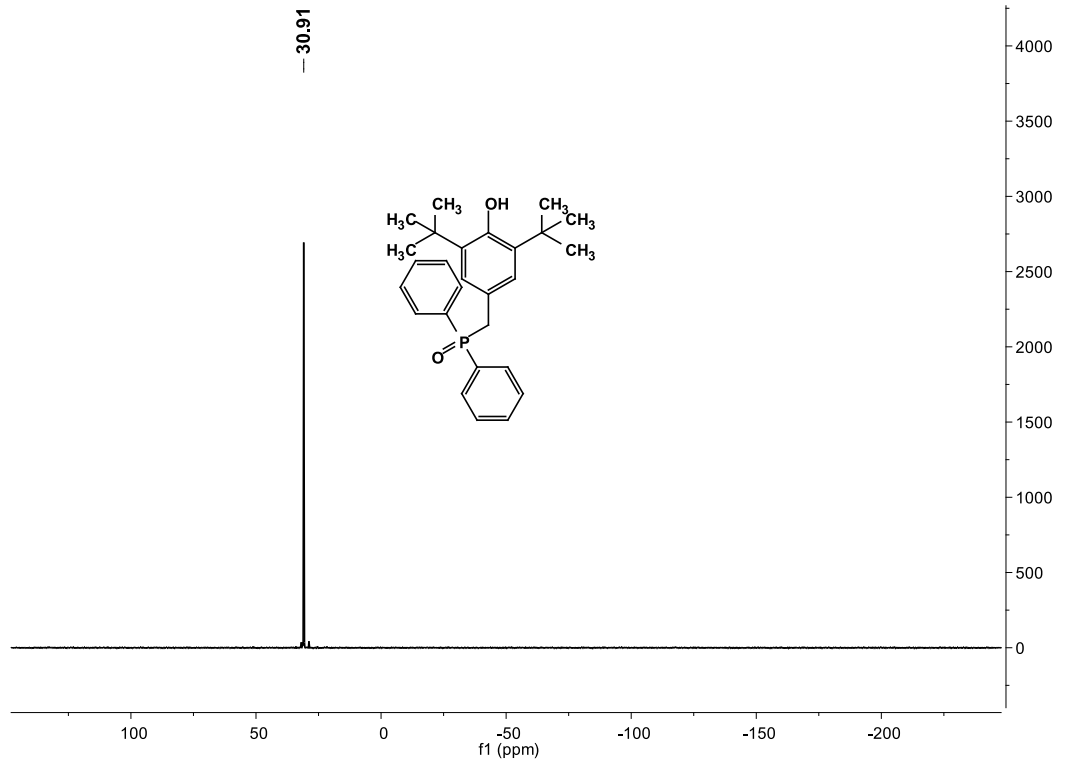

132.67
131.74
130.93
130.60
130.52
128.13
128.02
35.49
34.78
29.21
26.47
26.35
25.91
25.78
25.47
25.32
P
O
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10


39.75
140
110
80
60
40
20
0
-30
-60
-90
-130
-170
-210

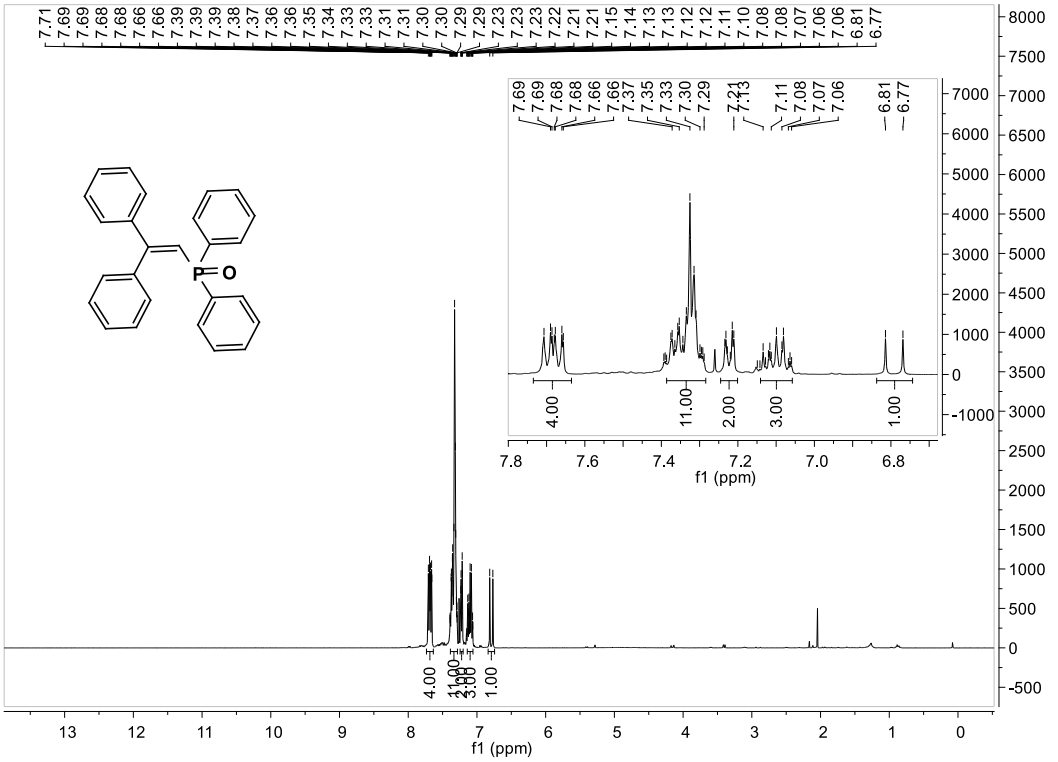
Compound 6

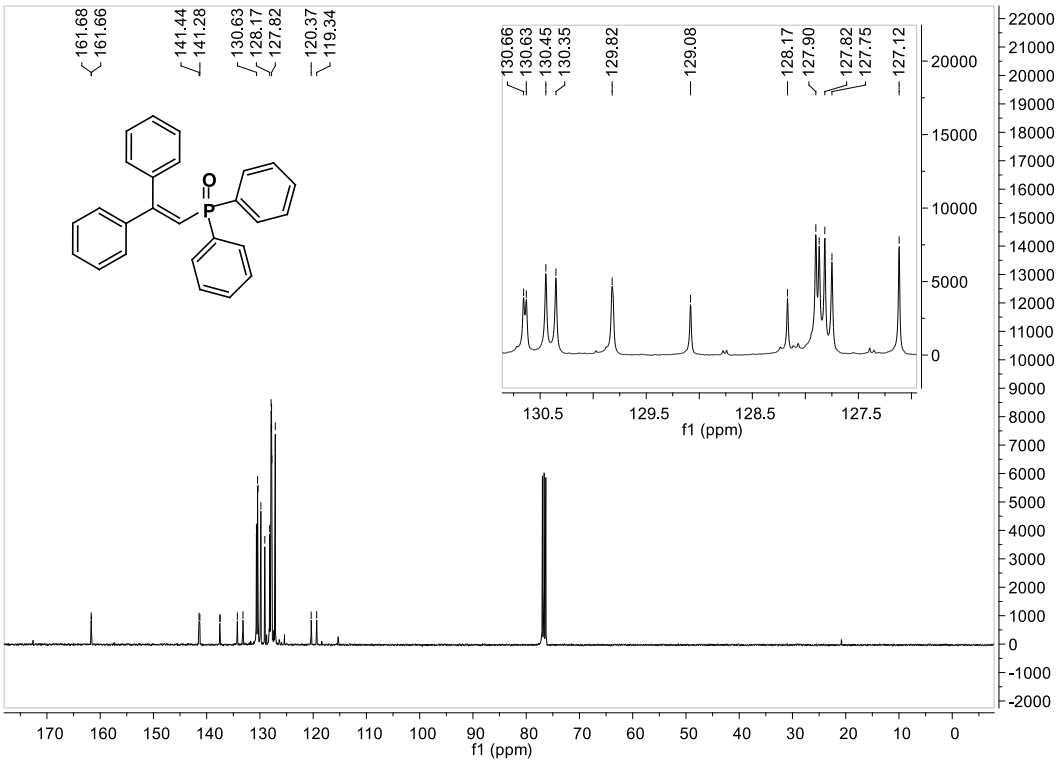

7.68
7.67
7.66
7.66
7.64
7.53
7.51
7.49
7.45
7.45
7.43
7.43
7.42
6.73
6.73
5.06
3.59
3.55
1.28
4.00
2.01
4.01
2.00
1.00
2.00
18.02
f1 (ppm)

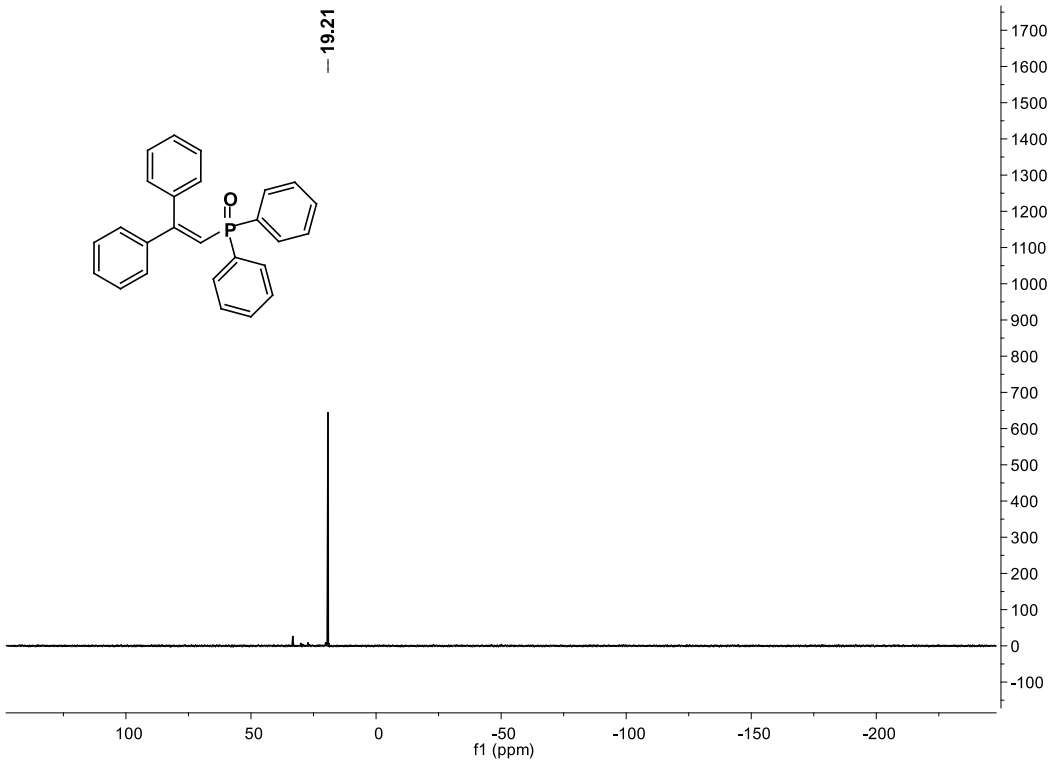

152.32
152.29
131.00
130.91
127.97
127.86
126.52
126.47
120.60
120.52
37.81
37.15
33.65
29.64
CH3
OH
CH3
H3C
CH3
H3C
CH3
O
P
f1 (ppm)


30.91
CH3
OH
CH3
H3C
CH3
H3C
CH3
O
P
f1 (ppm)

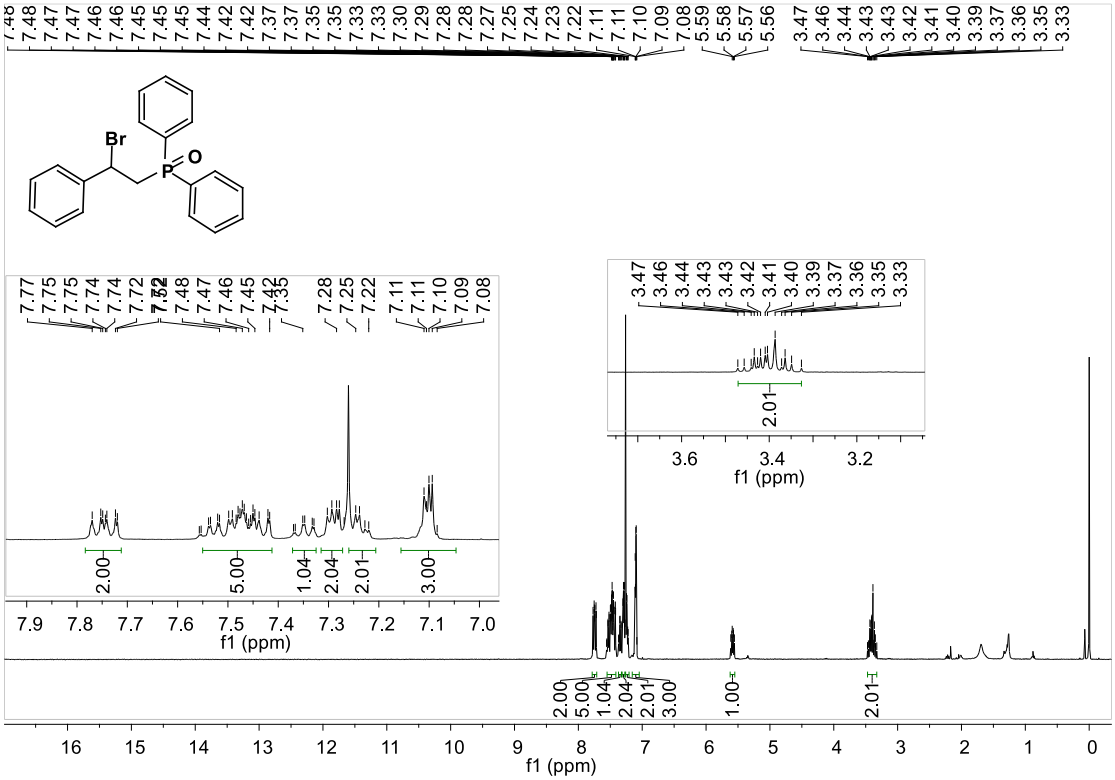
Compound 7

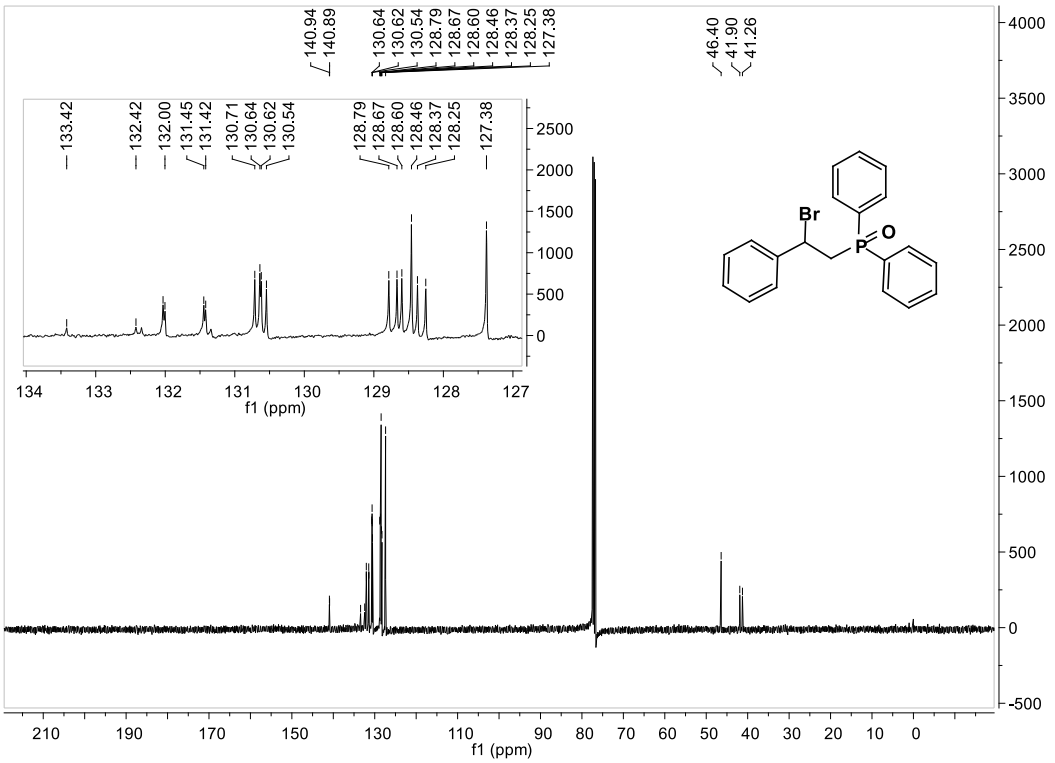

7.71 7.69 7.69 7.68 7.68 7.66 7.66 7.39 7.39 7.38 7.37 7.36 7.36 7.35 7.34 7.33 7.31 7.31 7.30 7.30 7.29 7.29 7.23 7.23 7.22 7.21 7.21 7.15 7.14 7.13 7.13 7.12 7.11 7.10 7.08 7.08 7.07 7.06 7.06 6.81 6.77
P O
4.00 11.00 2.00 3.00 1.00
f1 (ppm)

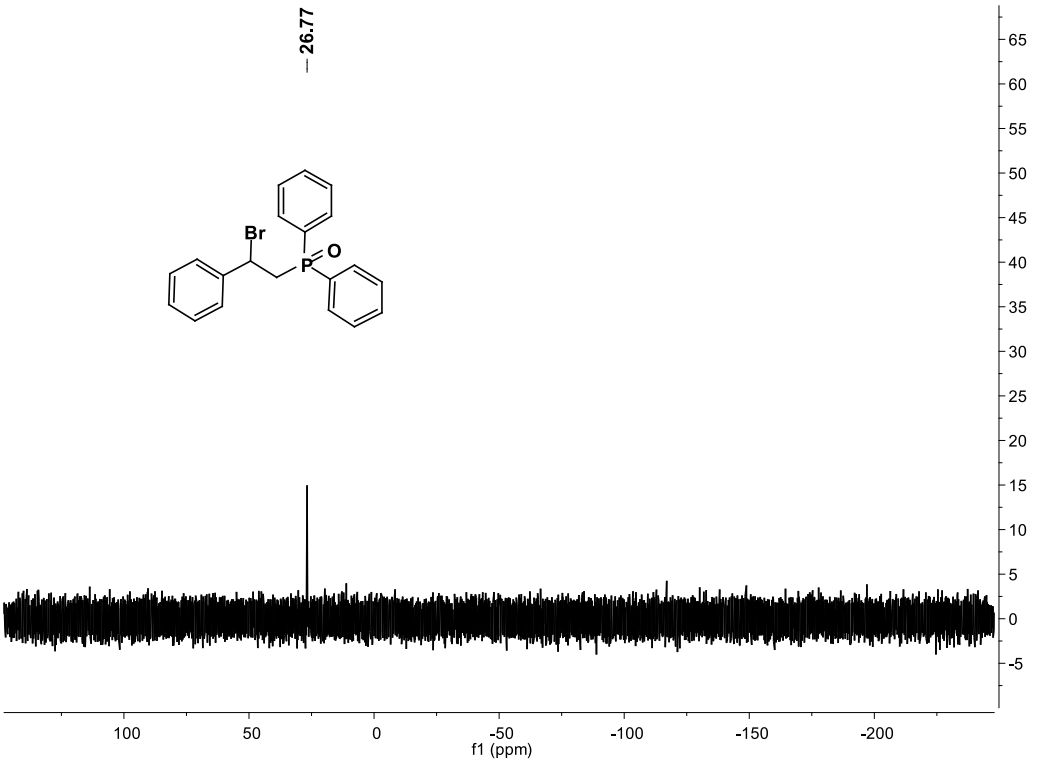

161.68 161.66 141.44 141.28 130.63 128.17 127.82 120.37 119.34
130.66 130.63 130.45 130.35 129.82 129.08 128.17 127.90 127.82 127.75 127.12
O P
f1 (ppm)


19.21
O
P
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
400
300
200
100
0
-100
100
50
0
-50
-100
-150
-200
f1 (ppm)

Compound 8


Br
O
P
7.48
7.47
7.46
7.45
7.44
7.42
7.37
7.35
7.33
7.30
7.29
7.28
7.27
7.25
7.24
7.23
7.22
7.11
7.10
7.09
7.08
5.59
5.58
5.57
5.56
3.47
3.46
3.44
3.43
3.42
3.41
3.40
3.39
3.37
3.36
3.35
3.33
2.00
5.00
1.04
2.04
2.01
3.00
1.00
2.01
f1 (ppm)


140.94
140.89
130.64
130.62
130.54
128.79
128.67
128.60
128.46
128.37
128.25
127.38
46.40
41.90
41.26
133.42
132.42
132.00
131.45
131.42
130.71
130.64
130.62
130.54
128.79
128.67
128.60
128.46
128.37
128.25
127.38
Br
O
P
f1 (ppm)


26.77
Br
O
P
f1 (ppm)